AN ABSTRACT OF THE DISSERTATION OF

Jose Antonio Silva Guzman for the degree of Doctor of Philosophy in Wood Science presented on December 15, 2003.
Title: Development of an Accelerated Method for Assessing Decay of Wood Plastic Composites (WPC's).

Abstract approved:

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This study examined the decay resistance of the pine and maple components of wood plastic composites (WPC's) of varying thicknesses exposed on several culture media to wood decay fungi under laboratory conditions. The ability of malt agar extract (MEA), potato dextrose agar (PDA), amended basal salts, sawdust (maple and red alder), soil (direct exposure, sandwich system and soil block tests), vermiculite, and malt liquid broth (stationary and rotary shaker conditions) to enhance WPC decay was compared with traditional soil block tests.

Modulus of elasticity (MOE), modulus of rupture (MOR), creep, ultimate tensile strength (UTS), moisture content, weight loss or scanning electronic microscopy were

used to assess fungal effects on the WPC. MOE, MOR, creep and UTS were poor parameters for assessing decay because the plastic component tended to dominate WPC properties and was not susceptible to fungal attack.

Agar and soil block tests were both suitable for assessing wood decay of the WPC's. Decay rates were strongly influenced by media type and test fungus. WPC specimens exposed to *Trametes versicolor* or *Postia placenta* on 1.5% PDA, and those specimens exposed to *Gloeophyllum trabeum* and *P. placenta* on MEA reached higher moisture contents and experienced greater weight losses than specimens exposed to the same fungi in the soil block test. Liquid media was unsuitable for enhancing WPC decay, probably due to oxygen limiting conditions.

Scanning electron microscopy (SEM) showed that surface wood particles exposed directly to fungal attack were partially or totally degraded, increasing the void volume and exposing more wood particles to fungal attack, regardless of fungus or composite type.

WPC specimens made of maple were more susceptible to fungal degradation than those made with pine. Increasing WPC thickness reduced moisture content and weight loss of the wood. Slow moisture uptakes sharply reduced fungal attack in thicker specimens. A strong relationship was found between weight loss and moisture content; higher weight losses were consistently associated with higher moisture contents.

The results indicate that WPC decay can be accelerated using both agar and soil as media, provided the specimens are small enough to rapidly sorb moisture.

Development of an Accelerated Method for Assessing Decay of Wood Plastic Composites (WPC's)

By
Jose Antonio Silva Guzman

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I understand that my dissertation will become part of the permanent collection of Oregon State University libraries. My signature below authorizes release of my dissertation to any reader upon request.

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Jose Antonio Silva Guzman, Author

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CONTRIBUTION OF AUTHORS

Camille Freitag assisted in the sample preparation and experimental set up of Chapter 2.

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DEDICATION

Dedicated to my Wife Alma, my daughters Deyanira, Karen and my little son Antonio, and also to my Mother Olaya Guzman and my brothers and sisters Jose, Reyna, Eugenio, Ruben, Rosa and Lurdes. In memory of my Father Antonio Silva and my grandmother Mamanita. They ALWAYS had been strongly influenced my life and showed their love and special dedication to me. Also, dedicated to all those persons who always believed in me.

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Chapter 1

Introduction, Objective and Background

1.1 Introduction

High lumber prices and customer desires for recycled products have encouraged more efficient use of wood. One potential approach to conserving wood is the development of commodity-engineered composites that blend wood with other materials, such as plastic. The idea of combining wood and plastic is to produce a product with performance characteristics that combine the positive attributes of both materials.

Wood and natural fiber composites are the fastest growing plastics and building products market segment, with more than 25% growth per year since 1998 (Morton et al., 2003). In the short term (2003 to 2005), markets for these products will grow 20% a year, and an annual growth rate of 14% is expected for wood/plastic composites (WPC's) by 2010 (Morton et al., 2003). The phase-out of chromated-copper-arsenate (CCA) treated wood for residential uses such as decks, playgrounds, and fencing at the end of 2003 may help to further increase market growth for WPC's (Clemons, 2002).

Decking represents 53% of the total WPC's volume, followed by windows, door framing and railings. Roofing, fencing, sea walls, garden structures and patio furniture are emerging applications for these composites (Morton et al., 2003). Although WPC decking is more expensive than pressure-treated wood, manufacturers promote its lower maintenance, lack of cracking, and high durability. The actual lifetime of WPC lumber

is currently being debated, but most manufacturers offer a 10-year warranty (Clemons, 2002).

Economics is the major factor in development of a WPC. Wood fibers are estimated to be one-third the cost of fiberglass (Global Hemp, 2000). Wood fibers have been used as filler and/or reinforcement to improve mechanical properties of a variety of products. The combination of wood and plastic creates the ability to develop products using many different manufacturing processes, such as extrusion or molding, as well as the ability to create an infinite array of products that vary in wood content as well as type of plastic. The use of smaller wood particles, new additives, and better processing technologies allow WPC manufacturers to produce an ever increasing array of materials, often without developing a full understanding of how these materials will behave under conditions conducive to fungal attack.

Plastics are generally resistant to fungal attack; however a major concern with these materials is that wood in the composite remains susceptible to biological degradation. Many manufacturers avoid this risk by producing products for interior uses where little or no water is present, thereby minimizing the risk of fungal attack (Tangram Technology, 2001). There is little data on decay patterns or effects of fungal attack on physical and mechanical properties of WPC's, although new reports are emerging in this rapidly expanding area. Initially, it was presumed that plastic encapsulated the wood fibers, protecting them from wetting and further decay, but a number of tests suggest that wood encapsulation by plastic is incomplete. As a result,

the wood component in these materials reaches moisture levels suitable for fungal attack (Naghipour, 1996; Mankowski and Morrell, 2000).

The expanding commercial production and marketing of WPC's for use in exterior applications has encouraged research on the durability and service life of WPC's. The traditional soil block method used for testing durability of solid wood is generally less effective for evaluating WPC's than for evaluating solid wood. This test produces low weight losses on WPC's in comparison to solid wood. While the soil block test can provide valuable information, it is evident that a new method will be required for assessing decay resistance of WPC's that accounts for the characteristics and properties of this composite.

An American Society for Testing and Material (ASTM) subcommittee composed of materials scientists, wood scientists and polymer scientists is attempting to develop standards for performance ratings for wood plastic composite deck boards (ASTM, 2003), but there is little published on actual method development.

The absence of definitive information on decay resistance of WPC's ultimately will limit the ability to reliably and rapidly assess the durability of new materials. This study seeks to develop an accelerated method for assessing decay resistance of wood plastic composites.

1.2 Objective

The overall objective of this project was to develop an accelerated laboratory method for assessing decay resistance of wood plastic composites.

This dissertation is divided into six chapters. The background literature is included in Chapter I. Chapter II assesses the effects of fungal attack on bending and creep of untreated and treated wood plastic composites. Chapter III evaluates the effectiveness of malt extract agar, red alder sawdust, maple sawdust, vermiculite, soil or liquid media (stationary and rotary shaker) as culture media for assessing fungal decay of wood plastic composites. This chapter also assesses the effect of culture media containing minimal salts amended with glucose, and the effect of carbon to nitrogen ratios (C:N) on decay rate. Chapter IV describes the effectiveness of liquid culture media and tension tests for assessing decay of wood plastic composites. The effects of maple and pine and the composite thickness on decay rate of wood polypropylene composites is described in Chapter V. Finally, the overall conclusions are presented in Chapter VI.

1.3 Background

1.3.1 Wood plastic composites

Combining lignocellulosic materials with thermoplastics is not new. The first wood-thermoset composites date to the early 1900s (Clemons, 2002). Wood plastic composites (WPC's) were widely investigated in 1960's, and substantial amounts of wood/plastic flooring were produced for airport terminals and office buildings in the 1960's (Sanadi et al., 1998).

The term "wood plastic composite" refers to any composites that contain wood and thermosets (plastic that do not melt by reheating) or thermoplastics (plastic that can be repeatedly melted). WPC's are also known as natural fiber composites, wood fiber

plastic composites, wood fiber thermoplastics composites, polymer/wood composites, and wood filled plastics (Forest Products Laboratory, 1999). The objective of wood plastic composite development is to produce a product with performance characteristics that combine the positive attributes of wood and plastics (Wolcott, 1996). The addition of wood significantly improves thermal stability, mechanical (stiffness) and working properties of the WPC. The disadvantages of using wood fibers are their low bulk density, low thermal stability, and high tendency to absorb moisture and susceptibility to fungal attack (Clemons, 2002). Plastic coating of wood particles in a WPC can reduce the moisture uptake while enhancing dimensional stability and protection against fungal attack (Wolcott, 1996). Furthermore, wood particles also reduce the need to use more costly thermoplastics (Oksman and Clemons, 1998).

In the mid 1980's, elevated lumber prices and the desire of customers for recycled products stimulated the production of lumber substitutes produced from recycled thermoplastics. However, the totally thermoplastic lumber exhibited limited applications and suffered from poor thermal stability, high heat retention, poor creep resistance, and low fastener holding properties (Wolcott, 1996). These problems stimulated interest in using wood as filler or reinforcement in combination with thermoplastics due to the advantages offered by wood fibers over inorganic fillers (calcium carbonate and mica) and reinforcements (glass or carbon fibers) in thermoplastics. In contrast to the glass and carbon fiber fillers, wood fibers are abundant, renewable, strong (high stiffness), lightweight (low density), less abrasive to

processing equipment, non-hazardous, non-toxic to mammals, relatively inexpensive, easy to process, and available from a variety of sources (English, 1996).

Perhaps the most common reason for the historically low use of these natural fibers in thermoplastics was unfamiliarity. The birth of the wood plastic industry involved the interfacing of two industries that historically had little in common (Clemons, 2002). Initial failures in the early stages of WPC's development occurred because thermoplastic manufacturers were unaware of the effects of the high hygroscopicity and thermal degradation of wood on composite production (Clemons, 2000). WPC's are currently produced for commercial purposes in many countries (Haquel, 1997; Sanadi et al., 1998), however, production has been limited because of poor interfaces between wood and plastic, low thermal resistance of plastic, high thickness swelling, and thermal degradation of wood fibers at high temperatures (Naghipour, 1996). As a result there is a continued need for development of these materials.

1.3.1.1 Wood species

Wood particles used for manufacturing WPC's can originate from sawdust, planer shavings, short solid pieces of lumber, conventional wood composite scrap (English et al., 1996), and scrap pallets (Stark, 1999). Both softwoods and hardwoods can be used for WPC production. Currently, most WPC's using softwoods are made with southern yellow pine while most that are made with hardwoods are made with oak, maple or aspen. The anatomical features, physical, mechanical and chemical properties of softwoods and hardwoods differ considerably among species, and may affect the

wood-polymer interface, and, as a consequence, composite properties and performance. The effect of wood species on the wood- polymer interface and on properties of WPC's has been little studied. Stark and Berger (1997) evaluated the effect of ponderosa pine, loblolly pine, maple or oak on the mechanical properties of polypropylene WPC's. In general, WPC's made with maple or oak exhibited slightly better tensile and flexural properties and heat deflection temperatures than either of the pines.

1.3.1.1.1 Particle size

A variety of wood particle sizes are used to produce WPC's depending on the type of product. The dimensions of wood particles are usually measured in mesh size, as the particles resulting from passing through a mesh with a given number of mesh squares in a square inch. Wood used in WPC's is most often in particulate form (wood flour), or very short fibers rather then longer individual fibers. Commonly, mesh sizes 20, 40, 60, and 80 are used in WPC production.

Particle size can affect stiffness, moisture resistance (ability to withstand water uptake), wood/plastic interactions, and susceptibility to fungal attack of the resulting WPC (Stark and Berger, 1997; Verhey et al., 2002). WPC's produced from smaller particle sizes tend to exhibit increased water resistance and modulus of rupture (MOR) (Tatakani, 2000). Stark and Rowlands (2003) found that aspect ratio, not particle size, had the greatest effect on strength and stiffness. Particle size affects formation of the wood-polymer interface. Large wood particles have been associated with the formation of voids on the wood fiber polymer interface. These voids can serve as pathways for moisture movement and fungal colonization (Mankowski and Morrell, 2000). In

contrast, small particles improve the interface between the wood fibers and the polymer, and decrease the fiber-fiber contact and voids in the interface area by increasing the probability that a particle will be coated by plastic. These characteristics limit the potential for moisture uptake as well as fungal growth. Small, well-dispersed particles are also associated with better composite properties; however, wood particles are often difficult to disperse because of their tendency to agglomerate (Oskman and Clemons, 1998). Early WPC manufacturers tended to use larger wood particles (10 to 30 mesh) due to their lower processing costs, but the industry has reduced particle sizes to as small as 80 mesh. Theses changes appear to produce a material with better performance and more fungal resistance (Clemons, 2002).

1.3.1.1.2 Moisture content of wood particles

The moisture content in air-dry wood fibers ranges from 6 to 7%, but processes for manufacturing plastics tolerate little or no water. Even 1% or 2% moisture is considered too high (English, 1996; Clemons, 2002). Removal of water is critical because any moisture remaining in the wood-plastic blend turns to steam and manifests itself in the form of foam, disrupting processes, resulting in poor surface quality, weak wood-plastic interface, and voids that are unacceptable for final sale (English et al., 1996; Forest Products Laboratory, 1999). As a result, particles must be pre-dried for blending.

1.3.1.1.3 Role of wood fibers (particles) on the wood plastic composites

Wood particles serve as both reinforcement and filler in a continuous plastic matrix. According to Simonsen (1997), when the wood particles are acting as

reinforcement, their primary function in the polymer matrix is to support load along the length of the fiber, providing strength and stiffness in the fiber direction. As a consequence, the WPC properties such as tensile and flexural strength would be improved by the presence of wood fiber.

Wood particles used as filler serve as inert material for bulking the WPC, reducing the amount of more expensive plastic required for producing a given volume. The lower weight of wood fibers compared to other inorganic fillers, such as mineral fibers (i.e., glass fiber) save considerable weight (English, 1996). The addition of wood fiber may also improve thermal stability of the WPC (English, 1996).

1.3.1.2 Types of polymers used in wood plastic composites manufacturing

Thermoplastic linear or branched polymers become rigid when cooled and soften at varying elevated temperatures (depending on the polymer). Only thermoplastics that melt below 200°C (392°F) are commonly used in WPC's because the wood cannot withstand higher extrusion temperatures. Several thermoplastics polymers including polypropylene, polyethylene and polyvinyl chloride, are currently used to produce WPC's. The polymer in the WPC's transfers stress between reinforcement fibers, acts as a glue to hold fibers together, protects fibers from mechanical and environmental damage, and improves durability (American Composite Timbers, 2003). Both virgin and recycled polymers can be used to produce WPC's. This flexibility creates the potential for using recycled plastics, although care must be taken to ensure reasonable uniformity in the recycled products to avoid plastics with higher melting temperatures due to concerns about cellulose decomposition. One great

advantage of WPC's is that they can be melt-processed or extruded for further processing (Haquel, 1997; English, 2000). This feature creates the potential to recycle the material by grinding or beading for later heating and extrusion.

1.3.1.3 Additives

There are several important reasons for using additives when producing WPC's. Additives improve the manufacturing process and/or enhance composite performance, durability, and aesthetics (Fulmer, 1999). Susceptibility to ultraviolet light (UV) degradation or fungal attack remain the two primary reasons for additive use in WPC's for exterior applications. Additives include adhesives, lubricants, and/or surfactants. Other additives utilized in WPC's include colorants, fungicides, foamers, and UV (ultraviolet light) stabilizers, which are essential for exterior applications since they prevent crazing (development of ultra-fine cracks), and disintegration due to UV absorption (American Composite Timbers, 2003), and improve aesthetics. Additionally, additives can modify surface energy; improve fiber dispersion and orientation, and increase interfacial adhesion through mechanical interlocking (Sanadi et al., 1998; Oskman and Clemons, 1998).

1.3.1.4 Wood/polymer ratio

Since the plastic is largely immune to fungal attack, the amount of wood and plastic (wood/plastic ratio) has a direct effect on WPC decay resistance. Currently, the most common wood/plastic ratio used to manufacture WPC's 50/50 wood/polymer, but 40/60, and even 70/30 ratios are also used (Pendleton et al., 2002; Clemons, 2002). The optimum wood/plastic ratio depends on the end use of the composite, and represents a

delicate balance between the lower cost of using wood versus the increased risk of wetting as the wood/plastic ratios rises. High wood contents are associated with faster water uptake because more lignocellulosic material is available for moisture sorption (Verhey et al., 2002; Clemons, 2002). The wood/plastic ratio also affects processing parameters and physico-mechanical properties of WPC's. Increased wood flour content improves flexural and tensile modulus, density, heat deflection temperature, and notched impact energy (energy required for crack propagation) (Stark and Rowlands, 2003). Increasing plastic content improves flexural and tensile strength, tensile elongation, mold shrinkage, melt flow index, and unnotched (minimum energy needed to initiate a crack) impact energy decrease (Stark and Rowlands, 2003).

1.3.1.5 Wood-polymer interface (interaction)

One key factor for producing acceptable WPC's is the interaction between the wood and the thermoplastic components (wood-polymer interface). It is difficult to achieve wood/plastic interaction because the hydrophobic thermoplastic (non-polar) and hydrophilic wood (polar) are energetically different (Wolcott, 1996; Sanadi et al., 1998). During wood/plastic mixing, the thermoplastic must first coat or spread over the wood fiber surface to interact (Wolcott, 1996).

No evidence of chemical reaction has been observed at the interface between wood and polymer; the interfacial adhesion between both materials appears to be the solely through mechanical interlocking (Sanadi et al., 1998). According to Wålinder and Gardner (2002), there are five main mechanisms of adhesion recognized in the interface between wood particles and plastic: 1) adsorption (also referred to as wetting), 2)

mechanical interlocking, 3) diffusion, 4) electrostatic forces, and 5) weak boundary layers and interfaces. These mechanisms may contribute to the intrinsic adhesion forces acting across the interface between wood fibers and plastic. Adsorption appears to be the most likely adhesion mechanism for WPC, (Wålinder and Gardner, 2002) where "adsorption" is defined as "macroscopic manifestations of molecular interaction between liquids and solids in direct contact at the interface between them" (Wålinder and Gardner, 2002). The dominance of adsorption as an adhesion mechanism helps to explain why wood particle geometry affects the wood-polymer interface.

Large wood particles tend to be associated with formation of voids at the interface between wood fiber and polymer (Mankowski and Morrell, 2000), while small particles improve the interface between wood fibers and polymer, limiting void formation on the interface, and reducing fiber-fiber surface contact (Stark and Rowlands, 2003). Large particles are less likely to be uniformly coated or wetted, leading to voids in the resulting WPC. There are few reports describing the nature of the interface between the wood particles and the plastic. According to Tze et al. (2002), the interfacial properties of cellulose fiber-polymer composites can be evaluated by the micro-Raman (Raman spectra) technique. This technique identify the strain distribution along the cellulose fiber/plastic interface by using the frequency of a cellulose mode at 895 cm^{-1} shifts in frequency with applied strain to map the local tensile strain.

1.3.1.6 Processing

Thermoplastic composite manufacturing is often a two-step process whereby the raw materials are mixed in a process called compounding, where fibers and additives

are dispersed in the molten polymer. This process may be done in either batch or continuous mixers. The molten product is then either extruded or injection- molded into its final shape (Clemons, 2002). Thermal degradation of wood during extrusion and the presence of excessive moisture in wood have major effects on subsequent WPC properties. Temperatures required for many low melting point plastics are still too high for wood, and, as a consequence, some thermal degradation of wood is expected during processing (Forest Products Laboratory, 1999).

New equipment has been developed for processing, including materials handling, drying and feeding systems, extruders, die designs, and downstream equipment (after extrusion equipment) making the manufacturing process more efficient and versatile, and improving the final quality of the resulting composite (Clemons, 2002). Typically, melt temperatures (temperature of molten material) used for processing WPC's are below 204°C (400°F). Degradation (smoke, odor, discoloration) becomes evident above this limit. In general, polyethylene-based formulations are successfully compounded at temperatures of 180°C (356 °F) or less, whereas polypropylene-based works well at temperatures near 190°C (374 °F) (English, 1996).

1.3.1.6.1 Profile extrusion

Profile extrusion is the most common process for making WPC's. The composite material is first heated so that the thermoplastic component can flow, then it is pumped and forced through a die of a given cross-sectional configuration. The material is supported as it cools, normally in a cold-water bath, and then cut to length. Pipe, tubing, furniture, moldings, and sheet goods are common products made using

profile extrusion (English et al., 1997). WPC's tend to be produced by extrusion because of the process speeds possible with this approach.

1.3.1.6.2 Injection molding

In this process, the material is heated, and pumped into a permanent mold, where it takes final shape and cools. The mold is then opened and the finished part discharged (English et al., 1997).

1.3.1.7 Physical and mechanical properties of wood plastic composites

1.3.1.7.1 Water absorption in wood plastic composites

Wood plastic composites tend to have better dimensional stability than solid wood when exposed to moisture (Clemons, 2002). WPC's with higher wood/plastic ratios (>50%) experienced water uptake when exposed to moisture sources. WPC's have a higher resistance to moisture absorption and thickness swelling (less than 1%) than do wood-based panels such as plywood and laminated veneer lumber or oriented strand board (>40%) (Falk et al., 2000). The plastic covering the wood particles in a WPC tends to reduce moisture uptake, however, a number of tests suggest that wood fiber encapsulation by the polymer is incomplete, especially near the surface. As a result, the wood component in these materials sorbs water when exposed to moisture sources (Naghipour, 1996). According to Wålinder and Gardner (2002), the wood substrate interacts with water during prolonged exposure to moisture, resulting in debonding of the wood/polymer interface by intrusion of water. Water movement through WPC's generally takes longer than through solid wood before reaching equilibrium, and cannot be directly achieved by vacuum/pressure cycles (Naghipour,

1996; Ibach and Clemons, 2002). The slow moisture uptake by WPC's creates moisture gradients between the surface and core. Apparently, moisture levels nearest the surface are more suitable for fungal growth, while moisture levels in the core are too low to support microbial activity (Wang and Morrell, 2003). This wetting pattern will ultimately affect the mode and patterns of subsequent fungal attack.

Higher levels of water uptake are associated with poor wood-fiber interfaces. Water sorption by WPC's can severely weaken wood adhesion to thermoplastic matrices, decreasing the mechanical properties of the WPC (Uerkanrak, 2001).

Swelling by moisture sorption of WPC's exposed in outside environments is associated with an increase in UV degradation as swelling develops new surfaces, exposing more polymer to degradation (Lange and Rowell, 1999). Moisture sorption in WPC's is associated with permanent reductions in strength and stiffness (Modulus of Elasticity (MOE) and Modulus of Rupture (MOR)), proportional to the moisture content of the wood in the composite (Rangaraj, 1999; Clemons, 2002). The processing methods and additives have a significant influence on the moisture diffusion in WPC's, and, as result, on the potential for further effects on composite properties (Rangaraj, 1999).

1.3.1.7.2 Mechanical properties

Most commercial WPC's are considerably more flexible than solid wood. WPC's creep more than solid wood, are less tough, and can handle less fatigue before failure (Rangaraj, 1999; Oskman and Clemons, 1998). The use of wood fibers as reinforcing agents rather than just as fillers, increases MOE, MOR and Ultimate Tensile

Strength (UTS) (Simonsen, 1997) and unnotched energy (Stark, 1999). These mechanical properties are strongly influenced by the amount, size and type of wood particles added to the matrix, and also by the additives incorporated during processing (Stark, 1999). Despite the potential improvements, the MOE's of most WPC's are less than half those of solid wood (English, 1996). In contrast, the tensile strength of WPC's is significantly reduced when wood fiber is added to the plastic matrix (Falk et al., 2000).

The processes used to make WPC's may influence the mechanical performance of the material. Extruded materials have higher stiffness and strength than material produced by injection molding (Rangaraj, 1999). Polypropylene blends tend to perform better than polyethylene blends (English, 1996). As noted previously, mechanical properties can be greatly improved by using additives to enhance wood/plastic adhesion (Oskman and Clemons, 1998).

1.3.1.8 Uses of wood plastic composites

The automotive industry has long used natural fibers in combination with plastics (Rowell, 1998; Clemons, 2000; Lee, 2002). Currently, WPC's are primarily used for exterior decking, windows and door framing, decorative trim, and railings (Clemons, 2000; Morton et al., 2003). Roofing, fencing, sea walls, garden structures and patio furniture are emerging applications for these composites (Morton et al., 2003). Significant markets are also emerging for railroad ties, flowerpots, furniture and marine piers (Clemons, 2000; Morton et al., 2003).

The lower creep resistance, stiffness and strength compared to solid wood and other structural materials severely limit the use of WPC's in applications that require considerable structural performance (Clemons, 2002).

1.3.2 Wood decay and related wood decay fungi

Wood is subject to decay by many microorganisms, but fungi are generally the most important degraders of this material. Fungi exhibit absorptive nutrition, releasing digestive enzymes into the external environment that break down large, relatively insoluble molecules such as carbohydrates and lignin into smaller, more soluble molecules that can be absorbed by hyphae (Alexopoulus et al., 1996). The decay rate is affected by an array of factors including the wood species, temperature, moisture content and oxygen level as well as the physiologic capabilities of the fungus. Mycologists have devised a number of laboratory procedures to study the decay process, but few can completely replicate the natural process. There are a diverse array of conditions under which fungal decay can be studied, but most fall into three broad categories: 1) immersion of wood in liquid cultures, 2) placing wood on or above fungal cultures on agar, and 3) placement of wood on soil.

1.3.2.1 Culture media for fungi

1.3.2.1.1 Liquid cultures

1.3.2.1.1.1 Static liquid cultures

Filamentous fungi grown in static liquid culture form a mat of hyphae at the surface of the culture (Carlile and Watkinson, 1994). The under-surface of the mat has access to abundant nutrients but receives little oxygen. The upper surface is exposed to

air but may be starved for nutrients. In addition, the growth is slow compared with submerged culture.

1.3.2.1.1.2 Rotary shaker liquid cultures

Filamentous fungi, grown in agitated liquid medium tend to produce submerged growth (Carlile and Watkinson, 1994). Fungi with septate hyphae grow well in submerged culture, but growth of the non-septate lower fungi is often reduced (Carlile and Watkinson, 1994). Considerable shearing forces may be generated within the hyphae in shaken cultures. This can improve the growth of higher fungi, probably due to the plugging of septal pores that limits cell damage (Carlile and Watkinson, 1994). Filamentous fungi may grow as nearly homogeneous suspensions of hyphae or as discrete pellets (Carlile and Watkinson, 1994).

1.3.2.2 Fungal growth in agar media

For general laboratory purposes, fungi are usually grown and maintained in agar media. Some fungi that grow rapidly in submerged culture grow slowly on agar, or vice versa (Carlile and Watkinson, 1994). Malt extract agar (MEA) or potato dextrose agar (PDA) are two of the more common media for growing wood decay fungi. Both are classified as undefined media that supply glucose and vitamins (Zabel and Morrell, 1992). The concentration of media can have major effects on both the growth rate and the ability of the fungus to produce wood degrading enzymes (Carlile and Watkinson, 1994).

1.3.3 Methods for testing wood resistance to fungal attack

1.3.3.1 Agar tests

1.3.3.1.1 Petri plates for screening tests

Preservative effectiveness against wood decay fungi can be screened initially in petri plates containing agar amended with a range of concentrations of a candidate fungicide. This method is most suited to testing water-soluble chemicals that can be readily incorporated into a simple nutrient agar and dispensed into petri dishes (Hilditch and Hambling, 1971; Behr, 1973; Zabel and Morrell, 1992).

Major advantages of the agar method are its low cost, easy set-up, and the relatively short time required for its completion (Baechler, 1949). The main drawback to the agar method is that the substrate is not wood. The fungal growth on artificial media is markedly different from growth in wood. For example, the addition of excess sugar can largely negate the protective value of some preservatives (Zabel and Morrell, 1992). Nevertheless, the agar test has been widely used and is one of the standard tests for assessing fungal toxicity of many chemicals (Behr, 1973).

1.3.3.1.2 Agar tests for assessing wood decay

The agar block test is another important method for assessing wood decay. The agar method was adopted in Europe in 1930, and was standardized in Germany in 1939 by Deutsches Institut für Normung (Hof, 1962). Currently, the European Norms (EN) EN 113 is the standard of The European Committee for Standardization (CEN) used for determination of toxic values of wood preservatives against wood destroying basidiomycetes cultured on agar medium (CEN, 1996). The test fungus is grown on sterile malt agar in a kolle flask until the medium is well covered with mycelium. Oven-dried wood blocks (one treated, one untreated) are placed on glass rods over the

mycelium, and then incubated for 12 to 16 weeks (Behr, 1973). Weight loss and visual examination, both naked eye and with magnification, are used to assess chemical efficacy (Hof, 1962, Eaton and Hale, 1993).

1.3.3.2 Soil block tests

The soil block test is the other important method for testing resistance to fungal attack. This method is widely used in the United States. Work done at the Forest Products Laboratory in the early 1920's led to the development and standardization of a short-term laboratory soil-block test method that served as a guide to long term performance of wood preservatives (Baechler and Gjovik, 1986). This method permitted rapid screening of candidate wood preservatives and allowed the comparison of different preservatives (Baechler and Gjovik, 1986). The soil block test was developed initially by Leutriz in 1946 (cited in Richards, 1947), and later modified and improved by Duncan and Richards, (1950; 1951a; 1951b).

Soil block tests are described in the ASTM standards, designations ASTM D 1413 – 76 and ASTM D 2017 (ASTM, 1999), as well as in American Wood Preservers Association (AWPA), designation E10-91 (AWPA, 1998). The standards have slight procedural variations from one another. In the soil-block test, oven-dried wood blocks are exposed to wood feeders strips colonized by a single decay fungus that is in direct soil contact in glass jars or plastic chambers (Eaton and Hale, 1993). The blocks are exposed for 12 to 16 weeks, then removed from the incubation chambers, oven-dried, and reweighed. The weight loss after the incubation period is used as the measure of resistance to fungal attack. The results are compared to those obtained from control

blocks not exposed to the test fungi (Zabel and Morrell, 1992). In the soil block test, a key issue is the moisture holding capacity of the soil. Accurate control of the soil moisture content is extremely difficult, and wide variations between soils can affect the decay process (Goodell et al., 2003).

The physical and chemical properties of soils are important factors to consider during testing. The results obtained on soil tests are strongly influenced by soil type and soil physical and chemical properties. According to Barr (1977), moisture content, pH, and temperature of the soil influence the decay process. Some soil types favor brown-rot fungi and others white-rot fungi, while garden compost favors soft-rot fungi (Goodell et al., 2003). In addition, the fungus strains can vary and may influence extent of decay. All these factors make it difficult to compare results among laboratories.

1.3.3.3 Vermiculite buried tests

The soil block and agar block tests both expose wood above the soil, and are not suitable for assessing soft rot attack, which requires higher moisture levels. The higher moisture contents required by these fungi are usually achieved by burying the test samples in either very moist vermiculite or mixtures of soil and compost. These methods are generally not suitable for basidiomycetes because the moisture conditions are unsuitable (too high) for fungal growth (Zabel and Morrell, 1992). Vermiculite is inexpensive and has been used as a substrate for testing the decay capability of soft-rot fungi (Zabel and Morrell, 1992).

1.3.3.4 Comparison of soil block, agar, and vermiculite burial tests

1.3.3.4.1 Soil block versus agar tests

A number of studies have compared agar and soil tests for assessing the effectiveness of wood preservatives against fungal attack (i.e., Richards and Addoms, 1947, Sexton et al., 1993/1994). The main goal of these studies was to establish a standard laboratory technique for determining relative preservative threshold values in comparatively short times (Richards and Addoms, 1947). Neither test is completely satisfactory, but both provide simple, rapid methods for assessing durability within certain limits. The agar method is easily set up, but special care must be taken to avoid direct exposure of the wood to the agar, to avoid excessive water sorption. The standardization of the soil, the selection of the test fungi, and the soil/wood moisture relations are major concerns for the soil block test. The soil block test is widely used in North America while the agar block is used in Europe.

1.3.3.4.2 Comparison of agar versus vermiculite burial tests

In a study that compared agar exposure and vermiculite burial methods for preparing basidiomycete-colonized wood, the vermiculite method was more effective, required fewer manipulations and permitted colonization of wood without the development of moisture gradients formed when agar was used (Sexton et al., 1993/1994). The vermiculite method also produced more uniform moisture distribution, and allowed many specimens to be inoculated per bag, reducing time and storage space.

1.3.3.5 Validity of laboratory mycological tests

Considerable debate exists concerning the ability of laboratory decay tests to predict field performance (Zabel and Morrell, 1992; Pendleton et al., 2002). In addition

to fundamental problems associated with reproducibility of natural environments, there is the age-old question of how to relate exposure to a single fungus under ideal conditions to field exposures with varying conditions and an array of organisms. The soil block and agar methods have a number of limitations, the most important of which are:

1) that the simplified biological environment fails to simulate the complexity of the real natural environment with its changing and interacting microflora and
2) aging of the preservative cannot be accurately reproduced by accelerated laboratory tests (Hilditch and Hambling, 1971; Baker, 1974).

Despite these disadvantages, laboratory tests can be used as indicators of decay susceptibility that must be confirmed by subsequent field tests.

1.3.4 Decay of wood plastic composites

The resistance of conventional plastics to fungal attack is due primarily to the low surface area and relative impermeability of plastic, and the very high molecular weight of plastic material (Naghipour, 1996). Microorganisms tend to attack the ends of large molecules. Because the number of the ends is inversely proportional to the molecular weight, it would be necessary to break large molecules into very small fragments with a large surface area in order to make plastic degradable (Naghipour, 1996). In addition, the fungi tend to lack enzymes capable of degrading these materials. Polyethylene, polystyrene, and polyvinyl chloride were not susceptible to fungal attack (Naghipour, 1996).

The perceived resistance of WPC's to fungal attack is based on the belief that plastic encapsulates the individual wood fibers in a continuous plastic matrix that acts as a barrier to protect wood fibers from wetting. However, a number of tests suggest that wood fiber is not completely encapsulated by the polymer, especially near the surface. As a result, the wood component in these materials does reach moisture levels suitable for fungal attack (>30%) (Naghipour, 1996; Mankowski and Morrell, 2000; Clemons, 2002). For example, WPC specimens exposed for 16 weeks to decay fungi in laboratory tests experienced weight loss exceeding 40% (Verhey et al., 2002). Morris and Cooper (1998) found WPC's manufactured from recycled wood and plastic that were attacked by brown rot, white-rot and blue stain fungi in the Everglades National Park, Florida, USA. According to Naghipour (1996), the brown-rot fungus *Gloeophyllum trabeum* was able to grow on WPC samples, whereas pure polyethylene and polypropylene were immune to fungal attack. Weight losses were less than 5% at the 60% wood level while WPC's with wood levels of 50% or less showed good resistance to fungal attack (Naghipour, 1996).

1.3.4.1 Decay mechanisms in wood plastic composites

Decay activity in WPC's is concentrated on the exterior surfaces of the composite, resulting in gradual roughening of the composite surface (Pendleton et al., 2002). Breakdown on the polymer surface leads to more wood particles being exposed, thereby increasing moisture uptake (Peyer and Wolcott, 2002). Surface wood particles exposed directly to fungal attack are generally totally decayed. This decay mechanism

is similar to that observed for microbial degradation of polyethylene-starch composites (Pendleton et al., 2002).

Microscopic observations of decayed WPC specimens showed mycelium concentrated in the interfacial voids between the wood and the thermoplastic component. This observation supported the premise that the primary mode of fungal degradation was via hyphal penetration through the voids on the wood/polymer interfaces (Mankowski and Morrell, 2000; Pendleton et al., 2002). The materials used in these studies tended to have large wood particles, which resulted in lesser wood/plastic adhesion and more voids for fungal entry.

1.3.4.2 Methods for assessing decay of wood plastic composites

Investigations of WPC's have focused largely on improving mechanical and other material properties. Despite the claims of decay resistance, few scientific studies address accelerated biodegradation of WPC's (Naghipour, 1996; Pendleton et al., 2002; Mankowski and Morrell, 2000; Verhey et al., 2001; Clemons, 2002; Simonsen et al., 2002; Ibach and Clemons, 2002; Ibach et al., 2003).

There are currently no standards for assessing the durability of WPC's in either controlled laboratory experiments or in field exposure. As a result, WPC durability has been assessed using solid wood methods such as the modified soil block tests (Mankowski and Morrell, 2000; Verhey et al., 2001; Pendleton et al., 2002; Clemons and Ibach, 2002) or agar tests (Naghipour, 1996). Weight loss has been the main assessment parameter, but mechanical properties have also been used to assess fungal attack. Both laboratory methods are characterized by a slow initial fungal colonization

followed by an increasing rate of decay. The initial lag is partially related to moisture since both methods are designed to allow blocks to reach 60% to 80% (based on the oven dry weight of the wood). These moisture levels are needed by microorganisms to attack wood, but neither of these methods produce adequate weight losses on WPC's. The relatively slow rate of decay is probably a reflection of the slow rate of moisture absorption by the WPC's.

The inadequacy of traditional tests for assessing decay of WPC has encouraged a search for alternative methods. Verhey et al. (2003) used strength loss instead of weight loss in a field test and showed that MOR decreased with exposure time, but this decrease was totally attributed to moisture uptake by WPC's from soil. Despite ideal moisture levels for fungal attack (nearly 60%), no evidence of decay was detected on WPC stakes (Verhey et al., 2003). Clemons (2002), Verhey et al. (2003), and Ibach et al. (2003) used MOE and MOR to assess decay, but it was very difficult to separate the irreversible effect of water absorption from any fungal attack. Weathering of WPC specimens prior to fungal exposure may be one method for increasing moisture uptake (Peyer and Wolcott, 2002). Ibach et al. (2003) used a preconditioning laboratory method using combinations of UV light and water exposure for testing the durability of WPC's. Moisture contents of WPC specimens preconditioned in water prior to fungal attack reached nearly 14% moisture content, where, according to Ibach et al. (2003), decay can potentially occur.

The relevance of accelerated laboratory tests to predict field performance of WPC is the subject of much debate, because laboratory tests fail to simulate the

complexity of the natural environment with its varying conditions and interacting microflora (Hilditch and Hambling, 1971; Baker, 1974). Accelerated laboratory tests can be more severe than most aboveground field exposures tests. Pendleton et al. (2002) reported that laboratory-based procedures such as the soil block tests were less suitable for predicting the performance of WPC's. Pendleton et al. (2002) suggested that solid wood test procedures can be used with minor modifications. The primary concerns are the differences between solid wood and composites with regard to decay susceptibility, moisture gradients, and the increased surface area to volume ratio of composite panel products (Falk, 2000).

The absence of applicable test methods has led the American Society for Testing and Materials (ASTM) to create a subcommittee to develop standards for assessing durability and performance of WPC's used in decking (ASTM, 2003). Before developing a new method, it is helpful to examine the properties of the test material with regard to the conditions presented by the test methodologies. For simplicity, it can be assumed that conditions in the current methodologies are suitable for growth of the test organisms on either or agar or wood feeder strips. New organisms could be considered, but there is little data suggesting that the fungi able to decay these materials will differ markedly from those found on other wood-based materials.

1.3.4.3 Factors that affect decay of wood plastic composites

1.3.4.3.1 Moisture

Moisture is essential for fungal colonization and decay of lignocellulosic materials. Water absorption on the surface is the key parameter because this is where

fungal attack is initiated (Naghipour, 1996). Wood plastic composites with high wood contents (>50%) clearly absorb water (Naghipour, 1996; Mankowski and Morrell, 2000). Naghipour (1996) showed that WPC's had slower moisture uptake then plastic composites, but were permeable and, as a consequence, subject to fungal decay, particularly at high wood–polymer ratios (> 50% wood). Polyethylene composites absorbed more water than those made with polypropylene at comparable wood/plastic ratios (Naghipour, 1996). Surface deterioration and delamination in the WPC's have been associated with weathering (Naghipour, 1996; Peyer and Wolcott, 2002).

Moisture sorption can lead to void formation at wood/polymer interface (Peyer and Wolcott, 2002). The voids and cracks that are present before water exposure will expand after exposure. These voids could create pathways for entry by water and fungal hyphae. Short-term boiling could be used to rapidly increase moisture absorption (Naghipour, 1996; Ibach and Clemons, 2002), however, care would need to be taken to avoid altering the wood/plastic interface or leaching any fungicides. Ibach and Clemons (2002) reported that chemical modification of the wood cell wall would reduce WPC moisture uptake below the level required for fungal attack. They evaluated the resistance of WPC's made with chemically modified fiber or flour and polypropylene to fungal attack. Overall weight losses were consistent with the lower moisture sorption of the composite. According to Ibach et al. (2003), moisture contents of WPC's above 15% led to significant weight losses, but these levels must be viewed cautiously because they do not appear to be favorable for inducing fungal attack. Wang and Morrell (2003) exposed samples to water for long periods and found that conditions on the surfaces of

commercial WPC's were suitable for fungal attack, while moisture levels 5 mm below the surface had changed only slightly. Clearly, test specimen sizes that maximize surface to volume ratios will result in conditions more suitable for decay development.

1.3.4.3.2 Wood particle size

Wood plastic composites containing large particles tend to experience more severe decay at similar wood ratios (>50% wood) (Verhey et al., 2002). Large wood particles create more pathways into the plastic matrix, exposing more surface area to water and fungal hyphae (Miller, 2001). Simonsen et al. (2002) noted similar effects using polyethylene and polypropylene. While small particles are preferable for slow moisture uptake, they are more costly to produce and therefore increase the final cost of the product.

1.3.4.3.3 Wood/plastic ratios

Mankowski and Morrell (2000) examined the influence of wood/plastic ratio and wood particle size on decay of commercial WPC's made with pine and high-density polyethylene. A 20% wood weight loss was observed in the WPC's made with 70% wood (small wood particles) after exposure to a brown-rot fungus. In contrast, little or no degradation was observed in two samples of WPC's made with 50% wood content, despite the use of larger wood particles. These results suggest that amount rather than size of the wood particles may have a greater effect on WPC durability (Stark and Berger, 1997; Pendleton et al. 2002).

1.3.4.3.4 Wood/polymer interface

Differences between the hydrophilic wood and the hydrophobic thermoplastic during processing can limit bond development, resulting in a poor adhesion between wood fibers and the plastic polymer. Without chemical or physical bonding, failures on the wood-polymer interface and interfacial voids can develop due to poor processing or as a result of external factors, such as moisture uptake or UV degradation. These failures in the wood-polymer interface are of critical importance for assessing WPC performance (Pendleton et al., 2002). Nearly all WPC's contain additives designed to produce some chemical interaction at the wood/plastic interface, but most are proprietary, and their effects on decay resistance of the final product are poorly understood.

1.3.4.3.5 Biocides

Reports of field decay of WPC's encouraged the incorporation of biocides into WPC's. The most common biocide additive is zinc borate because it is inexpensive, somewhat leach resistant and stable at elevated temperatures (Simonsen et al., 2002).

Verhey et al. (2001) evaluated the effectiveness of zinc borate against decay fungi for wood fiber/polypropylene composites. Incorporation of zinc borate (1%, 3% or 5%) into the composite provided good protection from fungal attack at any tested concentration. Simonsen et al. (2002) observed similar results in WPC's exposed to *G. trabeum*.

1.3.5 Challenges for testing durability of wood plastic composites

Whereas there is much work emerging on assessing the durability of WPC's, the methods remain less than ideal. The industry has the potential to produce an array of

WPC's with properties tailored to meet specific use conditions. However, these developments are limited by the slow rate of biological testing. For example, process laboratories capable of producing fifty test materials per day must wait 4 to 6 months to learn if these materials are durable. Clearly, methods must be developed or refined that accelerate both moisture uptake and decay potential.

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Chapter 2

Effect of fungal attack on creep behavior and strength of wood plastic composites

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2.1 Abstract

This study examined the effect of fungal attack on modulus of elasticity (MOE), modulus of rupture (MOR), and creep of zinc borate treated and untreated WPC's. WPC beams were exposed in vermiculite to the brown rot fungi *Gloeophyllum trabeum* and *Postia placenta* for 4 to 24 weeks.

Treated and untreated WPC beams experienced little decay degradation of wood particles on the WPC surface.

MOE, MOR, and creep were poor parameters for estimating early stages of WPC's decay. The influence of moisture content on MOE, MOR, and creep confounded the early effect of fungal attack.

Scanning electron microscopy indicated that all wood particles on the surface of untreated composites were totally degraded by both fungi. Zinc borate treated beams did not experience decay. The results confirm the susceptibility of untreated WPC's to fungal attack.

2.2 Introduction

Wood-plastic composites (WPC) are an increasingly attractive option for homeowners seeking durable, maintenance-free decking. These materials are also extending their markets into other applications where wetting is likely (Sellers et al., 2000). Initially, WPC manufacturers believed that these materials were relatively immune to biological attack since the plastic largely encapsulated the wood particles, limiting their ability to absorb moisture and thereby reducing the likelihood that

conditions would support fungal attack. Whereas WPC's absorb moisture more slowly than solid wood, moisture can eventually move into these materials at levels that support biological growth (Naghipour, 1996; Schmidt, 1993). Subsequent reports have shown that fungi can invade WPC's to produce fructifications on deck surfaces and mass loss of the deck (Morris and Cooper, 1998).

Manufacturers have responded to the risk of fungal attack by incorporating zinc borate into the furnish prior to extrusion (Wolcott and Englund, 1999). Zinc borate has become the dominant material used for this purpose because of its low water solubility, ability to withstand common extrusion temperatures and low cost.

The performance of WPC's under decay-conducive exposures has usually been assessed using mass loss following exposure to a single decay fungus under ideal decay conditions. While weight loss is a useful measure, it may mask the more subtle effects of fungal attack on WPC properties, particularly those effects related to bonding between wood and plastic. Wood is added to WPC's because it enhances the stiffness of the final products (Simonsen, 1997). While the exact nature of the wood/plastic interface still remains under study, subtle decay-induced changes in this interface could have dramatic impacts on physical properties, even at relatively low mass losses. As a result, mass loss may be a poor indicator of preservative efficacy in WPC's. Instead, testing mechanical properties such as creep and modulus of elasticity may be more suitable measures of fungal effects.

2.3 Objective

The objective of these tests was to evaluate the effects of fungal attack on creep behavior and bending properties of WPC's with or without a preservative.

2.4 Materials and methods

2.4.1 WPC's samples

A wood plastic composite composed of approximately 60% virgin southern pine and 40% high density polyethylene (HPDE) with or without 1.25% zinc borate was supplied in hollow core extruded planks (37.5 mm thick by 125 mm wide) of varying lengths. The planks were cut into beams measuring 8.5 x 8.5 x 136 mm long with the long direction following the extrusion direction. The outer layer of the original material, which tended to be very smooth as a result of the extrusion process, was removed during sawing.

The beams were oven dried at 49 °C and weighed prior to being submerged in water and subjected to a 30-minute vacuum followed by a 2-hour pressure period at 800 kPa. This process produced less than 2% weight gain in the beams. The beams were then tested in third point loading to approximately 30% of their maximum load (as determined by initial destructive tests of beams) and the resulting load-deformation curve was used to calculate modulus of elasticity. The beams were then sterilized by exposure to 2.5 mrads of ionizing radiation from a cobalt 60 source.

2.4.2 Test fungi

The test fungi evaluated were *Postia placenta* (Fries) M. Larsen et Lombard

(Isolate Mad 698) and *Gloeophyllum trabeum* (Pers. Ex Fr.) Murr (Isolate Mad 617), both of which cause brown rot and are commonly isolated from a variety of building components.

2.4.3 Standard procedures and methods

The fungi were grown on malt extract agar until needed. Small agar plugs (3 mm diameter) were cut from the actively growing edge of the fungus and added to flasks containing 1% malt extract. The flasks were incubated in stationary culture for 11 days, and then the mycelium was collected by filtration, washed with sterile distilled water and resuspended in distilled water before being blended for 10 seconds to fragment the fungal mycelium. The inoculum was diluted to 275 ml prior to use.

The decay tests were adapted from procedures described by Curling et al. (2000). Rubbermaid TM Space Saving containers (222 by 211 by 120 cm high) were washed with hot soapy water then sprayed four times with 95% ethanol. The boxes were dried in a laminar flow hood between sprays. Two holes (13 mm diameter) were drilled into the tight-fitting plastic covers and plugged with sterile cotton. These holes allowed for air exchange in the chambers. Eight hundred sixty grams of 0.5 % malt extract solution was added to 220 g of vermiculite in an autoclavable plastic bag. The bags were loosely sealed and autoclaved for 20 minutes at 121 °C. The contents of one plastic bag were placed into each incubation chamber and mounded into a ridge down the center using a sterile spatula. Ten sterile beams of one of the treatment groups (with or without zinc borate) were placed in the mounded vermiculite so that the center third

of each was buried, but the ends protruded. Twenty-five ml of the fungal inoculum was then poured over the vermiculite. The chambers were sealed and incubated at 20° C and 70% relative humidity.

Beam condition was assessed 4, 10, and 15 weeks after inoculation. At each interval, one chamber from each treatment was harvested to reduce the risk of contamination through repeated manipulation of individual chambers. Ten beams from each treatment combination were removed, scraped clean of adhering mycelium and vermiculite and weighed to determine beam moisture content. Five beams were then tested to failure in third-pointing loading according to procedures outlined in American Society for Testing and Materials Standard D 47610-96. (ASTM, 2000a). Creep tests were performed on the remaining five beams using a procedure developed by Xu et al. (2000) wherein the beams were suspended between two metal bars to produce an effective span of 122 mm. A mono-filament string was used to suspend a 1282 g load. The load was determined using the values developed in the third point load tests to calculate modulus of rupture (MOR) and modulus of elasticity (MOE). Long term deflection was measured using digital calipers 1, 6, 12, 30, 60, and 120 minutes after the load was applied, then after 5, 20, 50, 100, 200, 500, 700, and 1000 hours following the time series proscribed in ASTM Standard D6112-97 (ASTM, 2000b). Following testing, the beams were oven-dried and weighed to determine mass loss over the fungal exposure period.

We also exposed one set of sterile beams in sterile vermiculite for 4 weeks to

study moisture uptake and to assess the effect of increased moisture content on beam bending properties. These air-drier samples (approximately 2% moisture content) were exposed in sterile vermiculite and were removed after 2 and 4 weeks. Each beam was weighed, and then subjected to three-point loading according the procedures described above, to approximately 30% of the maximum load and returned to the exposure chamber. These values were used to calculate MOE.

The patterns of fungal attack were assessed by cutting 5-mm-thick cross-sections from the center of each beam. The beam cross-sections were surfaced using a sliding microtome, then sputter-coated with carbon prior to being examined using an AMR 1000A Scanning Electron Microscope (SEM) at an accelerating voltage of 20 kV, tilt angle of 30°, and a working distance of 12 mm.

The bending test data were subjected to an analysis of variance to test for the effect of chemical treatment and fungal species on bending properties over time.

2.5 Results and discussion

2.5.1 Moisture uptake

Beams subjected to the initial 2 hour pressure treatment absorbed little moisture (average 1.6% moisture content), but those exposed in moist vermiculite appeared to steadily absorb moisture over the first four weeks of exposure, then stabilized at 15 to 18% (Table 2.1). While this figure implies an MC that is too low for fungal attack (assumed to be approximately 30% for most fungi; Zabel and Morrell, 1992), wood represents approximately 60% of the material. If we assume minimal moisture sorption

by the HDPE, then wood moisture contents would be between 25 and 28%, assuming uniform moisture distribution. There appeared, however, to be a noticeable moisture gradient from the surface to the interior, resulting in more suitable growing conditions nearer the surface.

2.5.2 Weight loss

Weight losses in all beams were relatively low (<3.5%) over the course of the test (Table 2.1). Even when only the percentage of wood in the WPC is used to calculate the weight loss, the highest percentage weight loss was still only 5.7 %. These values must also be considered in terms of the small areas exposed to fungal attack. Only one third of the beam length was exposed directly to vermiculite and weight losses would be largely confined to this center zone of the beam.

2.5.3 Modulus of elasticity (MOE)

Modulus of elasticity of all beams declined precipitously between their initial and final testing, but most of this effect was the result of moisture increases. The MOE's of treated beams were initially higher than those for the untreated control and remained so after 4 weeks. The MOE's of beams exposed to *P. placenta* increased slightly between 4 and 15 weeks suggesting that the fungus had little effect on the beam. The MOE's of beams exposed to *G. trabeum* decreased approximately 10% between 4 and 10 weeks suggesting that this fungus may have begun to permanently affect material properties. MOE's for this treatment combination increased after 15 weeks of exposure, but the levels were still below those for the 4 week sample. While

moisture content was a confounding variable in this test, it would appear that *G. trabeum* was beginning to affect composite properties.

2.5.4 Modulus of rupture (MOR)

Modulus of rupture of all treatments declined markedly between initial dry and the one-month wet testing. This decline reflects the effect of moisture uptake on bending properties (Table 2.1). The inability to effectively wet the specimens prior to exposure largely precluded a direct comparison between the initial sample and those exposed for 4 to 15 weeks in the moist vermiculite; but progressive MOE tests of sterile beams in this environment showed that the primary effect on bending was moisture related for all treatments (Table 2.1).

Modulus of rupture of all treatments except the *G. trabeum* 10-week exposure averaged between 13.1 and 14.1 N/mm^2. MORs of beams in some treatments appeared to decline between 4 and 10 weeks, but increased after 15 weeks. We believe this fluctuation reflects the inherent variation in MOR among individual pieces rather than any biological effect. The absence of any immediate fungal effect on MOR probably reflects, in part, the inability of the fungus to significantly affect the plastic.

2.5.5 Creep test

Deformation of all materials steadily increased over time under load (Figure 2.1). The most rapid rates of deformation were found with untreated beams exposed to either test fungus, which differed little from one another. Deflection of treated beams varied only slightly from one another. Beams exposed to *G. trabeum* deformed slightly

more than those exposed to *P. placenta* suggesting that *G. trabeum* was beginning to affect creep behavior; however, considerably longer exposures to the test fungus would be required to better define fungal effects on material properties.

Non-fungal exposed control beams initially deformed in a manner similar to the treated, fungal exposed materials. Over time, however, deformation increased to levels that were slightly below those found with *P. placenta* exposed beams (Figure 2.1). The lack of effect on MOE by *P. placenta* suggests that the fungi are producing gradual changes in deformation that reflect the preponderance of attack near the beam surfaces. These results suggest that further studies on creep behavior are needed, particularly given the importance of the wood components in WPC performance.

2.5.6 Scanning electron microscopy (SEM)

Scanning electron microscopy examination of cross-sections revealed little evidence of substantial fungal penetration into the beams (Figure 2.2). Previous studies suggested that fungi were capable of substantial growth through wood particles distributed within the plastic matrix (Mankowski and Morrell, 2000); however, the wood particle sizes in the current study were far smaller and appeared to limit fungal attack. Instead, we noted a gradual roughening of the beam surfaces, particularly in untreated beams, suggesting that the fungal enzymes were acting primarily on wood particles near the surface. As these particles degraded, the plastic surrounding them appeared to flake away. Continued degradation of this nature would gradually erode the material surface much like conventional Type 2 soft rot (erosion) attack of solid wood

Table 2.1 Effect of exposure in a vermiculite decay system on moisture content, weight loss, MOE and MOR of wood plastic composite beams exposed to *G. trabeum* or *P. placenta* for 4 to 30 weeks.

Treatment	Fungus	Exposure period (wks)	Final Beam MC[1] (%)	Weight Loss[1] (%)	MOE[1] (N/mm^2)	MOR[1] (N/mm^2)
None	None	0	1.6 (0.1) K	0.0 M	2675 (355) A	18.6 (1.5) A
None	*G. trabeum*	4	15.7 (0.3) I	1.5 (0.1) IJKL	1506 (127) FGH	13.4 (0.8) CDEF
		10	15.9 (0.4) I	2.8 (0.4) C	1365 (69) H	12.6 (0.4) EF
		15	18.2 (0.3) C	3.4 (0.0) B	1470 (60) GH	13.3 (0.5) BCDE
		22	17.3 (0.3) E	2.0 (0.6) D	1614 (83) DEFG	13.6 (0.4) BCD
		30	20.0 (0.2) A	5.0 (0.5) A	1373 (111) H	12.3 (0.8) F
	P. placenta	4	15.3 (0.2) J	1.3 (0.1) L	1547 (145) EFGH	13.5 (0.8) BCDE
		10	16.5 0.2) HG	1.7 (0.0) HIJK	1603 (71) EFGH	13.4 (0.4) BCDE
		15	16.7 (0.2) FG	1.7 (0.1) HIJ	1696 (21) CDEF	13.9 (0.3) BC
		22	17.0 (0.2) F	1.8 (0.1) GHI	1622 (133) DEFG	13.3 (0.8) BCDE
		30	17.3 (0.2) E	1.9 (0.0) DEFGH	1605 (129) DEFG	12.9 (0.6) DEF
Zinc	*G. trabeum*	4	16.5 (0.2) HG	1.4 (0.0) JKL	1710 (148) CDE	13.1 (0.5) BCDE
borate		10	16.9 (0.2) F	1.8 (0.1) EFGH	1843 (69) BC	13.7 (0.5) BCD
		15	18.1 (0.3) C	1.9 (0.1) EFGH	1971 (165) BC	14.2 (0.6) B
		22	18.2 (0.1) C	2.1 (0.1) DEF	1896 (139) BC	13.9 (0.7) BC
		30	18.6 (0.2) B	2.1 (0.1) DE	1874 (189) BC	13.8 (0.5) BCD
	P. placenta	4	16.4 (0.2) H	1.4 (0.0) KL	1779 (167) BCD	13.5 (0.8) BCDE
		10	17.5 (0.1) DE	1.8 (0.1) FGH	1877 (161) BC	14.1 (0.8) BC
		15	18.1 (0.2)	1.7 (0.1) HI	1856 (13) BC	13.9 (0.6) BC
		22	17.7 (0.2) D	1.9 (0.1) DEFGH	1834 (68) BC	13.6 (0.4) BC
		30	18.5 (0.3) B	2.1 (0.1) DEFG	1845 (138) BC	12.3 (0.6) BCDE

[1] Values represent means of 5 replicates per treatment. Figures in parentheses represent one standard deviation. Values followed by the same letter(s) do not differ significantly by Duncan's multiple-range test ($\alpha = 0.5$). Moisture content and mass loss were calculated based on the total weight of the WPC beam not on a wood basis.

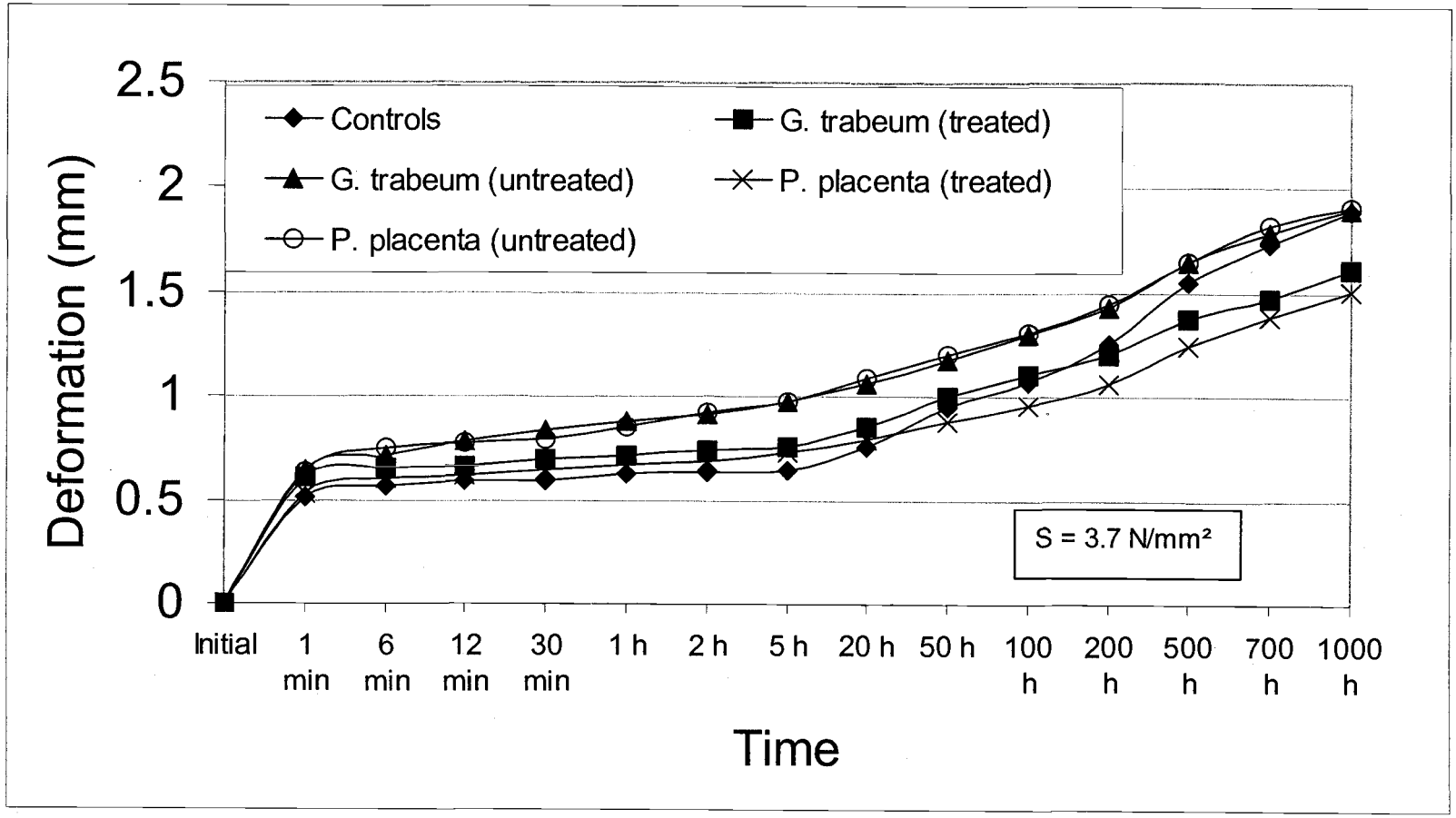

Figure 2.1 Deformation of untreated and zinc borate treated wood-plastic composites beams exposed to *G. trabeum* or *P. placenta* for 4 to 10 weeks.

(Morrell, 1981). This attack pattern is probably not a concern over much of an exposed composite surface except where the WPC creates a water-trapping joint or is in direct soil contact.

2.6 Conclusions

The presence of zinc borate markedly reduced the potential for fungal attack of the WPC tested. In the absence of zinc borate, *G. trabeum* reduced both MOE and creep properties after only 4 weeks of exposure, while *P. placenta* affected only creep behavior. The ability of the decay fungi to cause substantial effects on material properties over relatively short exposure periods suggests that treatment is essential for adequate performance under adverse conditions.

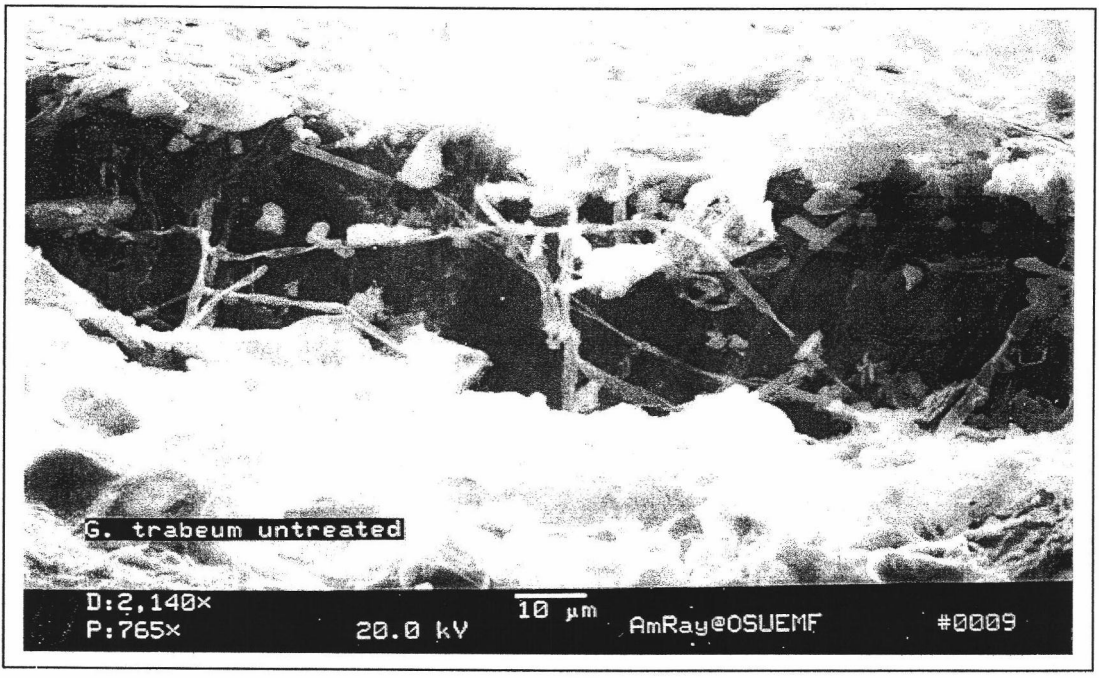

Figure 2.2 SEM of untreated wood-plastic composite beams exposed to *G. trabeum* for 10 weeks showing extensive fungal growth on the WPC surface as a result of fungal attack (magnification = 765x).

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Chapter 3

Influence of culture media on tests for assessing the decay resistance of wood plastic composites (WPC's)

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3.1 Abstract

The ability of malt extract agar (MEA), potato dextrose agar (PDA), amended basal salts, malt extract broth, sawdust (maple or red alder), vermiculite and soil to enhance fungal attack of wood plastic composites (WPC's) was examined. Moisture content and weight loss were used to assess the extent of decay of WPC's. The results were compared with a modified traditional soil block test.

Rates of decay in WPC specimens exposed in agar were greater than in those exposed in the soil block tests. Both MEA and PDA enhanced decay of WPC's. PDA was a better media for *Trametes versicolor*, while MEA was a better media for *Gloeophyllum trabeum*. Both PDA and MEA were suitable media for *Postia placenta*.

A strong relationship was found between weight loss and moisture content; WPC specimens that experienced severe decay also exhibited high moisture content. The results indicate that WPC decay can be accelerated using both agar and soil as media. The conditions that faster moisture uptake must be included in any test methodology.

3.2 Introduction

The demand for wood/plastic composite (WPC) products has steadily grown, largely due to their reputation for high decay resistance and low-maintenance. However, reports of decay in some WPC's have motivated a search for methods to assess decay resistance of these materials.

The lack of a standard test method for assessing the decay resistance of WPC's has resulted in the use of modified soil block and agar tests to assess fungal resistance

of these materials. While these tests produce weight losses, the levels of decay remain far below those found with solid wood. The low weight loss is likely due to the slow rate of wetting and inherent decay resistance of these composites. As a result, decay tests of WPC's require longer exposure periods than solid wood. These factors make the standard American Society for Testing and Materials (ASTM, 1999) or American Wood Preservers Association (AWPA, 1998) soil block tests less suitable for predicting WPC performance than they are for testing solid wood.

This chapter describes a series of three tests to assess the decay resistance of WPC's in comparison with traditional soil block tests.

1) *Screening tests*: These tests evaluated the ability of selected fungal species to enhance decay of WPC's in different types of culture media. The media tested were: agar (malt agar extract (MEA) and basal salts amended), sawdust (maple and red alder), soil (direct exposure and sandwich system), vermiculite, and liquid (stationary and rotary shaker).

2) *Optimization tests*: The most promising combinations from the screening tests were examined in a second series of tests using a range of MEA and potato dextrose agar (PDA) concentrations.

3) *Soil block tests*: Modified standard ASTM soil block tests (ASTM, 1998) were performed to provide a baseline against which to measure the decay results obtained with the screening and optimization tests.

Finding suitable conditions that provide nutrients and moisture for fungal growth and colonization of WPC's would greatly facilitate the development of WPC's.

3.3 Objective

The objective of this research was to find one or more culture media suitable for accelerating decay of WPC's.

3.4 Materials and methods

Unless otherwise noted, the section 3.4.1, 3.4.2, and 3.4.3 apply to all the experiments.

3.4.1 WPC's and wood samples

Wood/plastic pellets used to make WPC samples contained 60% sugar maple (*Acer saccharum*) (ground to pass a 80 mesh screen) and 40% polypropylene. Samples were made using a 150 x 150 x 0.5 mm mold. The mold containing the pellets was heated to nearly 180 °C (350 °F), and pressed for 10 minutes at 1500 KPa, then cooled at room temperature to about 100 °C (180 °F). The resulting samples were cut to 10 x 20 x 0.5 mm. Sugar maple (*Acer saccharum*) wafers, cut to the same size, were used as controls to ensure that conditions in the incubation chambers were suitable for fungal decay.

All samples (WPC's and maple wafers) were oven-dried at 104 °C for 24 hours to remove water, weighed to the nearest 0.0001 grams, and submerged in distilled water for 48 hours to introduce enough water to allow fungal attack. The samples were then sterilized by heating at 121 °C for 20 minutes in an autoclave.

3.4.2 Test fungi

The brown-rot fungi *Gloeophyllum trabeum* (Pers. ex. Fr.) Murr. (Isolate Madison 617) and *Postia placenta* (Fr.) M. Larsen & Lomb. (Isolate Madison 698), and the white-rot fungus *Trametes versicolor* (L:Fr) Pilat (Isolate R105) were maintained on 1% malt extract agar (MEA) in petri dishes until needed.

3.4.3 Standard procedures and methods

3.4.3.1 Production of inoculum

Fungi were grown for 21 days either in petri dishes containing 1% MEA or in 500 ml Erlenmeyer flasks containing 150 ml of 1% malt broth. These media were inoculated with 4 mm-diameter plugs of mycelia cut from the actively growing edges of the culture (Figure 3.1). One 4 mm-diameter plug was placed in the center of the petri dish containing either MEA or amended basal salts.

The liquid fungal inoculum was prepared by placing two, 4 mm-diameter discs in Erlenmeyer flasks containing 150 ml of 1% malt broth. The liquid cultures were incubated under stationary conditions for 21 days at room temperature (22-25 °C). After incubation, the contents of the flasks containing a given fungus were poured through a sterile Buchner funnel with no filter paper. The mycelial mass caught in the funnel was resuspended in 80 ml of sterile distilled water and blended at high speed for 30 seconds. The macerated mycelia were then poured into a sterile 100 ml squeeze bottle for inoculation of either culture media or samples.

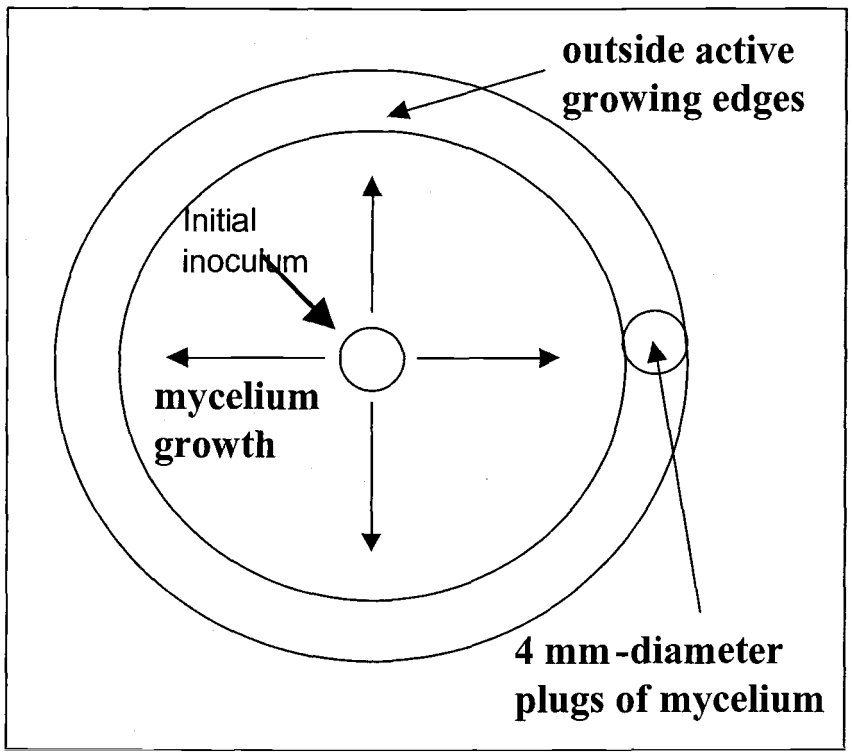

Figure 3.1 Location from which plugs of mycelium were taken from the petri dishes cultures.

3.4.3.2 Sterilization

Prior to inoculation, all culture media were sterilized by heating for 20 minutes at 121 °C in an autoclave, except the soil jars (soil block tests) which were sterilized by heating for 45 min at 121 °C, allowing the chambers to cool overnight, and reheating for another 20 minutes at 121 °C.

3.4.3.3 Incubation chambers and test temperatures.

Incubation chambers were 115-mm diameter petri dishes containing one of the following: malt extract agar (MEA), potato dextrose agar (PDA), amended basal salts, soil (direct exposure and sandwich) or vermiculite. In addition 150 ml Erlenmeyer

flasks containing either 1% malt extract broth, maple sawdust, or red alder sawdust, or 454-ml glass jars containing garden soil were used. All chambers were incubated at 28 °C, except for the liquid media tests, which were incubated at room temperature (22-25 °C).

3.4.3.4 Exposure periods

Samples exposed in the screening tests were incubated for 4, 8, 11, 14 or 17 weeks. The basal salts tests were carried out after the first set of the screening tests, and because of the promising results obtained, a shorter exposure period of 12 weeks with sampling every 3 weeks was used. Samples in the optimization and soil block tests were exposed for the latter times.

3.4.3.5 Moisture content and weight loss sampling

Samples were removed and adhering mycelium, agar, soil, sawdust or vermiculite was carefully removed. Samples were weighed (nearest 0.0001 g), oven-dried for 24 hrs at 104 °C, and weighed again to determine both moisture content and weight loss.

3.4.3.6 Parameters used to estimate the extent of decay

The moisture content was used to determine whether conditions were suitable for fungal growth, while weight loss was used to estimate the extent of decay. These values were calculated based on the estimated amount of wood in the WPC's. It was assumed that the "moisture content" changes would occur in the wood, not in the plastic, and that the plastic was not subject to decay (Naghipour, 1996). As a result, all moisture content and weight loss calculations were expressed on a wood basis.

The moisture content of the WPC was calculated as follows:

Moisture content (%) = ((W1 – W2) / W2) x 100 (*equation I*)

Where:

W1 = Initial oven-dried weight of the sample.

W2 = Weight of the WPC sample immediately after removal from incubation chamber and after adherent mycelium has been brushed off.

The weight loss was calculated as follows:

Weight loss (%) = (100 ((W1 – W3) / W1) x 100 (*equation II*)

Where:

W1 = Initial oven-dried weight of the sample.

W3 = Final oven-dried weight of the sample after fungal exposure.

Because the wood plastic ratios of the WPC samples was 60% wood/40% polypropylene, the final moisture contents and weight losses were calculated as follows:

Wood weight loss = ((T x 100)/60) (*equation III*)

Where T = weight loss of the WPC sample calculated in *equation II.*

The final wood moisture content was calculated following the same procedure.

3.4.3.7 Statistical analysis

Statistical analyses were performed using Statistical Analysis System (SAS Release 8.02, 2002). The procedures Analysis of Variance (ANOVA) and General Linear Models (GLM) were used to analyze the data. Mean values were compared using Duncan's multiple-range test ($\alpha = 0.5$).

3.5 Screening tests

The combinations of media, concentration, exposure periods and test fungi for the screening tests are shown in Figures 3.2 and 3.3.

3.5.1 MEA and amended basal salts tests

Solutions of MEA and amended basal salts were prepared in 1000 ml Erlenmeyer flasks (see Appendix A for concentrations). Twenty petri dish decay chambers were prepared, each containing about 20 ml of MEA (Figure 3.4). Five petri dishes were inoculated with each of the three fungi, and one additional set of five petri dishes were left uninoculated to serve as controls.

When the fungal mycelia reached the edges of the petri dish containing MEA, one sterile glass rod was placed in the center on the agar surface. Ten sterilized WPC's and five maple wafers were exposed to the fungi in each petri dish as follows. Six WPC samples and three maple wafers were placed on the glass rods completely above the agar. Four WPC samples and two wafers were placed so that one end of the specimen was on the glass rod while the other end was in direct contact with the agar. Direct contact with the agar was intended to allow moisture to wick up the ends of samples.

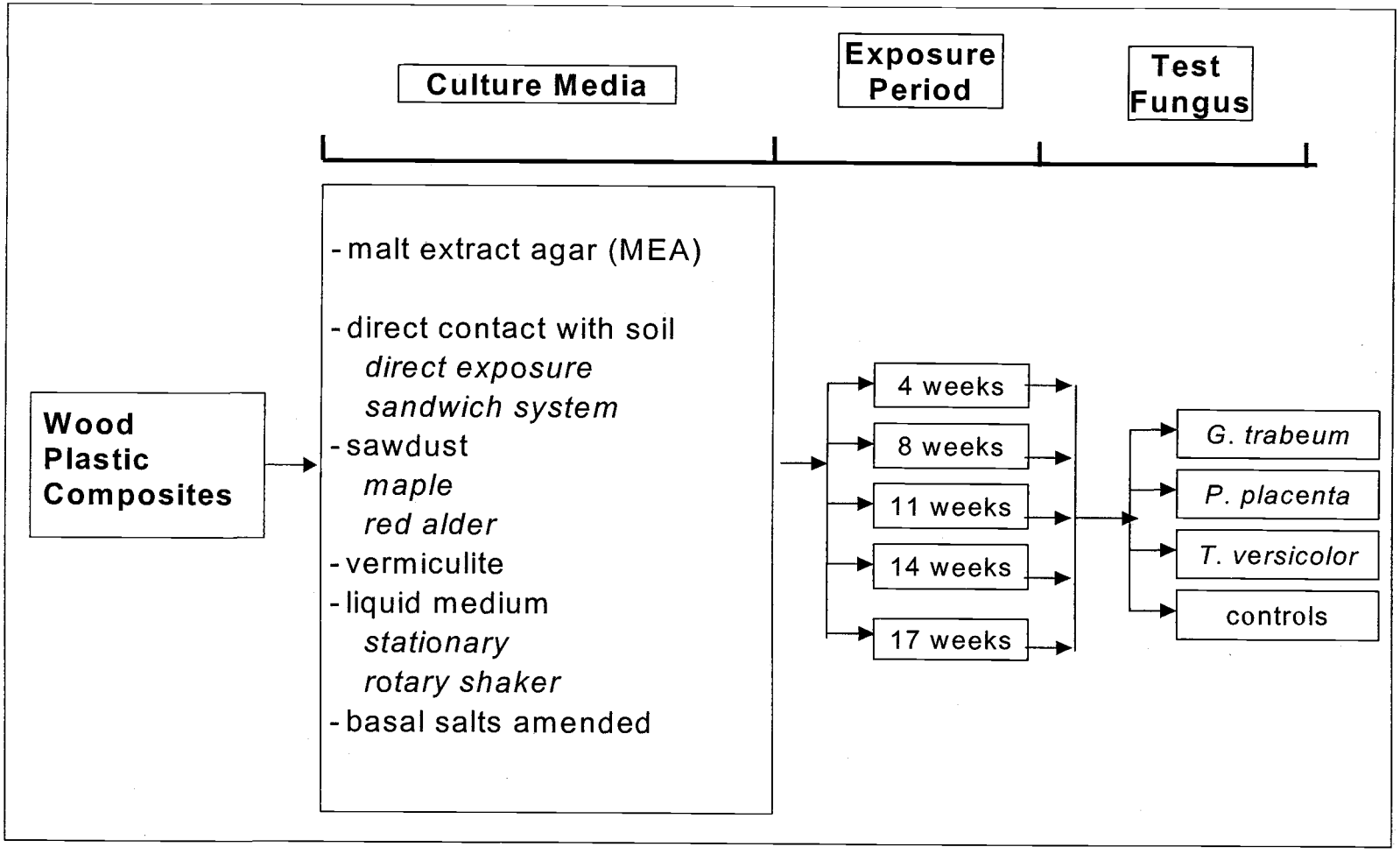

Figure 3.2 Combinations of culture media, exposure periods and fungi used in the screening tests to assess the decay resistance of WPC's.

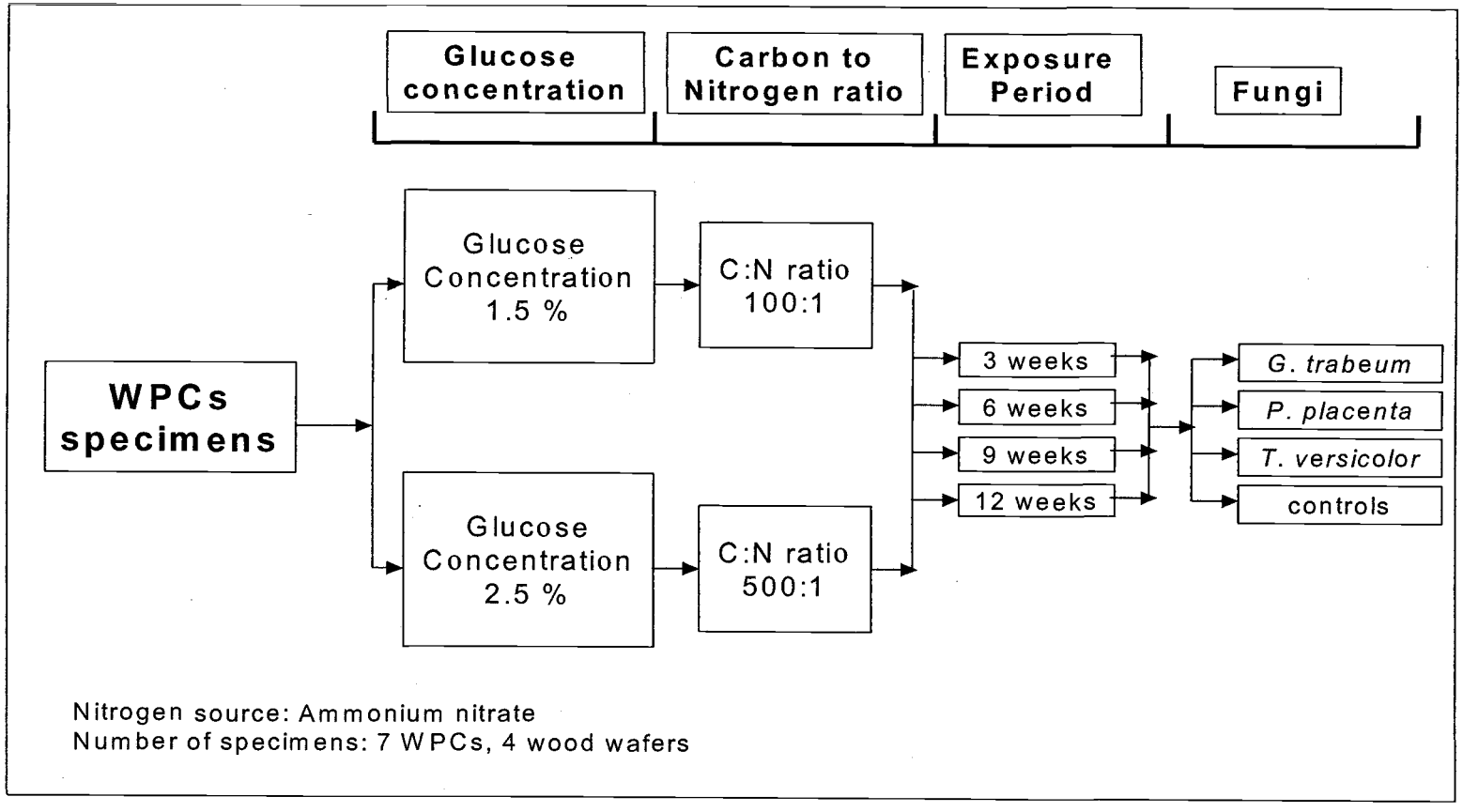

Figure 3.3 Combinations of glucose concentrations, carbon to nitrogen ratios (C:N), exposure periods and fungi used to assess the merits of an amended basal salts media for enhancing decay of WPC's.

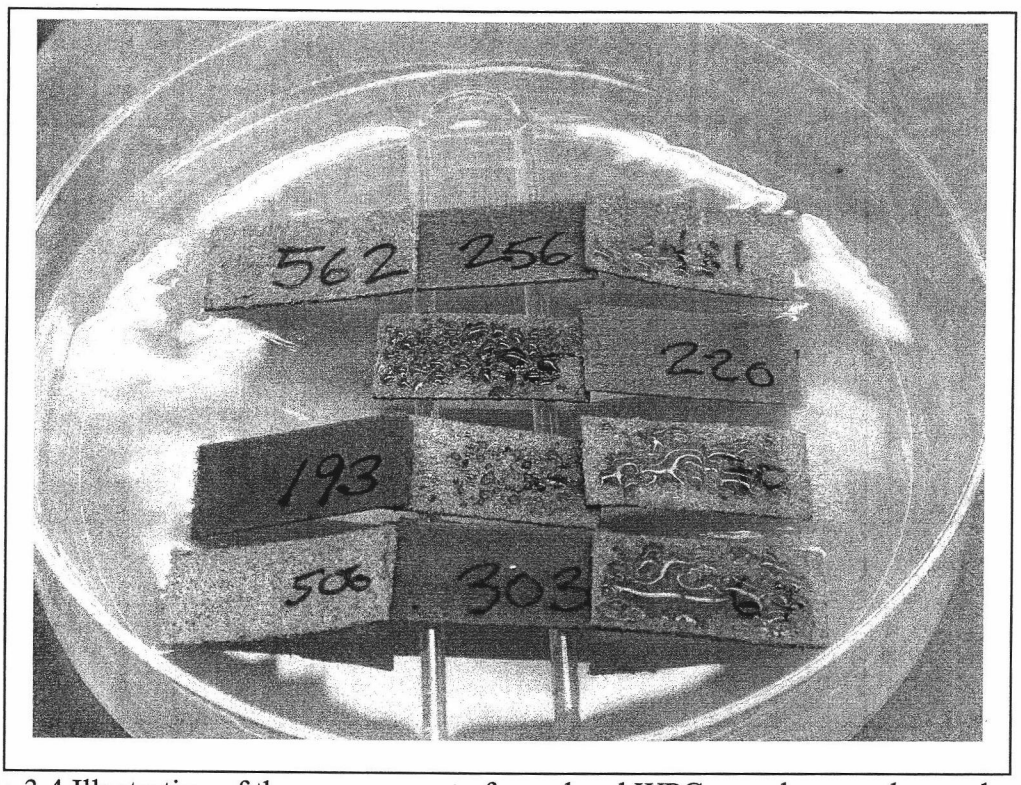

Figure 3.4 Illustration of the arrangement of wood and WPC sample on a glass rod over the agar.

A procedure similar to that used for the specimens exposed on 1% MEA was followed for those samples exposed in basal salts with the following modifications: 1) only seven WPC's and four wood wafers were used for each petri dish, 2) exposure times were 3 to 12 weeks with sample removal every 3 weeks, and 3) sets of four incubation chambers were prepared containing 40 ml of each combination of glucose and carbon to nitrogen ratio, producing a total of 64 incubation chambers. Combinations of glucose concentration, carbon to nitrogen ratios (C:N), exposure periods and test fungi used in the in basal salts media are shown in Figure 3.3.

3.5.2 Direct soil contact soil tests

3.5.2.1 Direct soil exposure tests

Twenty decay chambers were prepared by placing 17 g of garden soil in each petri dish. The moisture content of the soil was below the level required to provide favorable conditions for fungal growth (35%). For this reason, distilled water was added to raise the moisture content of the soil to 70%. Five maple wafers (10 x 70 x 0.5 mm) were placed in each petri dish and sterilized. After cooling, the edges and center of the wafers were inoculated with 4 ml of liquid inoculum of one of the three test fungi and with a 4 mm-diameter mycelial plug of the same fungus. Five petri dishes were prepared for each type of fungus. Five additional decay chambers were left uninoculated to serve as controls. The decay chambers were sealed with paraffin film and incubated for four weeks, at which time the mycelia covered nearly all the wafers. Ten WPC's and five sterile maple wafers were then placed on top of the wood wafers feeders strips in each petri dish and incubated at 28 °C (Figure 3.5).

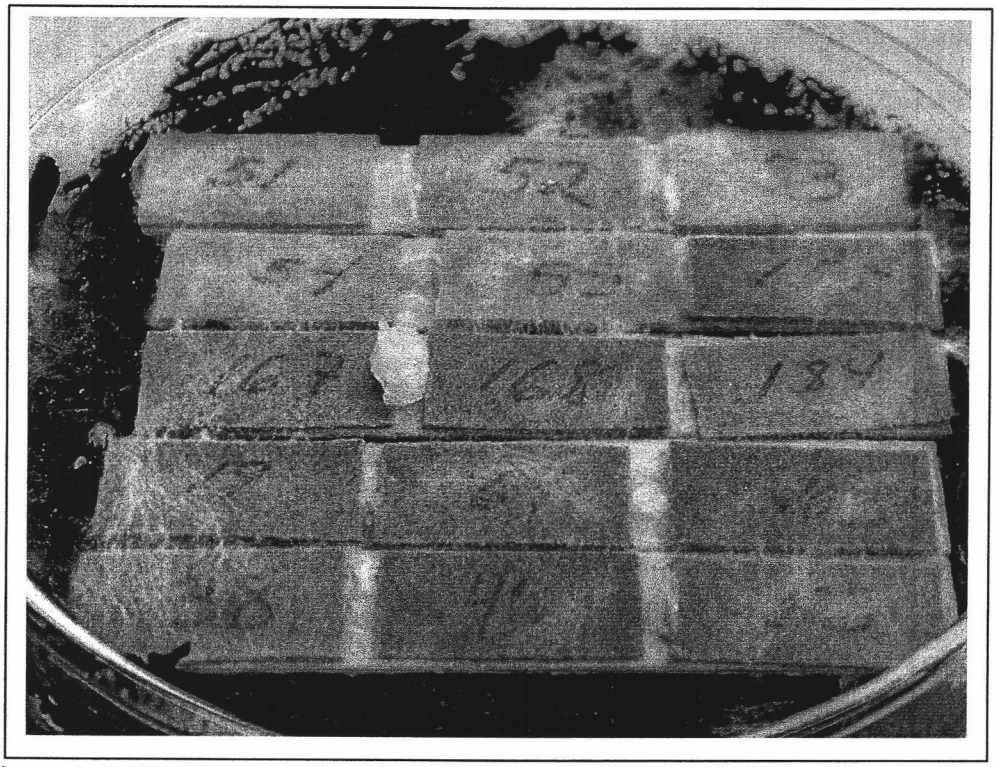

Figure 3.5 WPC's exposed on feeders strips on soil.

3.5.2.2 Sandwich tests

Twenty decay chambers were prepared following the procedure in 3.4.4.2.1. Sets of five petri dishes, each one containing 20 wood wafers (10 x 20 x 0.5 mm), were sterilized and inoculated with 4 ml of liquid inoculum of one of the three test fungi. The remaining five petri dishes were left uninoculated to serve as controls. The decay chambers were sealed with paraffin film and incubated at 28 °C until mycelia covered nearly all the wafers. These wafers were then used to assemble ten sandwiches with two fungal inoculated wafers surrounding one WPC sample. These sandwiches were held together using sterile paperclips (Figure 3.6), and the assemblies were placed above the soil in petri dishes, and incubated (Freitag and Morrell, 1990).

Figure 3.6 Sandwich test setup showing wood feeders clipped on side of the WPC.

3.5.3 Sawdust media tests

Four grams of either maple or red alder sawdust (20 for each) were added to each of forty 150 ml flasks (Figure 3.7). Before sterilization, four ml of distilled water was added to raise the moisture content of the sawdust from 5% to 100%. The flasks were inoculated with 4 ml of the macerated mycelium, and then incubated until the mycelia covered nearly all the sawdust. Ten sterile WPC and five maple wafers were placed randomly on the sawdust and incubated (Figure 3.7) at 28 °C.

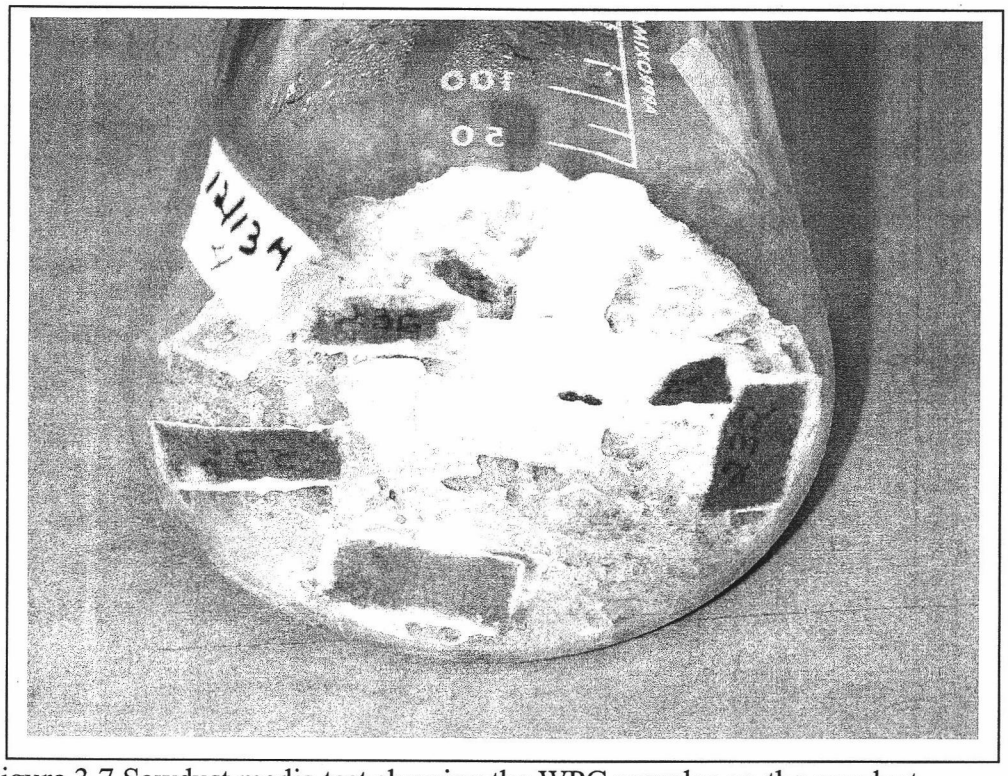

Figure 3.7 Sawdust media test showing the WPC samples on the sawdust.

3.5.4 Vermiculite tests

Nine grams of vermiculite were placed in each of twenty petri dishes. Ten WPC's and five maple wafer samples were buried in the vermiculite. Thirty ml of water containing 0.5% malt extract was added to raise the moisture content of the vermiculite, and to provide additional carbon for the fungus. The incubation chambers were sterilized following the procedure described in 3.2.3.2. After cooling, three sets of five petri dishes were inoculated with 4 ml of fungal inoculum of one of the three test fungi. Five additional petri dishes were left uninoculated to serve as controls. The petri dishes were sealed with paraffin film and incubated at 28 °C (Figure 3.8).

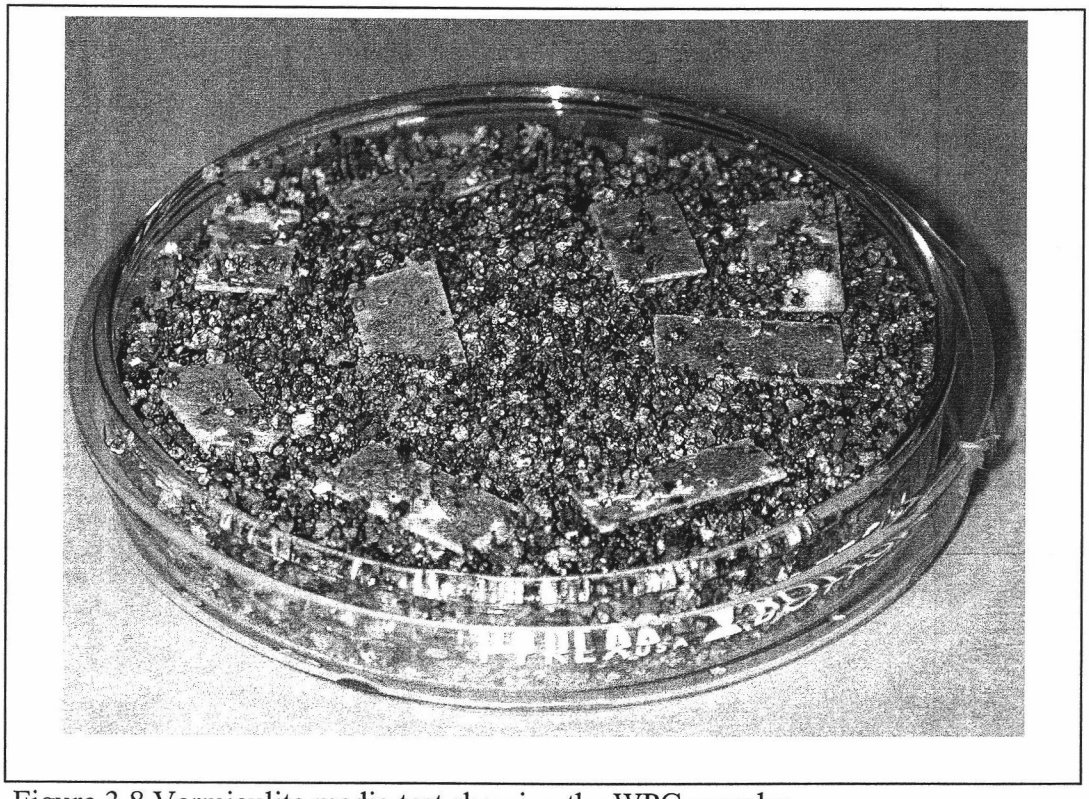

Figure 3.8 Vermiculite media test showing the WPC samples.

3.5.5 Liquid media (rotary shaker and stationary) tests

Three sets of ten incubation chambers were prepared by placing two 4 mm-diameter discs of each fungus into 150 ml Erlenmeyer flasks containing 12 ml of 1% malt broth. Ten additional decay chambers were left uninoculated to serve as controls (Figure 3.9). Five of each set of 10 flasks containing a given fungus, and the controls were incubated under either stationary conditions or in a rotary shaker (90 revolution/minute) for 21 days at room temperature (22-25 °C). Ten WPC's and five maple wafers were placed in each flask, and incubated. During incubation, the water

evaporated making it necessary to add 12-ml of 1% malt extract broth midway through the test.

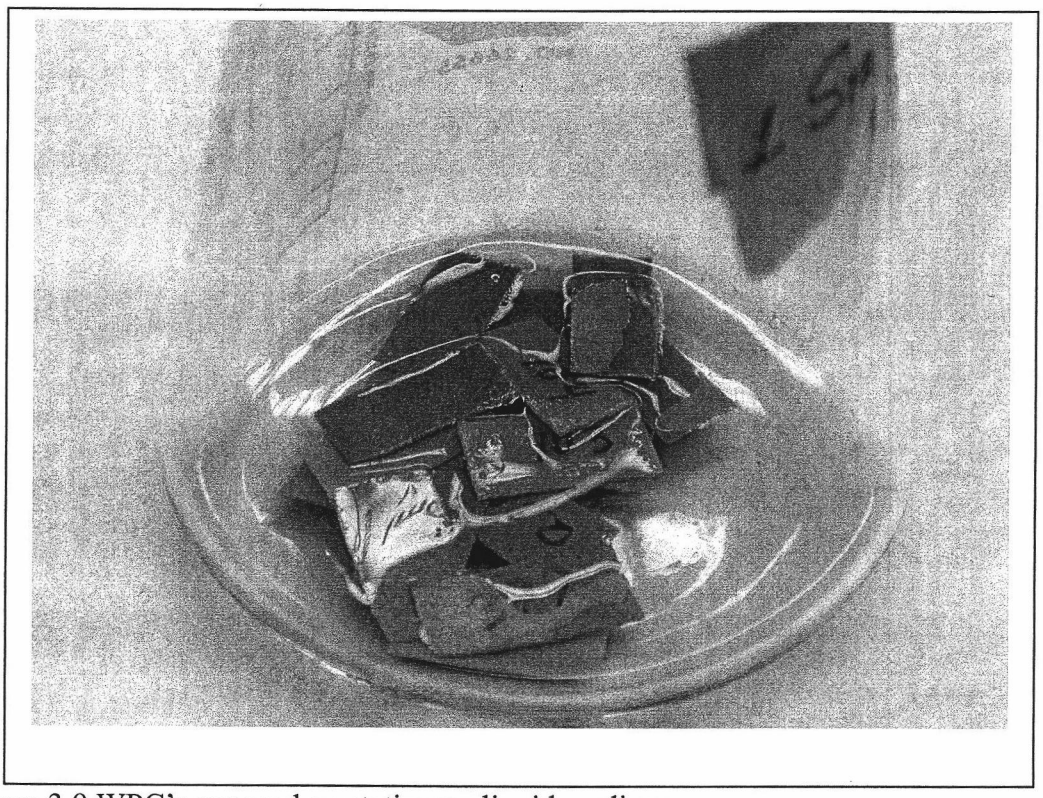

Figure 3.9 WPC's exposed on stationary liquid medium.

3.5.6 Results and discussion

3.5.6.1 Standard Procedures

The screening tests were performed because there was little information about how to accelerate decay of WPC's. Most of the procedures employed have been used to accelerate decay of solid wood, and, therefore, may not have been appropriate for WPC's. As a result, the methods produced widely variable results (Tables 3.1 and 3.2).

All of the water in some incubation chambers containing MEA, soil, or sawdust evaporated over the course of the test (Table 3.1). As a result, the resulting wood

moisture contents and weight losses were lower than would have been expected had there been adequate moisture available during the entire test. These moisture losses were the result of using petri dishes instead of jars. Dishes are more easily prepared and take up less space but are more prone to desiccation. The use of higher initial moisture levels or the addition of water during the tests may be necessary to maintain conditions for active fungal growth.

Some incubation chambers were contaminated during tests, possibly due to inefficient sterilization of the soil chambers. Contamination was also a problem with the sandwich system setup because the assembly process required manual manipulation that increased the possibility of contamination.

3.5.6.2 Moisture content and weight loss

Culture medium, exposure period, and test fungus all generally affected the rate of WPC decay. Moisture contents and weight losses increased as exposure period increased (Table 3.1). Moisture content and weight losses increased steadily in specimens exposed to *G. trabeum* in MEA and vermiculite (Figures 3.10 and 3.11).

All exposed wood wafers (serving as controls) experienced severe weight losses (>70%), except the wood specimens exposed in liquid media. These results suggest that the conditions in most incubation chambers were favorable for fungal attack except in the liquid media.

Table 3.1 Moisture contents (MC) and weight losses (WL) of the wood component in a WPC at selected times following exposure to *G. trabeum*, *P. placenta*, or *T. versicolor* in various types of decay chambers.

Fungi	Decay chambers	Exposure Period [1]									
		4 weeks		8 weeks		11 weeks		14 weeks		17 weeks	
		MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)
G. trabeum	liquid shaker	28.1 (2.3)	0.6 (0.2)	6.8 (3.0)	-3.2 (1.3)	9.4 (2.8)	-1.1 (1)	7.2 (1.2)	0.1 (1.8)	ND[2]	ND[2]
	liquid stationary	31.9 (2.3)	1.6 (0.8)	23.5 (2.2)	-0.1 (1.0)	22.8 (1.4)	-2.3 (1.2)	8.5 (1.2)	2.7 (1.4)	ND[2]	ND[2]
	MEA (plates)	39.3 (4.9)	7.7 (3.2)	41.8 (4.3)	12.7 (3.7)	5.1 (1.9)	11.0 (9.3)	62.6 (8.1)	31.1 (7.4)	54.5 (7.5)	25.1 (7.3)
	maple sawdust	32.7 (1.0)	2.1 (0.7)	30.8 (19.8)	2.3 (0.7)	30.6 (3.5)	2.3 (1.5)	26.9 (1.8)	3.8 (1.2)	26.0 (1.2)	3.0 (1.4)
	alder sawdust	33.6 (1.5)	3.2 (0.9)	30.0 (1.6)	1.2 (0.7)	28.4 (1.3)	3.0 (1.0)	28.2 (2.2)	3.6 (0.9)	21.0 (1.6)	1.2 (0.8)
	soil (sandwich)	35.1 (1.2)	5.5 (1.3)	32.4 (2.9)	7.3 (1.9)	35.5 (4.1)	6.3 (2.5)	26.0 (1.4)	8.8 (2.0)	34.3 (2.5)	7.3 (4.7)
	soil (plates)	32.4 (9.2)	2.8 (2.6)	34.1 (3.4)	7.8 (3.8)	20.9 (2.6)	14.5 (3.9)	26.1 (3.7)	6.0 (1.9)	31.3 (1.8)	6.5 (3.7)
	vermiculite	30.7 (1.9)	4.6 (1.6)	40.1 (4.5)	18.4 (4.7)	39.9 (7.2)	32.3 (4.8)	62 (12.1)	41.6 (9.2)	74.4 (18.1)	47.4 (11.0)
P. placenta	liquid shaker	29.4 (2.3)	0.9 (0.5)	6.7 (1.4)	-1.6 (1.6)	11 (3.4)	-1.7 (0.4)	8.0 (2.2)	-0.3 (1.1)	ND[2]	ND[2]
	liquid stationary	26.8 (5.7)	0.7 (0.3)	21.8 (2.6)	-0.5 (0.9)	26.8 (1.1)	-0.4 (1.1)	7.3 (1.2)	1.9 (2.1)	ND[2]	ND[2]
	MEA (plates)	33.6 (2.9)	3.3 (1.8)	37.6 (3.8)	10.9 (3.2)	46.5 (8.5)	21.2 (6.8)	9.1 (1.3)	16.5 (6.8)	8.6 (1.0)	21.2 (6.0)
	maple sawdust	35.1 (3.0)	4.7 (1.6)	36.4 (3.2)	11.9 (3.4)	43.8 (4.9)	20.4 (3.2)	58.4 (5.7)	29.9 (4.1)	11.9 (1.3)	16.7 (5.3)
	alder sawdust	34.2 (2.7)	4.6 (1.4)	39.4 (3.7)	13.0 (3.1)	52.8 (8.2)	26.1 (6.3)	60.4 (10.9)	32.6 (4.7)	10.9 (1.0)	30.4 (6.0)
	soil (sandwich)	31.9 (1.4)	1.6 (0.6)	30.9 (2.0)	3.6 (1.6)	34.3 (1.4)	4.9 (1.8)	23.2 (1.6)	3.3 (2.3)	27.4 (1.3)	8.7 (4.4)
	soil (plates)	30.6 (1.3)	1.4 (0.5)	29.8 (1.3)	2.4 (1.6)	24.6 (1.2)	2.3 (1.4)	11.1 (1.1)	1.9 (0.6)	32.2 (1.4)	2.8 (1.2)
	vermiculite	30.5 (2.5)	1.9 (1.2)	31.2 (3.1)	6.1 (7.2)	27.0 (4.2)	11.2 (3.1)	38.4 (8.6)	18.5 (6.5)	46.6 (6.1)	21.5 (5.5)

[1] Average moisture contents and weight losses are the means of 10 specimens per treatment. Values in parentheses are standard deviations.

[2] ND = Not determined.

Table 3.1 (continuation)

Fungi	Decay chambers	Exposure Period [1]									
		4 weeks		8 weeks		11 weeks		14 weeks		17 weeks	
		MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)
T. versicolor	liquid shaker	30.2 (2.1)	0.8 (0.5)	6.7 (1.2)	-0.3 (1.6)	30.3 (2.3)	0.6 (1.2)	6.8 (0.7)	-0.2 (1.1)	ND[2]	ND[2]
	liquid stationary	29.3 (1.8)	1.9 (0.6)	10.3 (0.4)	0.0 (2.1)	27.5 (2.6)	1.9 (2.1)	8.7 (1.8)	-0.1 (2.4)	ND[2]	ND[2]
	MEA (plates)	33.5 (2.1)	3.3 (1.0)	38.9 (5.2)	9.7 (2.3)	48.7 (6.3)	17.4 (1.7)	55.6 (7.9)	21.1 (4.0)	58.1 (10.7)	30.2 (5.6)
	maple sawdust	33.6 (1.7)	3.6 (0.7)	32.9 (3.5)	5.2 (1.2)	37.4 (3.1)	9.2 (2.0)	40.9 (4.2)	10.1 (2.8)	36.3 (4.8)	16.2 (3.4)
	alder sawdust	32.9 (1.8)	4.3 (0.8)	33.8 (2.4)	6.2 (1.2)	29.5 (3.3)	9.6 (1.5)	33.8 (5.8)	10.5 (2.2)	32.3 (6.4)	23.8 (5.7)
	soil (sandwich)	34.3 (1.8)	3.9 (1.1)	34.1 (4.0)	5.7 (2.9)	40.4 (5.4)	13.0 (4.8)	29.4 (2.7)	13.7 (4.0)	38.8 (4.9)	17.0 (5.5)
	soil (plates)	31.0 (0.9)	1.2 (0.7)	44.2 (26.3)	9.3 (5.6)	33.2 (3.9)	16.2 (3.5)	46.1 (8.9)	31.6 (4.8)	43.8 (7.6)	31.9 (8.6)
	vermiculite	27.1 (1.7)	1.4 (0.7)	28.4 (2.1)	1.4 (1.1)	23.2 (3.3)	1.8 (1.3)	21.9 (1.4)	2.5 (1.5)	29.0 (1.5)	1.6 (1.4)
controls	liquid shaker	26.5 (1.8)	0.0 (0.5)	26.2 (1.3)	-0.2 (0.6)	5.5 (1.2)	-5.5 (1.9)	ND[2]	ND[2]	ND[2]	ND[2]
	liquid stationary	25.9 (0.9)	1.1 (0.8)	25.0 (1.2)	-0.1 (0.4)	7.6 (2.2)	-3.0 (1.3)	ND[2]	ND[2]	ND[2]	ND[2]
	MEA (plates)	29.2 (2.4)	0.8 (0.6)	29.7 (2.0)	-0.3 (0.4)	27.7 (2.0)	1.2 (0.7)	ND[2]	ND[2]	10.5 (1.5)	-0.3 (1.0)
	maple sawdust	28.1 (1.9)	1.2 (0.7)	27.3 (1.3)	1.6 (0.2)	29.4 (1.3)	0.7 (1.2)	23.2 (2.5)	0.9 (1.9)	15.9 (0.7)	1.8 (0.5)
	alder sawdust	31.2 (1.9)	0.9 (0.5)	29.6 (1.8)	2.3 (0.8)	32.0 (1.2)	1.8 (0.9)	26.4 (2.9)	1.7 (0.8)	27.3 (1.7)	1.4 (0.6)
	soil (sandwich)	33.6 (1.3)	1.3 (0.5)	29.7 (1.7)	1.1 (0.6)	21.0 (1.5)	0.4 (0.6)	25.3 (1.4)	0.9 (0.6)	28.4 (1.7)	0.6 (0.6)
	soil (plates)	21.8 (1.2)	1.8 (0.6)	22.1 (1.1)	1.5 (0.7)	7.0 (2.5)	1.9 (2.1)	8.3 (0.4)	1.1 (0.6)	13.0 (0.5)	0.9 (0.6)
	vermiculite	24.9 (1.7)	1.5 (1.1)	26.8 (1.6)	1.3 (1.0)	25.7 (3.4)	2.0 (0.7)	24.2 (1.2)	1.5 (1.2)	22.1 (3.2)	1.9 (0.8)

[1] Average moisture contents and weight losses are the means of 10 specimens per treatment. Values in parentheses are standard deviations.

[2] ND = Not determined.

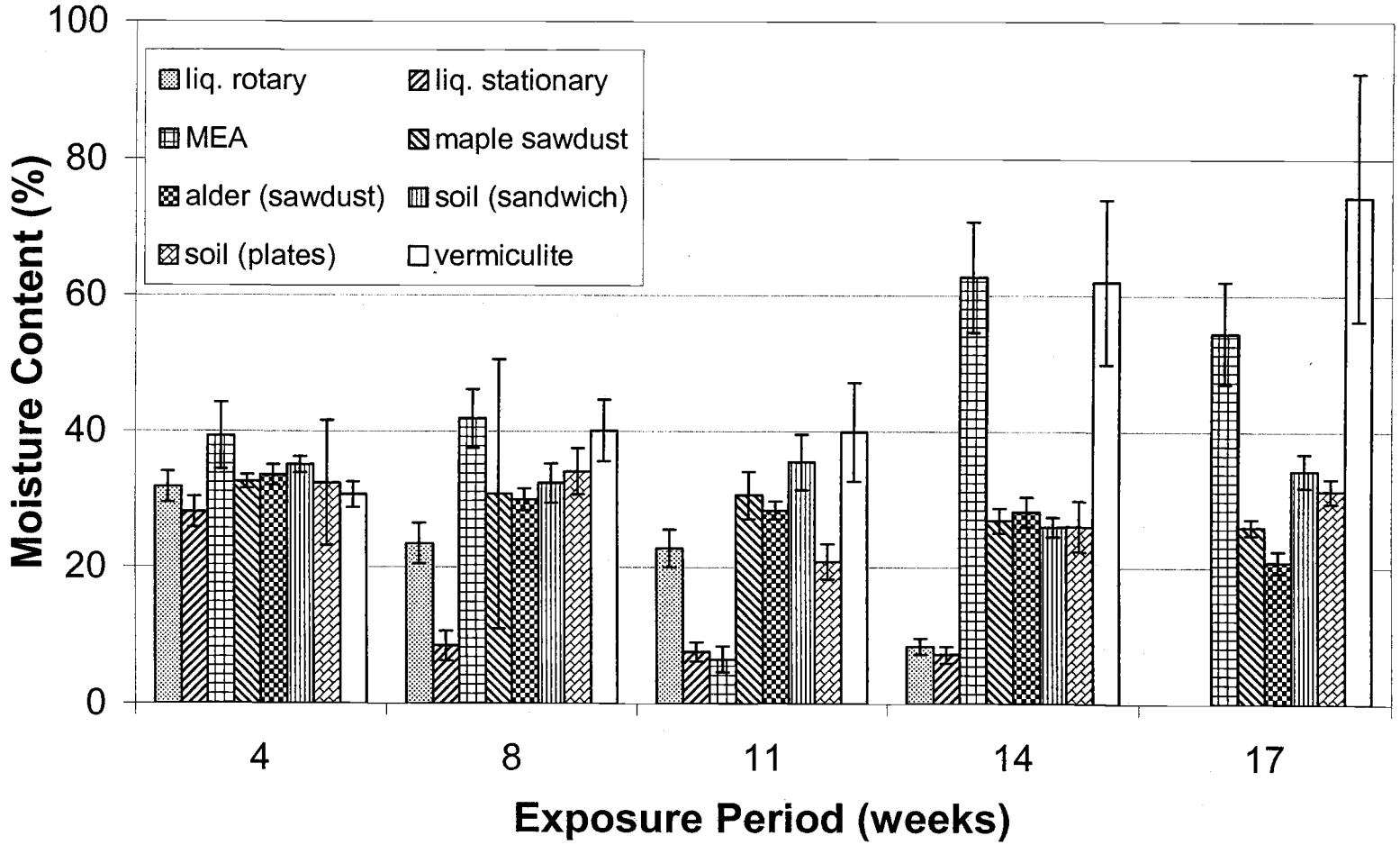

Figure 3.10 Moisture contents of the wood component of a WPC following exposure to *G. trabeum* for 4 to 17 weeks using various decay systems. (Error bars represent one standard deviation).

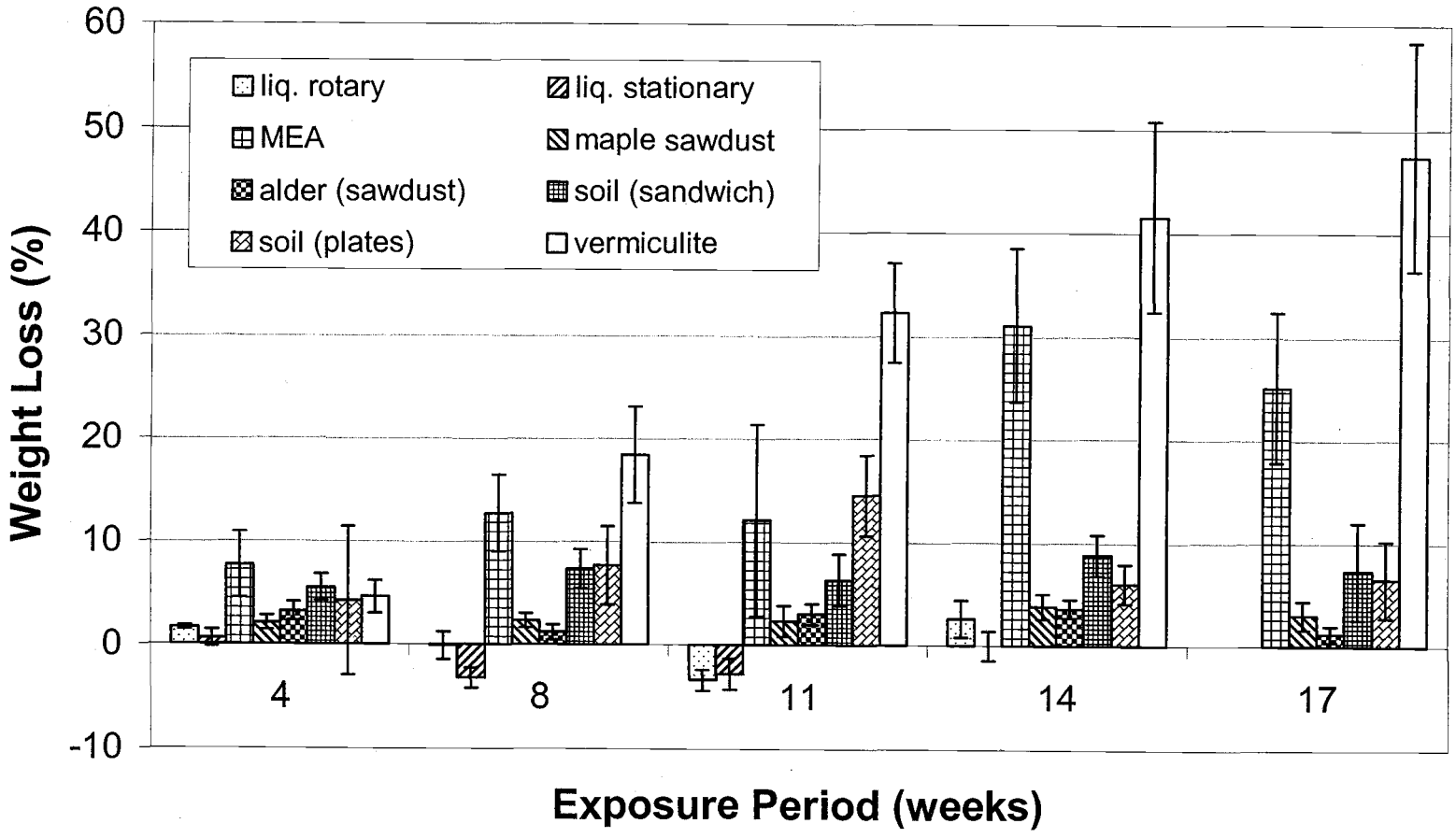

Figure 3.11 Weight losses of the wood component of a WPC following exposure to *G. trabeum* using varying types of decay systems. (Error bars represent one standard deviation).

There were several anomalies in the results of the specimens exposed in MEA. Moisture contents and weight losses increased steadily over the first eight weeks, then declined or stabilized after that point for *G. trabeum* (Figures 3.10, 3.11). These declines appeared to be related to desiccation of the media, rather than to any change in fungal capability.

Moisture contents and weight losses of the WPC's exposed to *P. placenta* increased steadily in all three decay chamber types (soil, MEA and vermiculite) (Figures 3.12 and 3.13). There were several anomalies in these tests:

- Weight losses of the specimens exposed in MEA for 14 and 17 weeks were lower than expected, possibly because moisture was limiting.
- Declining moisture content in the sawdust appeared to reduce the rate of decay.

Vermiculite produced the most uniform weight losses in the WPC specimens (Figures 3.10 and 3.12) exposed to *G. trabeum* and *P. placenta*, however, these weight losses were lower then those found with the WPC specimens exposed on MEA or sawdust (Table 3.1).

Moisture contents and weight losses in specimens exposed in both the MEA and the soil chambers were higher when exposed to *T. versicolor* (Figure 3.14). The relatively small change in weight loss between 14 and 17 weeks in specimens exposed in MEA, soil or sawdust (Figures 3.10 and 3.11) may have been due to inadequate moisture in the incubation chamber. In some cases leaks in the wax film around the glass petri dishes may have allowed excess evaporation.

The lower weight losses found with vermiculite than with MEA or sawdust (Figure 3.15) may be due to the inability of *T. versicolor* to grow under lower moisture regimes. The use of higher initial moisture levels may be needed to encourage growth by this fungus. Unexposed WPC's specimens, serving as controls, did not exhibit increases in moisture contents or weight losses in comparison with fungal exposed controls (Figures 3.16 and 3.17).

Culture media can play important roles in both fungal nutrition and the ability to maintain wood moisture content at levels capable of supporting fungal attack. Amended basal salts did not appear to provide suitable conditions for fungal attack of samples exposed to *G. trabeum* or *P. placenta* (Table 3.2). Lack of nutrients in the medium could be an explanation for this failure; however, WPC specimens exposed to *T. versicolor* on amended basal salts experienced significant increases in moisture contents and weight losses (Table 3.2, Figures 3.18 and 3.19). This result suggests that this media provided more favorable conditions for the white rot fungus. White rot fungi tend to be more sensitive to sugar level than brown rot fungi, primarily due to feedback inhibition of cellullase by excess glucose. The more defined basal salts may have reduced feedback encouraging more rapid decay by the white rot fungus.

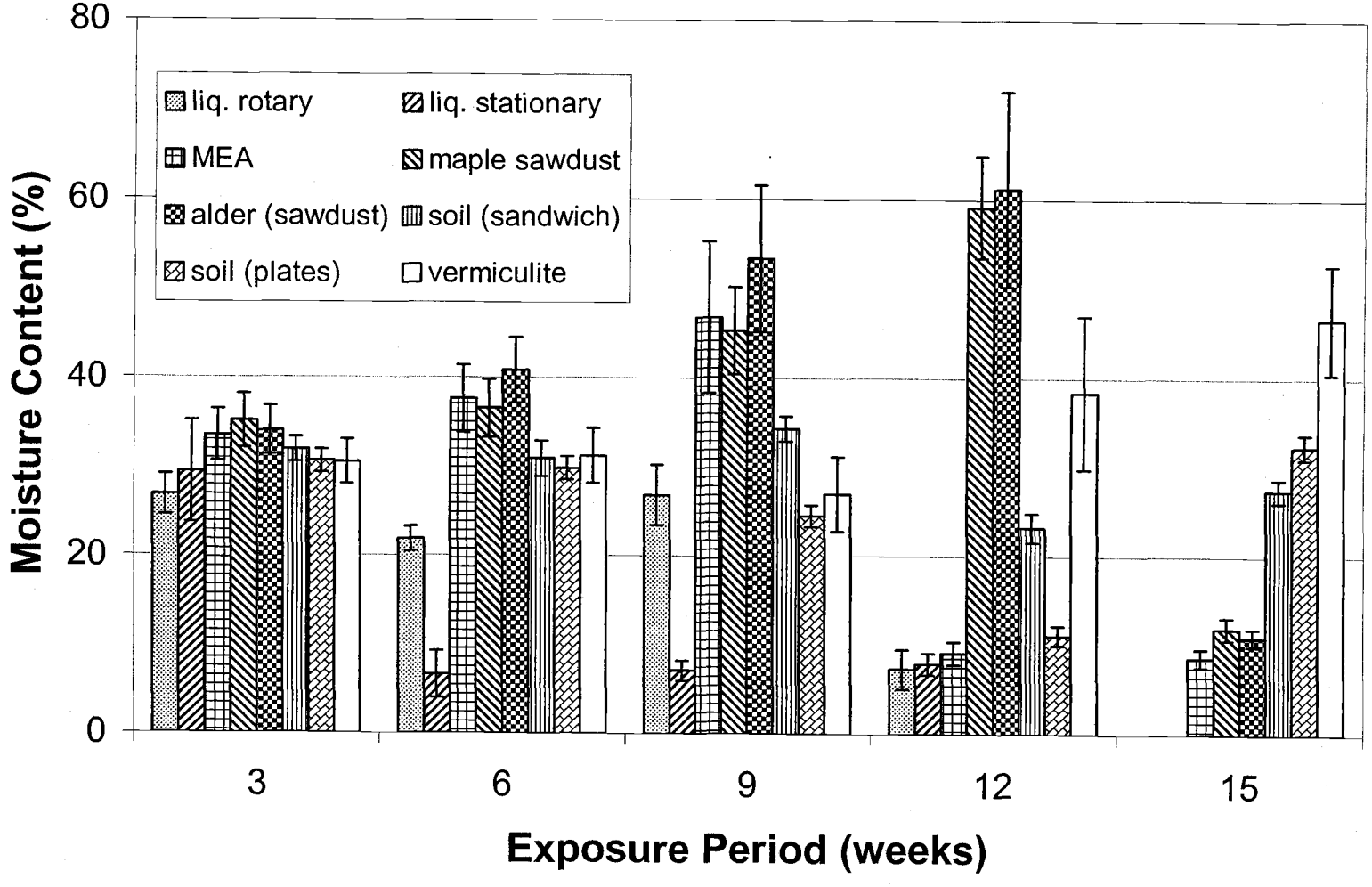

Figure 3.12 Moisture contents of the wood component of a WPC following exposure to *P. placenta* for 4 to 17 weeks using various decay systems. (Error bars represent one standard deviation).

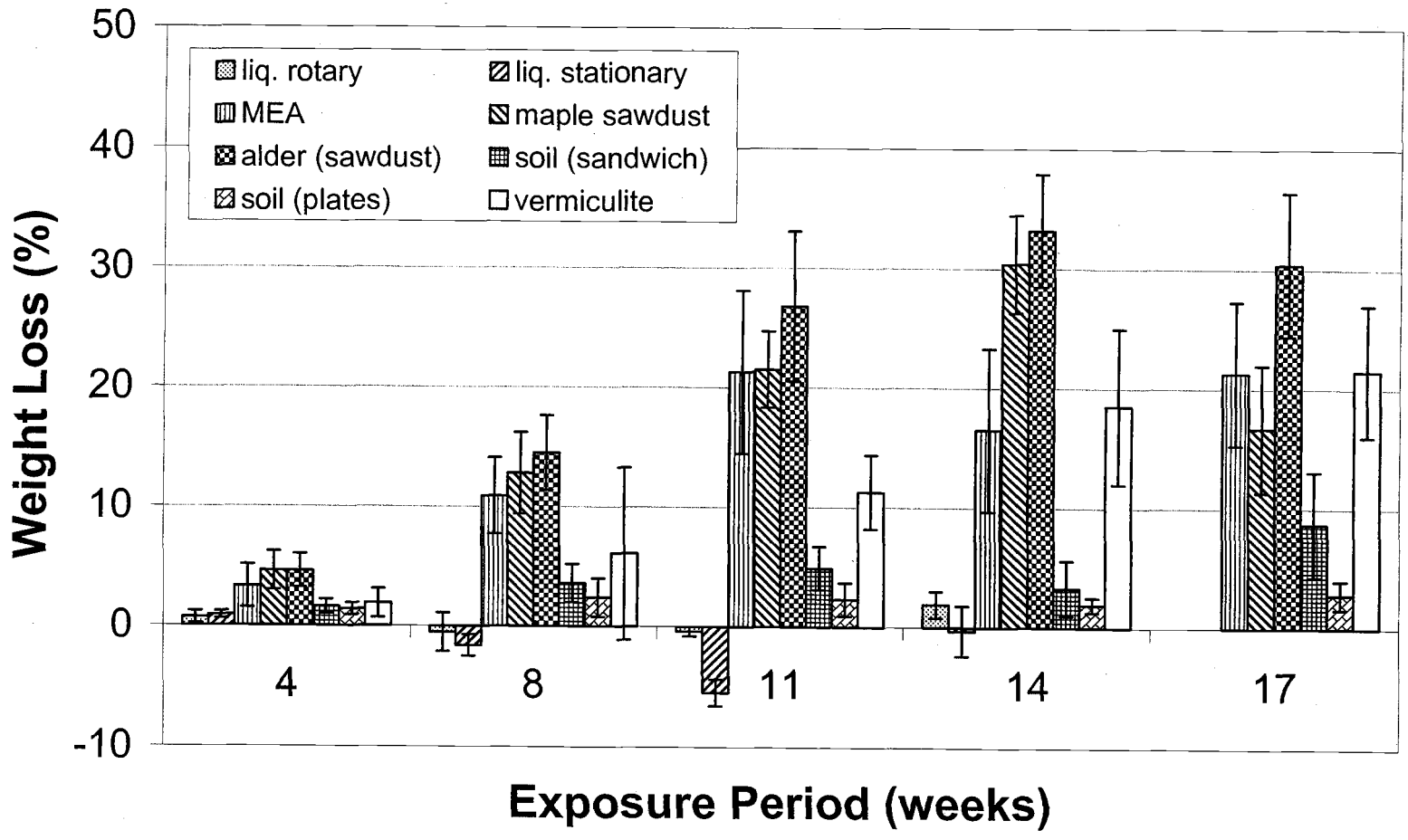

Figure 3.13 Weight losses of the wood component of a WPC following exposure to *P. placenta* using varying types of decay systems. (Error bars represent one standard deviation).

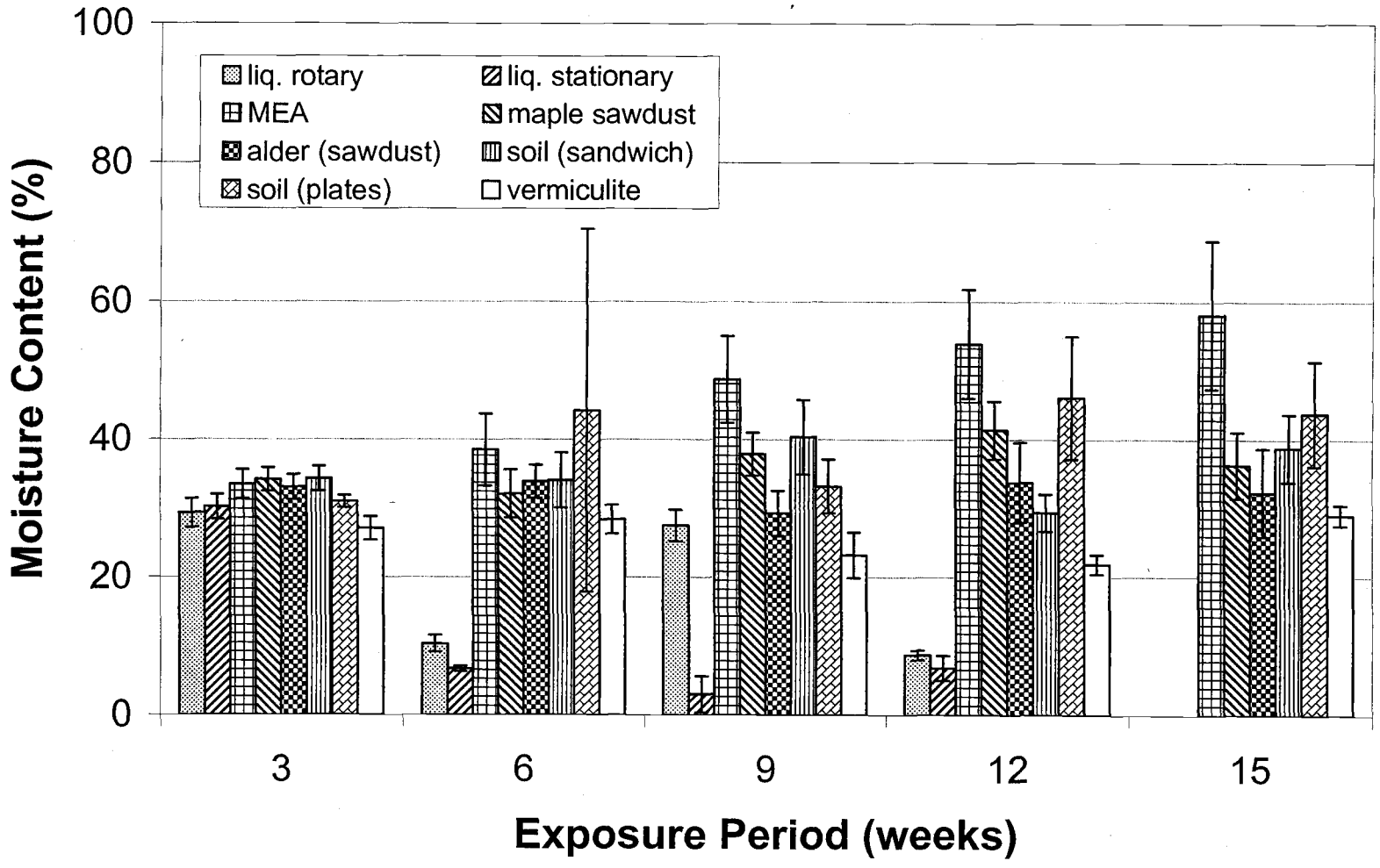

Figure 3.14 Moisture contents of the wood component of a WPC following exposure to *T. versicolor* for 4 to 17 weeks using various decay systems. (Error bars represent one standard deviation).

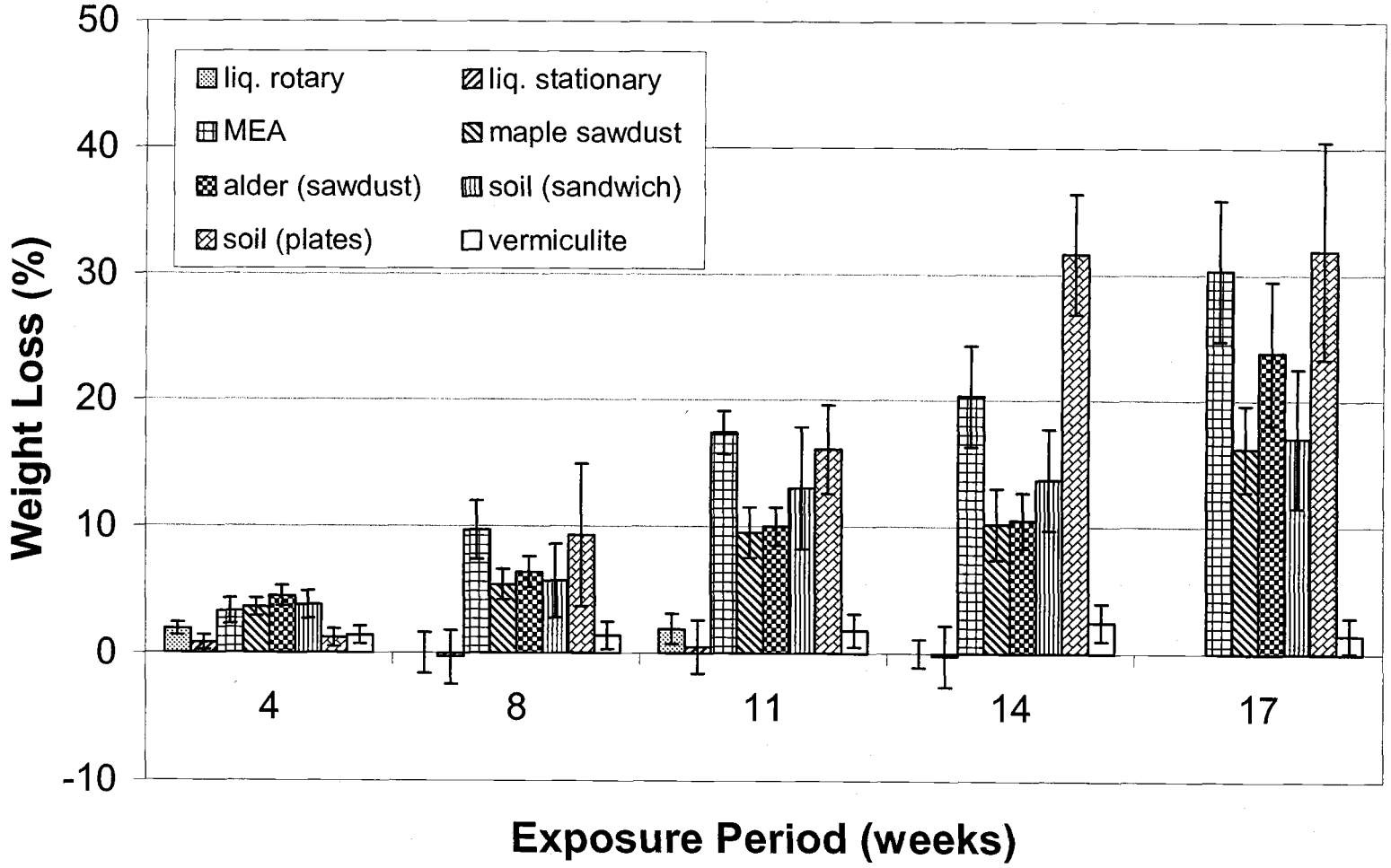

Figure 3.15 Weight losses of the wood component of a WPC following exposure to *T. versicolor* using varying types of decay systems. (Error bars represent one standard deviation).

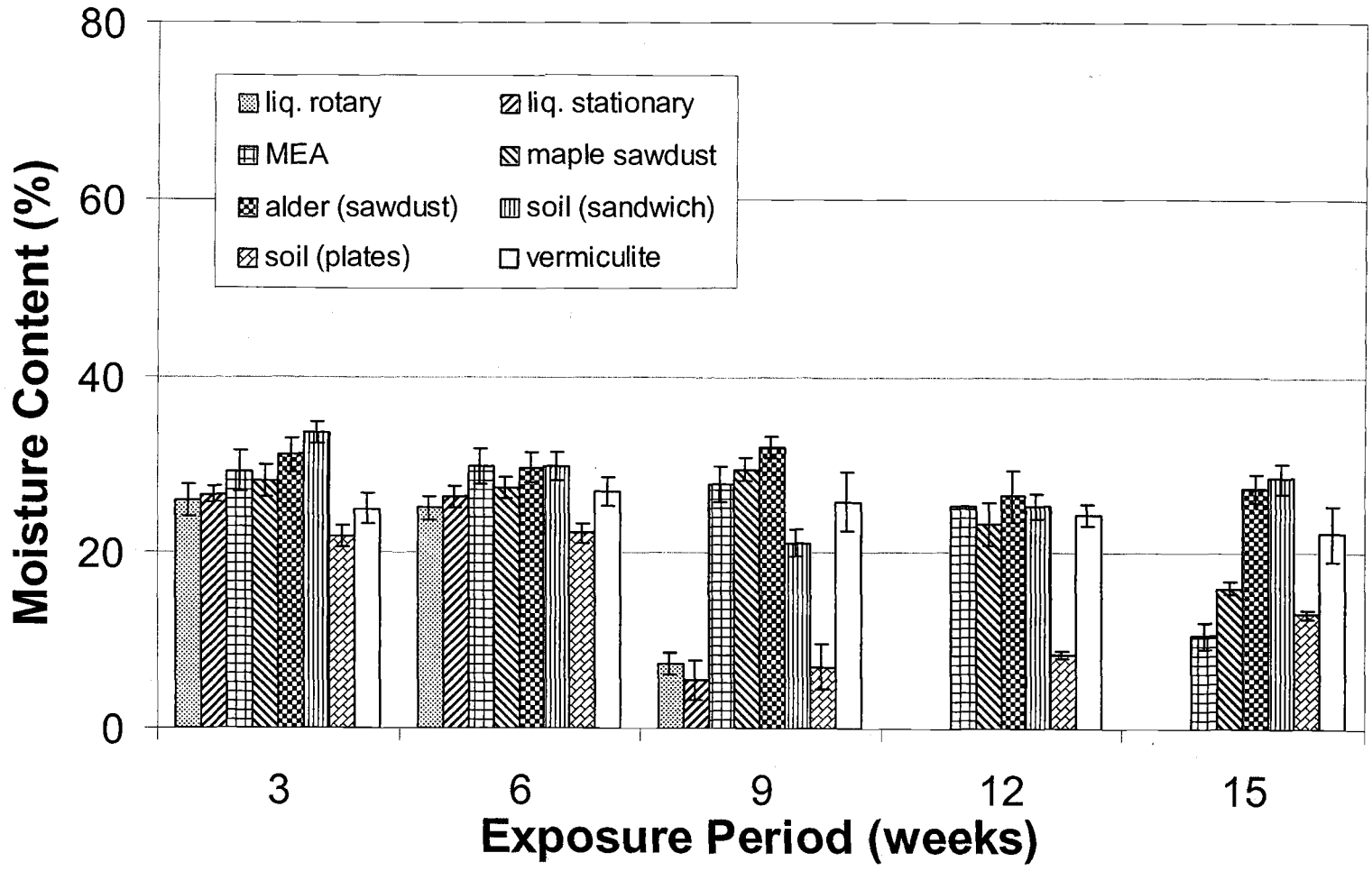

Figure 3.16 Moisture contents of the wood component of a WPC following exposure for 4 to 17 weeks under various systems in the absence of fungi. (Error bars represent one standard deviation).

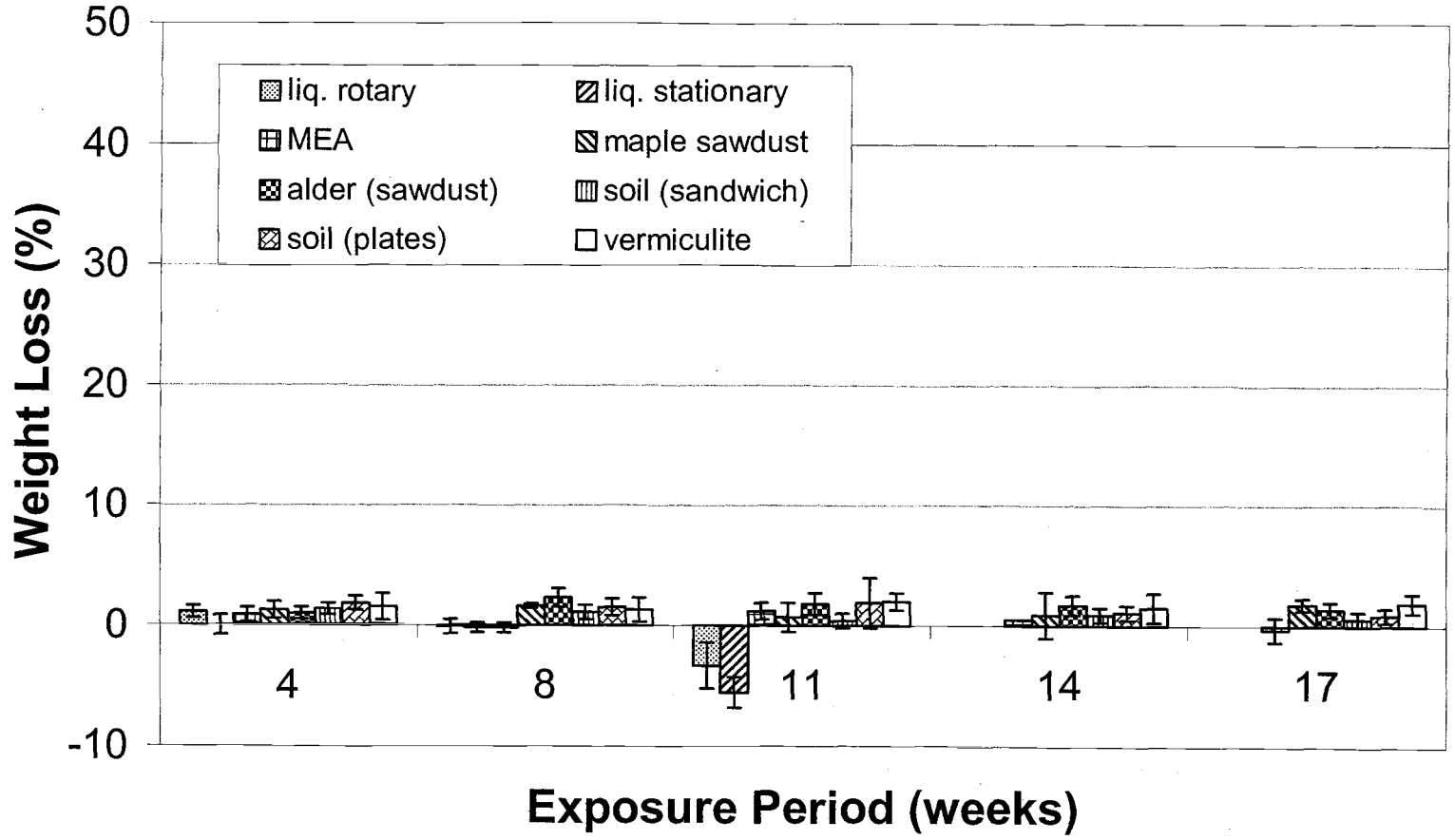

Figure 3.17 Weight losses of the wood component of a WPC following exposure for 4 to 17 weeks under various systems in the absence of fungi. (Error bars represent one standard deviation).

Table 3.2 Effect of glucose concentration and carbon to nitrogen ratio (C:N) on moisture content (MC) and weight loss (WL) of the wood component in a WPC exposed to selected decay fungi on basal salts media.

Fungi	Glucose (%)	C:N ratio	Exposure Period [1]							
			3 weeks		6 weeks		9 weeks		12 weeks	
			MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)
G. trabeum	1.5	100:1	28.7 (1.0)	2.4 (1.4)	31.6 (1.0)	2.3 (0.6)	32.2 (1.9)	1.8 (0.4)	31.5 (3.1)	1.9 (1.7)
	1.5	500:1	29.3 (1.7)	0.7 (1.0)	33.6 (1.5)	3.0 (1.7)	32.3 (2.4)	0.7 (1.8)	35.7 (3.1)	0.9 (0.7)
	2.5	100:1	27.3 (2.3)	1.3 (1.2)	33.7 (1.7)	0.8 (0.8)	33.3 (0.7)	1.4 (0.9)	31.5 (3.5)	1.1 (1.3)
	2.5	500:1	29.7 (1.9)	2.9 (2.4)	33.3 (1.7)	2.3 (1.1)	32.9 (1.4)	1.5 (1.2)	34.7 (2.9)	4.1 (2.0)
P. placenta	1.5	100:1	30.0 (2.9)	1.2 (1.3)	33.0 (1.5)	2.2 (0.6)	32.4 (0.7)	2.2 (0.6)	33.3 (2.4)	1.0 (0.8)
	1.5	500:1	30.2 (1.8)	1.5 (0.6)	33.1 (1.3)	2.1 (0.8)	33.2 (1.5)	2.5 (0.7)	34.4 (2.4)	1.2 (0.7)
	2.5	100:1	29.7 (1.8)	1.3 (0.7)	33.2 (2.1)	0.6 (0.8)	32.6 (1.0)	0.8 (0.6)	31.6 (2.8)	5.3 (10.7)
	2.5	500:1	29.9 (2.5)	1.9 (1.3)	33.6 (1.3)	1.5 (1.1)	33.0 (0.9)	0.3 (0.9)	34.3 (2.7)	2.4 (1.0)
T. versicolor	1.5	100:1	31.5 (1.6)	4.7 (1.1)	40.1 (6.9)	9.4 (4.8)	58.7 (7.1)	28.1 (5.6)	46.9 (8.4)	15.1 (7.1)
	1.5	500:1	30.6 (2.2)	2.2 (0.8)	40.2 (4.9)	10.8 (4.0)	53.3 (13.2)	23.7 (9.6)	57.7 (6.0)	26.4 (4.4)
	2.5	100:1	30.9 (1.2)	3.4 (1.1)	38.3 (4.2)	6.9 (3.6)	43.6 (2.8)	13.0 (2.7)	44.3 (8.3)	13.4 (8.0)
	2.5	500:1	31.9 (3.1)	3.0 (1.9)	41.8 (2.6)	10.4 (1.9)	59.5 (11.5)	25.3 (8.3)	60.5 (20.3)	22.8 (11.7)
controls	1.5	100:1	24.3 (4.2)	1.9 (2.4)	33.3 (2.0)	0.4 (0.8)	31.9 (1.9)	1.4 (0.7)	33.4 (1.7)	1.9 (1.0)
	1.5	500:1	31.3 (0.7)	0.0 (1.0)	32.3 (1.0)	4.6 (6.2)	32.9 (0.7)	1.4 (0.6)	30.6 (3.7)	1.6 (0.8)
	2.5	100:1	30.9 (3.7)	1.2 (0.6)	31.0 (1.8)	1.5 (1.0)	31.5 (2.5)	1.1 (1.0)	31.8 (1.5)	1.1 (0.2)
	2.5	500:1	25.5 (0.8)	1.3 (0.7)	32.6 (1.1)	1.9 (0.9)	31.4 (1.5)	1.1 (0.9)	32.8 (1.7)	0.8 (0.4)

[1] Average moisture contents and weight losses are the means of 7 samples per treatment. Values in parentheses are standard deviations.

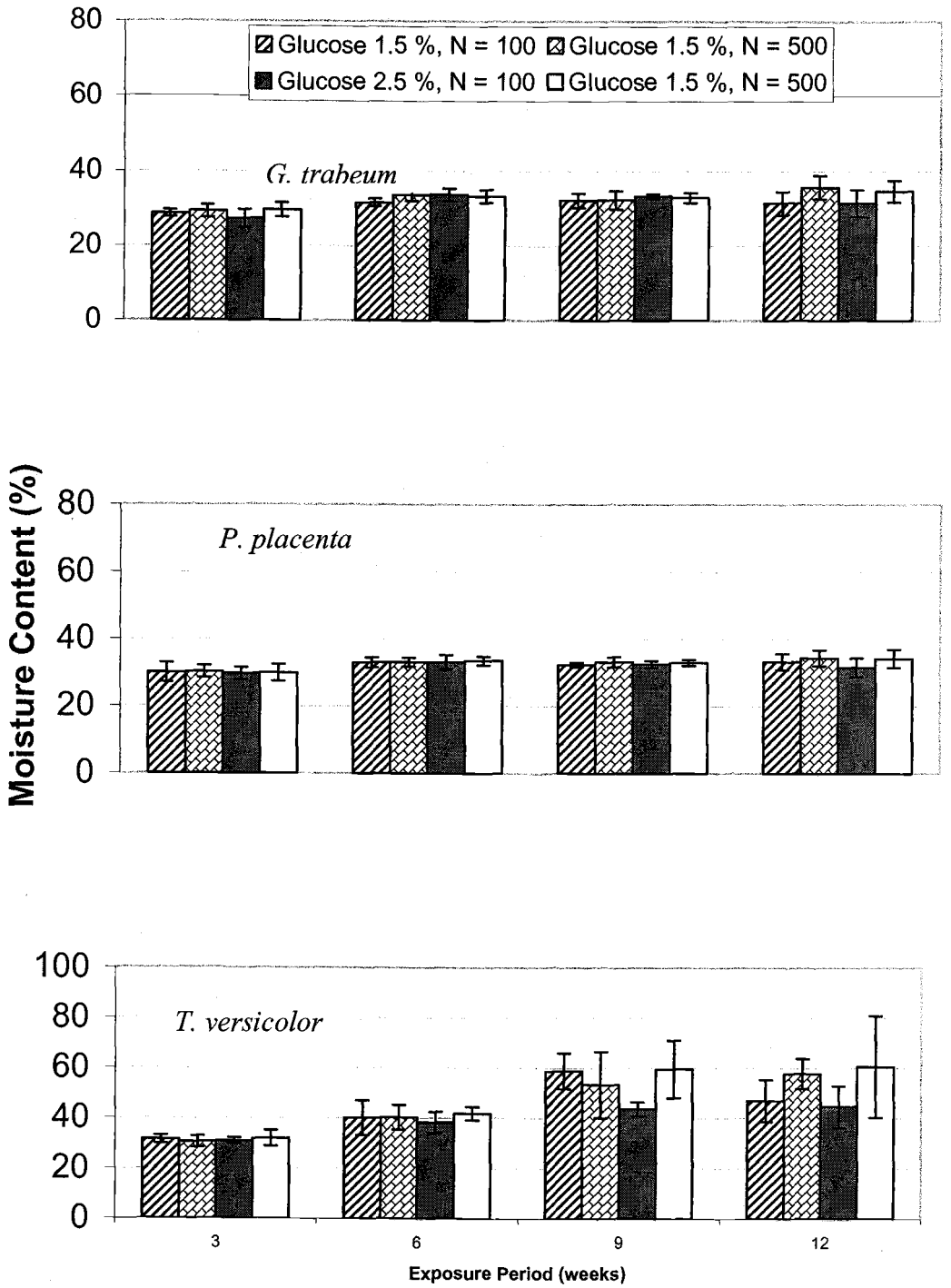

Figure 3.18 Effect of glucose concentrations and C:N ratio in a basal salts media on the moisture contents of the wood component of a WPC following exposure to fungal attack for 3 to 12 weeks. (Error bars represent one standard deviation).

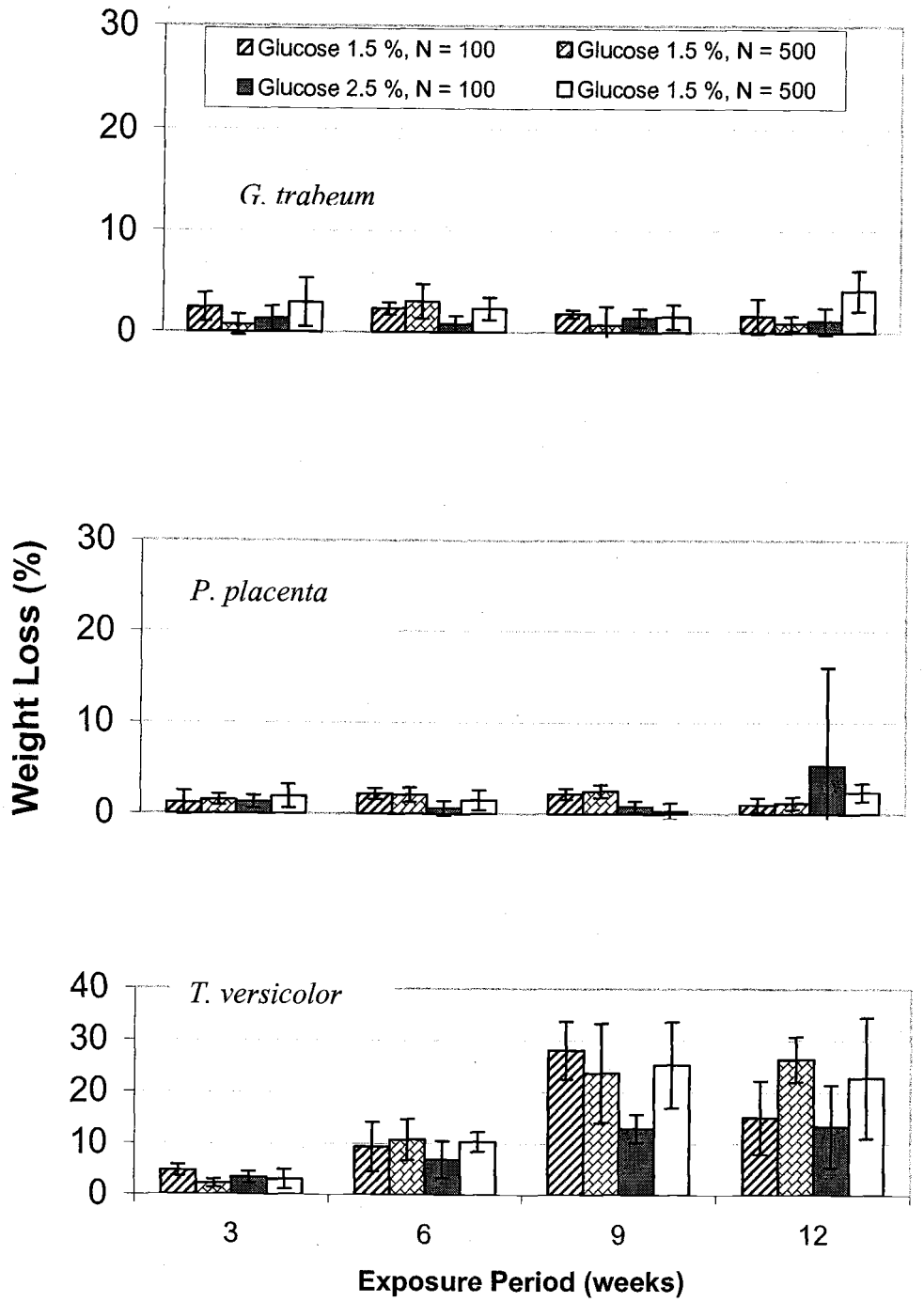

Figure 3.19 Effect of glucose concentrations and C:N ratio in a basal salts media on the weight losses of the wood component of a WPC following exposure to fungal attack for 3 to 12 weeks. (Error bars represent one standard deviation).

3.5.7 Conclusions

These results indicated that MEA was the most suitable medium for all three fungi because it enhanced fungal attack, was easy to prepare, did not experience contamination, and produced acceptable results in a relatively short period. Despite the good results found with the vermiculite, it was decided not to run further tests with this medium because it required liquid inoculum. The production of liquid inoculum requires additional procedures beyond those needed for MEA. Vermiculite also required longer exposure periods to induce decay, although the final levels of decay were close to those found with MEA.

3.6 Optimization tests

Based on the results of the screening tests, a second series of tests were performed to determine the most effective media for producing decay in WPC's.

There were two objectives to these tests:

1) Determine whether MEA or PDA (potato dextrose agar) was the most suitable media, and
2) Determine which concentration of each medium produced the most rapid and greatest amount of decay in the WPC.

3.6.1 Procedures and methods

Decay chambers were prepared following procedures described in the screening experiment for MEA (section 3.2.4.1). Petri dishes were prepared with 40 ml of 0.5, 1.0 or 1.5% MEA or PDA and inoculated. Once the fungus had cover the agar surface, seven WPC and four maple wafers were placed in each petri dish, and the chambers

were incubated for three to 12 weeks. All samples were placed within one petri dish for each treatment. Seven WPC's and four wood wafer samples were removed every three weeks for each fungus and processed as described previously.

The combinations of media, concentration, exposure periods and test fungi for the optimization tests are shown in Figure 3.20.

3.6.2 Results and Discussion

3.6.2.1 Standard procedures

Both the MEA and PDA produced reasonable fungal attack of the WPC's. These media offered several advantages over soil including abundant nutrients, favorable moisture levels for fungal attack, lower risk of contamination, and low cost.

3.6.2.2 Moisture content and weight loss

Media type, exposure period, and test fungus had strong effects on moisture contents and weight losses of the WPC's. Moisture content and weight losses of the WPC specimens and wood wafers increased steadily with increased exposure period. WPC samples exposed for six weeks to *G. trabeum* experienced weight losses exceeding 20% while those samples exposed in 1.5% PDA to *P. placenta* or *T. versicolor* exhibited weight losses over 15% (Table 3.3). These results suggest that agar tests can produce acceptable results in a relatively short period.

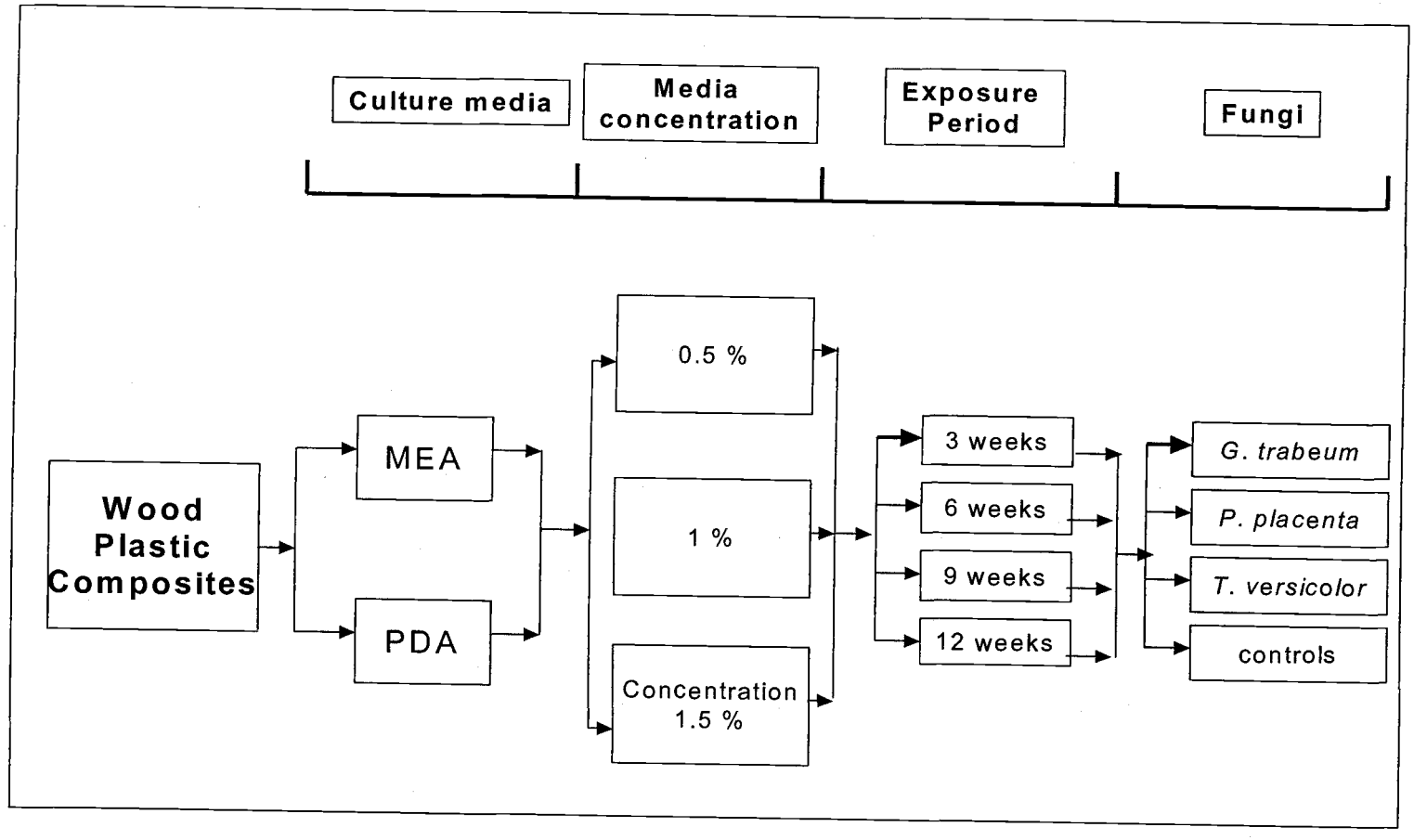

Figure 3.20 Media types and concentrations, exposure periods and test fungi evaluated in the tests to optimize accelerated decay of WPC's.

Potato dextrose agar appeared to be a more suitable medium for *T. versicolor* and *P. placenta*, but it was unsuitable for *G. trabeu*m. In contrast, MEA was a more suitable medium for use with *P. placenta* and *G. trabeum,* but was less suitable for *T. versicolor* (Table 3.3, Figures 3.21 to 3.26). Unexposed WPC specimens serving as controls did not experience increases in moisture contents and weight losses (Figures 3.27 and 3.28) during exposure.

All wood wafers (serving as controls) exposed to fungal attack in any concentration of MEA or PDA experienced severe weight losses (>75%). These results suggest that conditions in the incubation chambers containing either MEA or PDA were favorable for fungal attack.

The reasons for the higher weight losses with *T. versicolor* than *G. trabeum* at higher PDA concentrations are unclear (Table 3.3, Figure 3.26). Most likely one or several factors within the PDA medium limited *G. trabeum* growth, but not *T. versicolor* growth. The pH, the concentration of sugars or nitrogen content may have enhanced enzyme production by the white-rot fungus. PDA is rich in dextrose (D-glucose) while the main sugar in MEA is maltose and might influence favorably fungal metabolic activity of *G. trabeum* but not the *T. versicolor* (Figure 3.22). It appears that each fungus has different nutritional requirements and/or the successful growth of each fungus was highly dependent on the medium employed.

Table 3.3 Moisture contents (MC) and weight losses (WL) of the wood component in a WPC exposed for 3 to 12 weeks on MEA or PDA to *G. trabeum, P. placenta* or *T. versicolor,* or left non-exposed.

Test Fungus	Culture Media	Concentration (%)	Exposure Period [b]							
			3 weeks		6 weeks		9 weeks		12 weeks	
			M C (%)	W L (%)	M C (%)	W L (%)	M C (%)	W L (%)	M C (%)	W L (%)
G. trabeum	MEA [a]	0.5	32.3 (1.6)	2.9 (1.1)	40.2 (7.4)	19.6 (3.8)	61.6 (7.1)	32.2 (4.9)	69.9 11.0)	38.3 (8)
		1.0	27.0 (1.9)	4.5 (1.2)	42.3 (6.5)	23.3 (3.9)	54.9 (4.1)	25.8 (2.6)	68.3 (10.1)	36.7 (8)
		1.5	30.1 (2.0)	5.9 (1.2)	48.8 (5.5)	20.2 (6.2)	54.6 (6.8)	26.3 (4.6)	68.9 (7.3)	35.4 (5.6)
	PDA	0.5	28.1 (2.6)	2.5 (1.4)	31.7 (1.4)	2.4 (0.5)	32.5 (1.7)	2.7 (1.3)	35.0 (3.6)	6.9 (3.6)
		1.0	36.1 (1.5)	2.5 (0.6)	32.4 (2.0)	1.5 (0.5)	33.6 (1.5)	2.8 (0.5)	36.6 (2.1)	2.2 (1.6)
		1.5	33.9 (2.6)	2.9 (1)	33.6 (1.1)	1.5 (0.4)	37.3 (3.3)	5.5 (3.5)	43.3 (12.4)	12.0 (12.4)
P. placenta	MEA	0.5	26.1 (2.6)	3.4 (1)	34.0 (4.0)	13.1 (3.2)	50.9 (7.5)	23.0 (6.4)	67.5 (9.8)	37.3 (7.1)
		1.0	30.2 (2.6)	3.6 (0.6)	33.8 (4.1)	10.4 (2.4)	59.1 (5.0)	29.2 (3.5)	70.8 (7.2)	39.8 (5.8)
		1.5	28.7 (2.2)	2.6 (0.4)	39.8 (4.4)	12.4 (3)	46.3 (4.9)	19.0 (3.7)	67.8 (7.5)	36.9 (3.9)
	PDA	0.5	29.0 (2.4)	2.8 (1.3)	45.8 (5.9)	15.0 (5.5)	59.9 (8.2)	30.0 (6.4)	74.3 (12.7)	41.7 (8.6)
		1.0	34.8 (2.6)	2.0 (0.8)	45.0 (2.7)	14.3 (2.1)	34.3 (1.7)	2.4 (0.5)	87.2 (4.4)	47.7 (4.6)
		1.5	37.4 (0.8)	5.9 (0.4)	48.2 (5.3)	16.1 (4.3)	68.0 (6.5)	35.6 (4.1)	81.2 (8.3)	45.4 (6.3)
T. versicolor	MEA	0.5	27.0 (4.7)	0.6 (0.3)	33.3 (6.1)	6.5 (3.2)	42.9 (3.4)	12.8 (3.3)	52.4 (8.7)	17.2 (5.8)
		1.0	25.7 (3.3)	1.7 (0.8)	31.6 (2.9)	6.6 (2.7)	39.0 (3.1)	8.1 (2.2)	45.0 (4.2)	15.1 (2.7)
		1.5	24.7 (1.8)	2.2 (0.6)	34.0 (1.1)	3.6 (1.3)	45.4 (2.5)	14.5 (2.3)	44.3 (5.0)	12.2 (3.9)
	PDA	0.5	36.7 (2.4)	6.4 (2.3)	41.4 (4.5)	9.1 (3.7)	47.3 (10.1)	15.8 (9.5)	56.9 (16.1)	20.5 (12.5)
		1.0	39.8 (5.7)	7.3 (3.8)	47.0 (3.5)	16.0 (2.3)	67.4 (12.0)	32.1 (8.7)	64.8 (16.3)	29.3 (12.5)
		1.5	39.2 (2.5)	6.0 (1.2)	47.2 (2.6)	15.2 (2.6)	67.2 (11.1)	32.3 (8.9)	95.6 (21.2)	49.1 (11.5)
controls	MEA	0.5	28.0 (2.0)	0.3 (0.7)	30.5 (2.1)	0.8 (0.5)	31.1 (2.4)	1.0 (1.2)	31.8 (2.5)	1.8 (0.7)
		1.0	27.7 (1.9)	0.0 (0.7)	32.5 (1.2)	1.3 (0.8)	31.8 (2.2)	1.5 (0.3)	30.7 (1.2)	1.6 (0.7)
		1.5	28.0 (1.8)	1.6 (0.5)	32.2 (2.0)	1.0 (0.3)	31.7 (2.5)	2.1 (1.2)	33.3 (4.0)	0.7 (0.3)
	PDA	0.5	31.7 (1.2)	1.8 (0.3)	31.6 (2.5)	1.1 (0.4)	30.9 (2.1)	2.4 (1.5)	30.9 (2.3)	1.1 (0.5)
		1.0	33.2 (1.3)	1.5 (0.6)	33.0 (1.0)	1.5 (0.7)	31.5 (1.6)	0.5 (0.6)	33.7 (1.8)	1.6 (0.7)
		1.5	34.3 (2.1)	2.0 (0.6)	32.3 (1.1)	0.6 (0.2)	31.0 (1.3)	0.8 (0.3)	33.7 (2.4)	1.7 (0.9)

[a] MEA =malt extract agar, PDA = potato dextrose agar. [b] Values represent means of seven samples and one standard deviation.

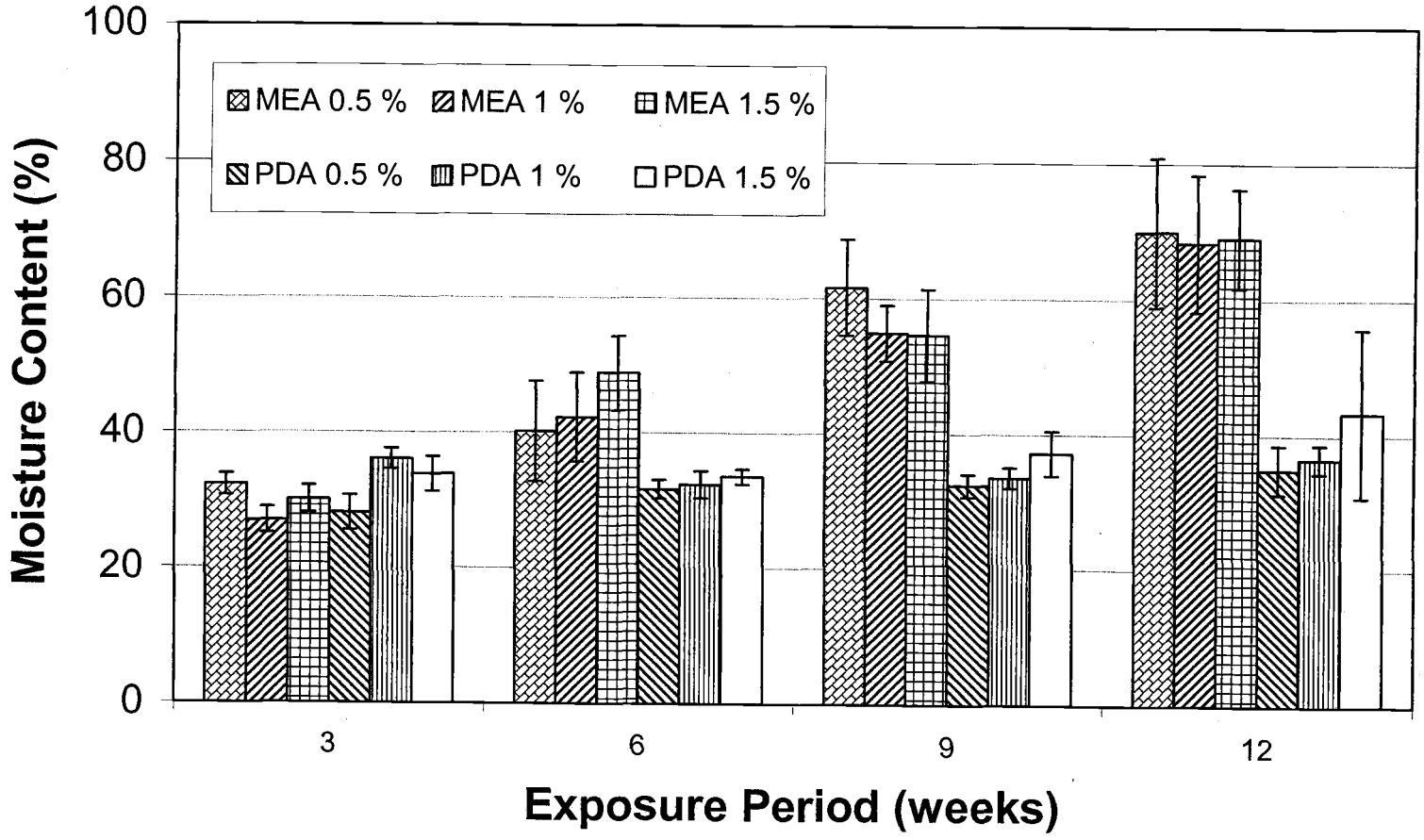

Figure 3.21 Moisture contents of the wood component in a WPC exposed for 3 to 12 weeks to *G. trabeum* in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA). (Error bars represent one standard deviation).

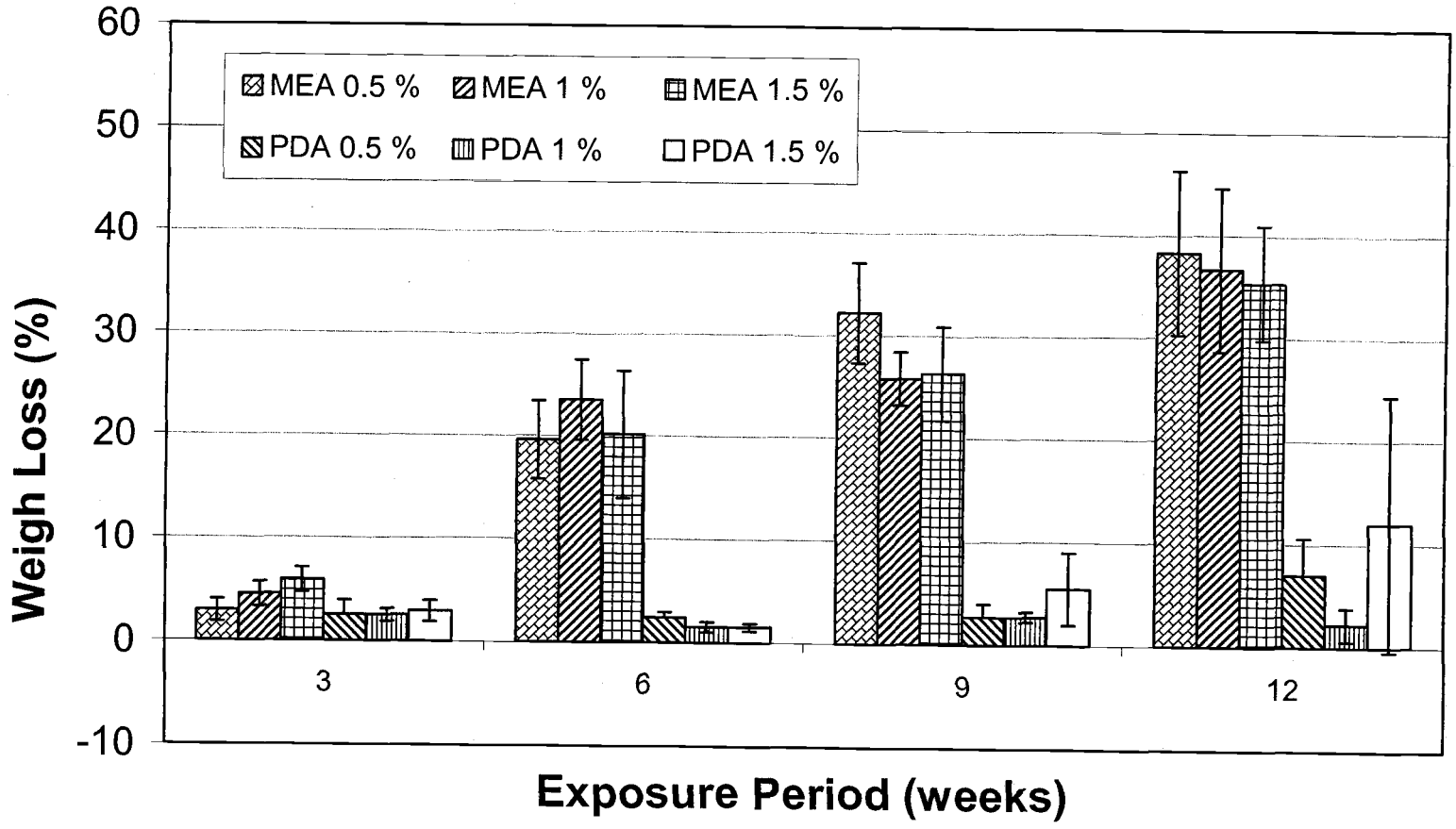

Figure 3.22 Weight losses of the wood component in a WPC exposed for 3 to 12 weeks to *G. trabeum* in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA). (Error bars represent one standard deviation).

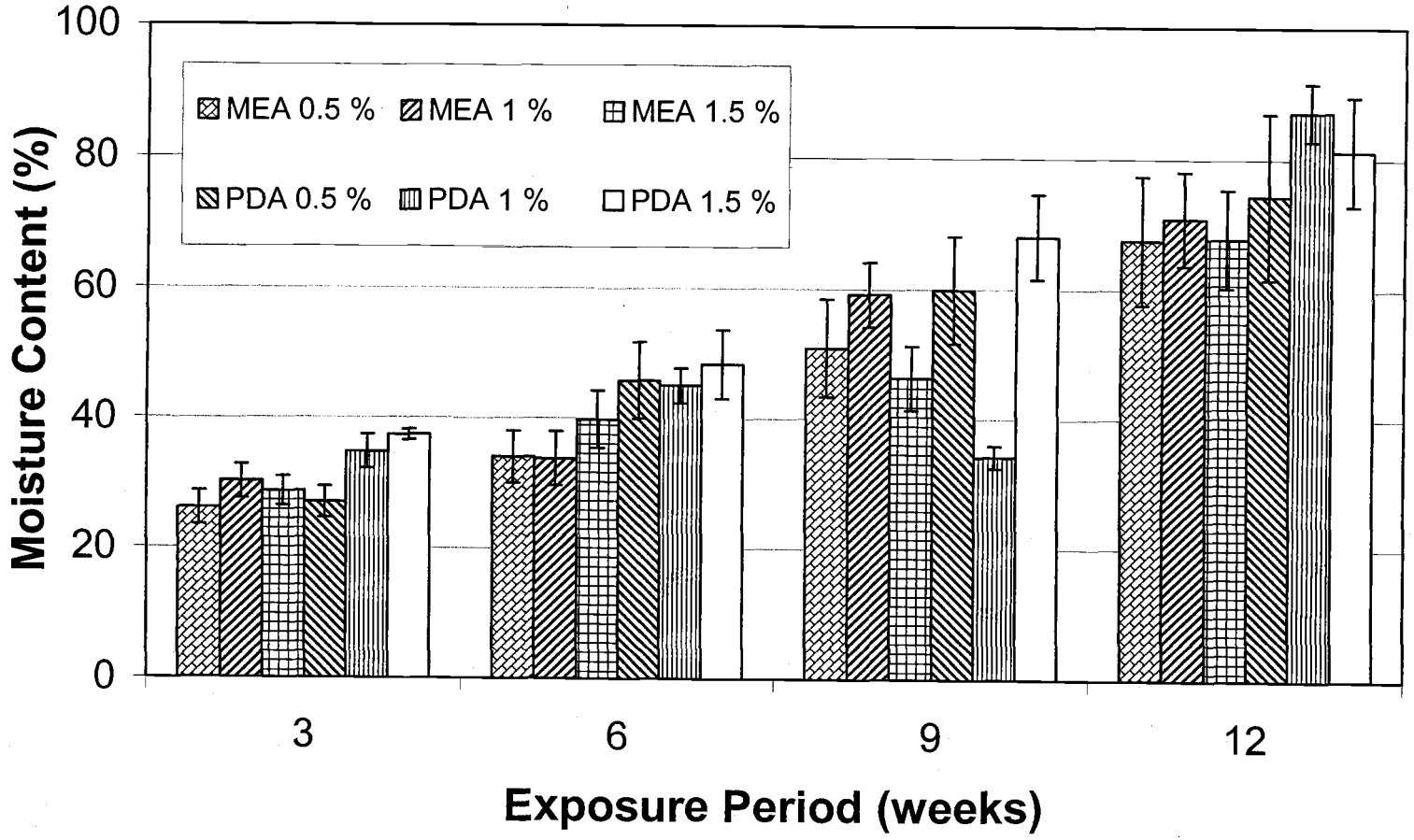

Figure 3.23 Moisture contents of the wood component in a WPC exposed for 3 to 12 weeks to *P. placenta* in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA). (Error bars represent one standard deviation).

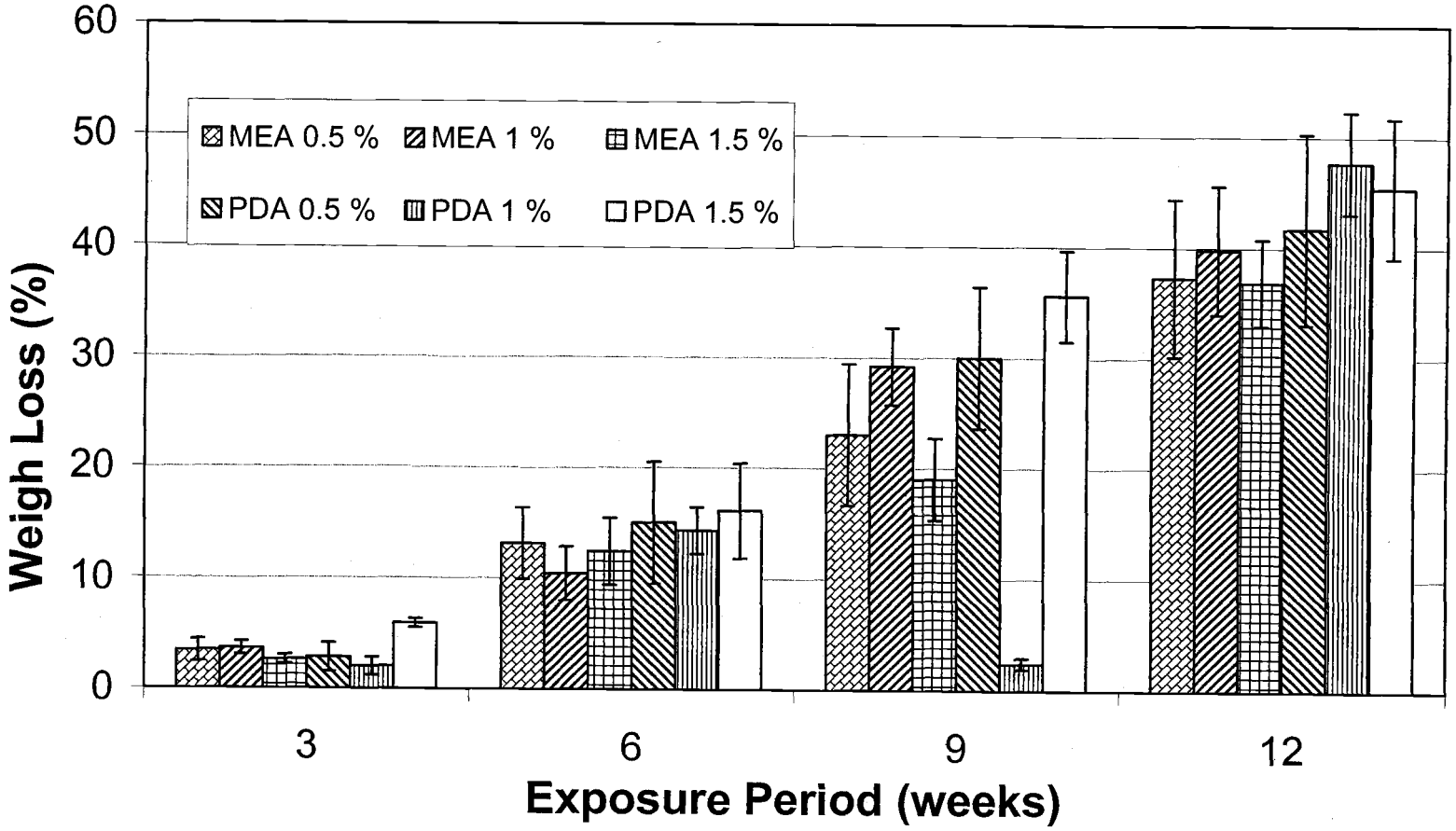

Figure 3.24 Weight losses of the wood component in a WPC exposed for 3 to 12 weeks to *P. placenta* in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA). (Error bars represent one standard deviation).

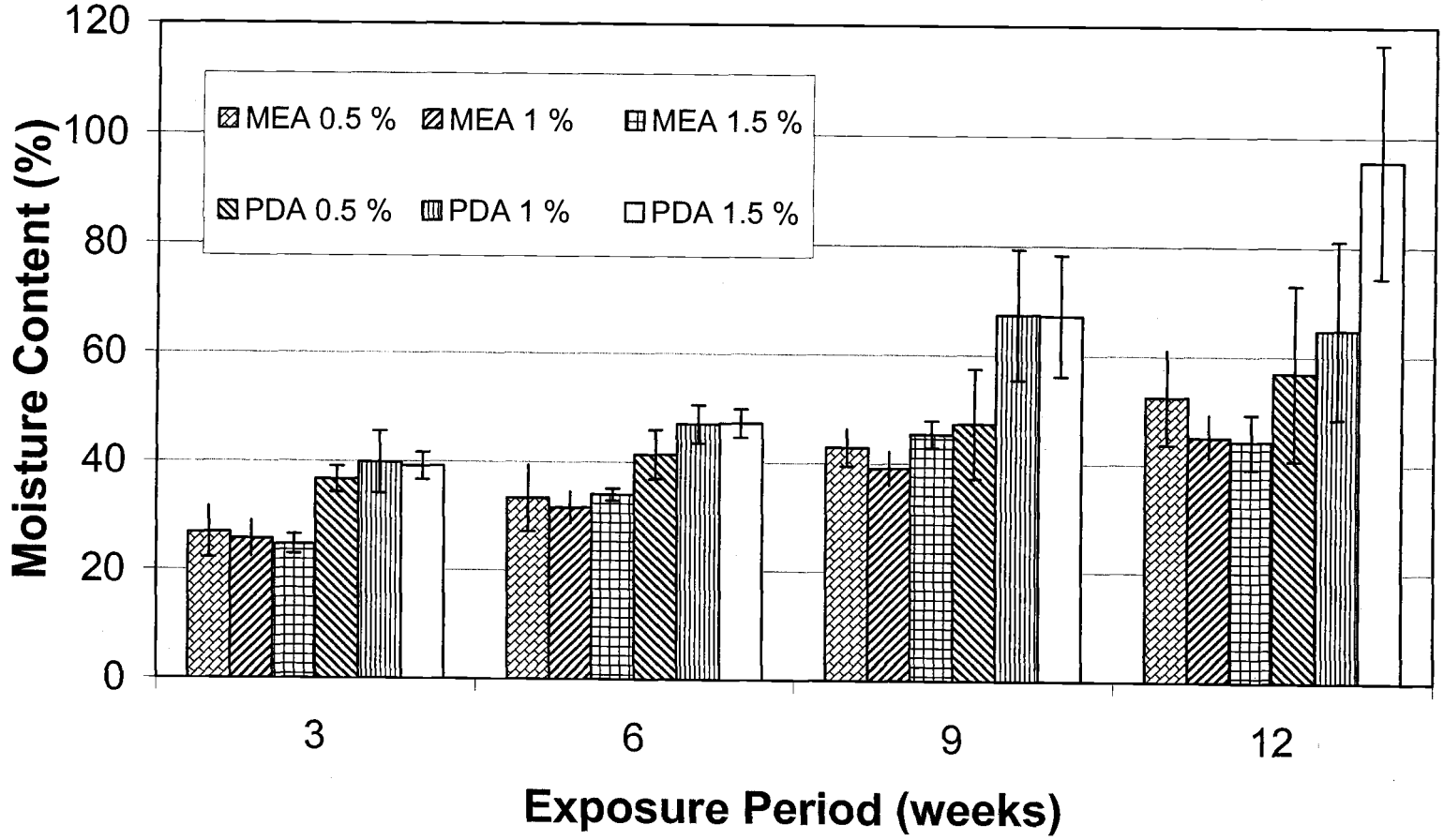

Figure 3.25 Moisture contents of the wood component in a WPC exposed for 3 to 12 weeks to *T. versicolor* in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA). (Error bars represent one standard deviation).

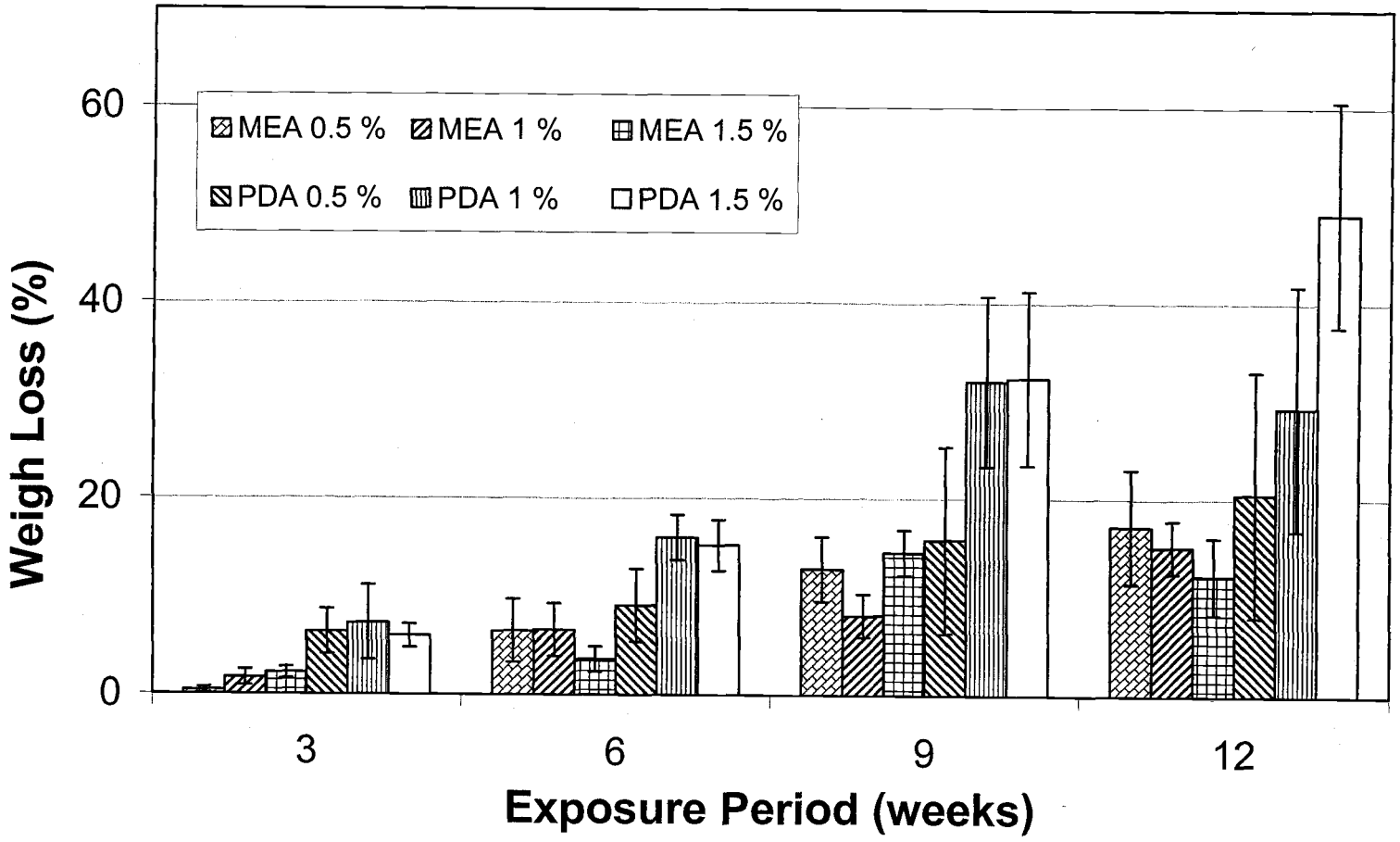

Figure 3.26 Weight losses of the wood component in a WPC exposed for 3 to 12 weeks to *T. versicolor* in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA). (Error bars represent one standard deviation).

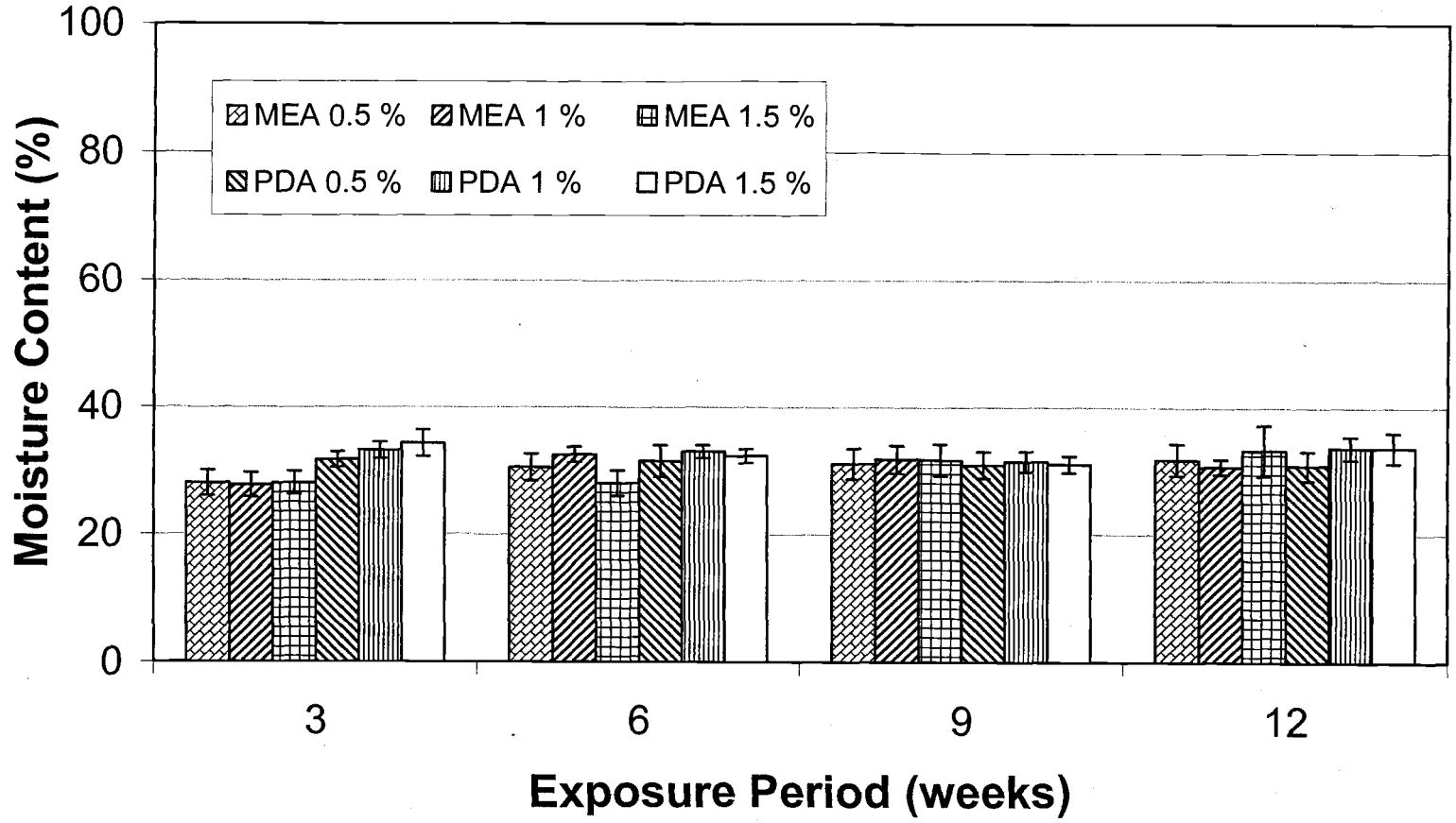

Figure 3.27 Moisture contents of the wood component in a WPC exposed for 3 to 12 weeks in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA) with no fungi. (Error bars represent one standard deviation).

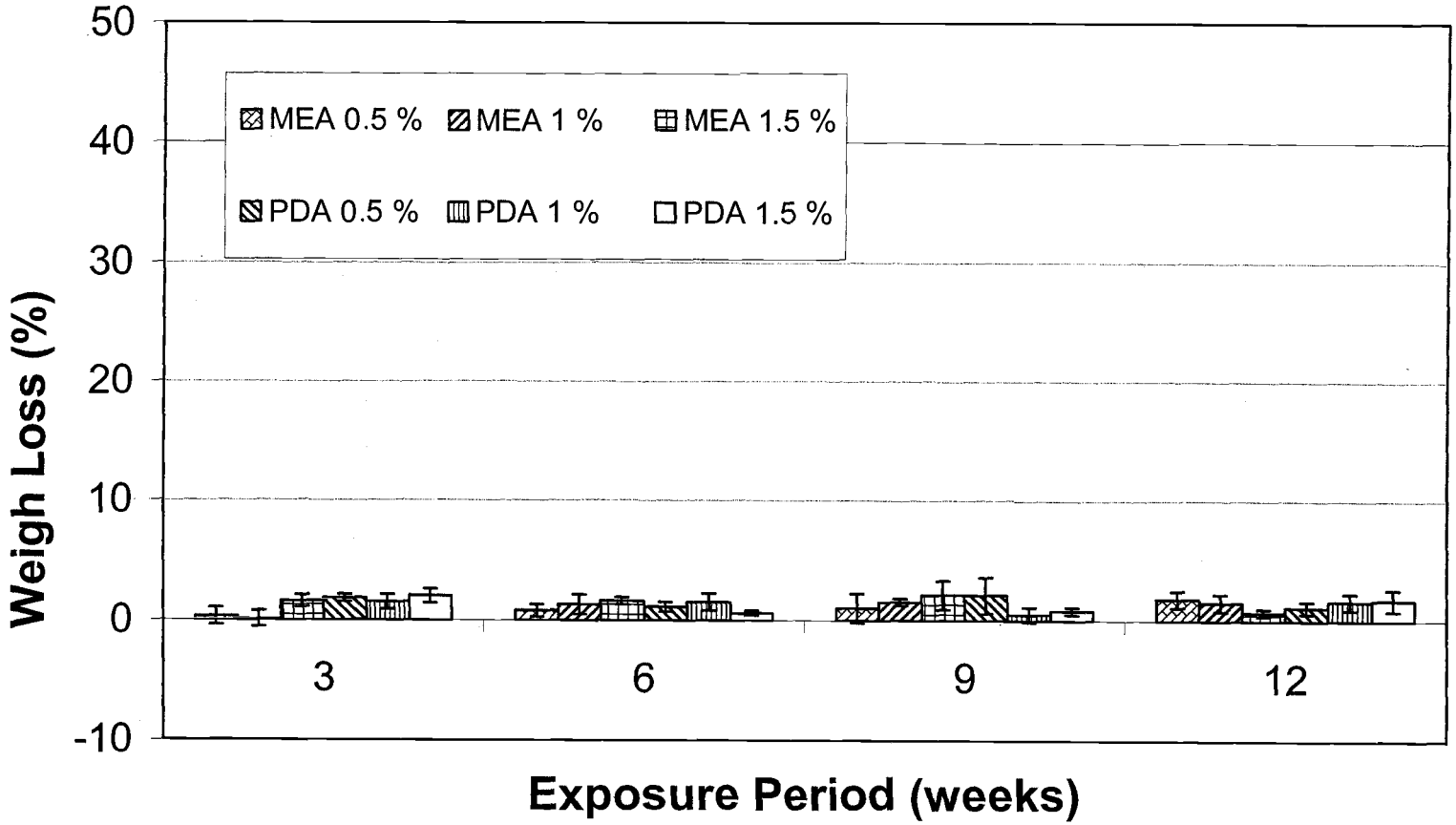

Figure 3.28 Weight losses of the wood component in a WPC exposed for 3 to 12 weeks in petri dishes containing 0.5, 1.0, or 1.5% malt extract agar (MEA) or potato dextrose agar (PDA) with no fungi. (Error bars represent one standard deviation).

Naghipour (1996) showed that WPC's had slower rates of moisture uptake than solid wood, but were permeable and, as a consequence, subject to fungal decay, particularly at high wood–polymer ratios (> 50% of wood). Peyer and Wolcott (2002) reported void formation at the interface of the wood particles and polymer following exposure to water. Clemons and Ibach (2002) and Ibach et al. (2003) reported wood moisture contents of nearly 25% for 3 mm thick WPC's made with pine and HDPE exposed for 12 weeks to *G. trabeum*. In contrast, moisture contents of WPC specimens exposed in MEA and PDA were higher than those reported in previous investigations. Wood moisture contents ranged from 80% to 96% for the WPC's exposed to *P. placenta* and *T. versicolor* in 1.5% PDA, respectively, and were nearly 70% for specimens exposed in MEA (Table 3.3).

The weight losses found in this current research (nearly 50%) were higher than those previously reported. Weight losses below 10% have been reported in most studies of WPC's exposed to fungal attack. For example, Nagiphour (1996) reported weight losses around 5% for 3 mm thick WPC's made with maple and polypropylene (PP) exposed for 12 weeks to *G. trabeum* in MEA. Clemons et al. (2002) reported weight losses approaching 6% for 3 mm thick WPC's made with pine and HDPE. Pendleton et al. (2002) reported weight losses from 4% to 8% in specimens made of 70% maple, 24% HDPE, and 6% processing additives. Verhey et al. (2001) reported higher weight losses, roughly 40%, in 3 mm thick WPC's made with 60% pine (20 mesh), and 40% PP. Mankowski and Morrell (2000) reported weight loses of nearly 16% for WPC's

made of 70% wood and 30% HDPE exposed to *P. placenta,* and about 20% weight loss for those exposed to *G. trabeum.*

The high weight losses in the current study occurred despite the small wood particle size (80 mesh). High weight losses have been associated with large particle sizes (20 to 50 mesh) and higher wood plastic ratios (>50) (Verhey et al., 2002). According to Miller (2001), the use of small wood particles appears to result in more effective encapsulation of the wood in the plastic matrix, providing better protection against both moisture uptake and fungal attack. The susceptibility of WPC's to fungal decay increases with particle sizes larger than 60 mesh (Verhey et al., 2002).

Previous studies have demonstrated that fungal attack of WPC's is concentrated on the exterior surfaces of the composite, a process that gradually roughens the composite surface (Mankowski and Morrell, 2000). The high surface area to volume ratio of the samples tested could explain the higher weight losses observed during this research. The fungal attack could also have been facilitated by delamination due to wetting that may have weakened the wood/plastic interface. In our test, maple particles on the WPC surface that were exposed directly to fungal attack were severely degraded after 12 weeks exposure. High water uptakes in the WPC samples may have been caused by the weak interface between the maple and the polypropylene in the composite. In our test, microscopic observation of selected WPC surfaces after weathering showed surface deterioration and delamination in WPC's made with maple. This problem was more severe after samples sterilization, possibly due to the heating effect. As a result, the wood component in these materials reached moisture levels

suitable for fungal attack. Separation of wood and plastic in the composite may provide a pathway for water penetration and fungal colonization.

The only effect of media concentration was observed in specimens exposed to *T. versicolor,* where the higher concentration (1.5%) significantly enhanced the decay rate. This would seem to contradict the notion that sugar depletion triggers secondary metabolism in the white rot fungus, leading to attack of all wood polymers.

3.6.2.3 Moisture content and weight loss relationship

Weight loss is an important parameter for assessing decay of solid wood; for this reason the discussion will only focus on the percentage of wood weight loss in a WPC. Fungal attack was directly related to moisture content. Increased weight loss was invariably accompanied by an increase in moisture content. In contrast, samples that did not exhibit decay had lower moisture contents (Figure 3.29).

Wood particles exposed to fungal attack on the WPC surface were largely degraded when exposed to fungal attack One consequence of this degradation would be an increase in void volume on the WPC surface which would be create more pathways for moisture to penetrate the composite to reach previously unexposed wood particles. It appears that some free water was present in the new voids created when wood particles were decayed, increasing the amount of water in the WPC. This water may also be a result of fungal respiration, or due to water transported by hyphae.

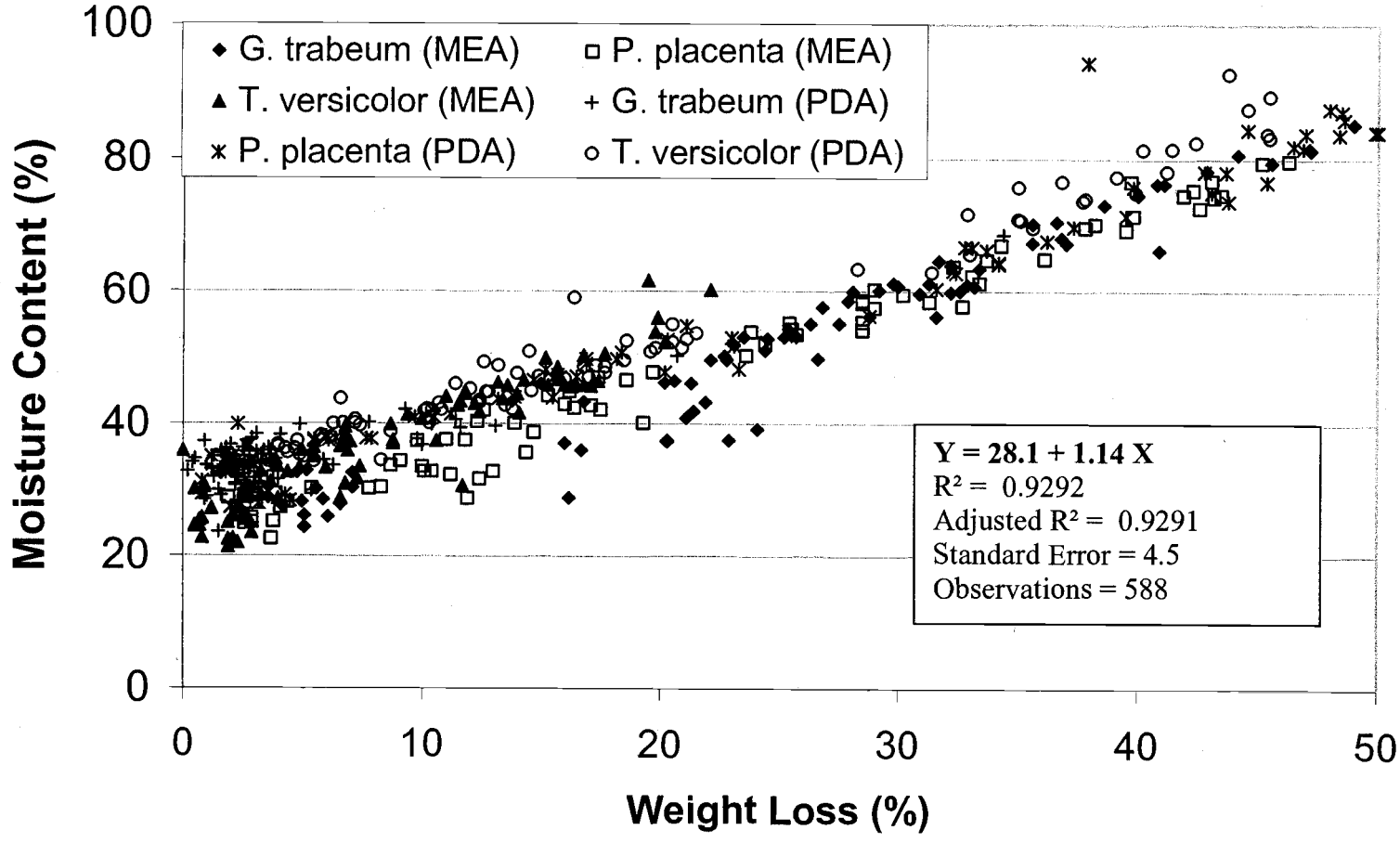

Figure 3.29 Relationship between moisture content and weight loss in WPC specimens exposed to selected decay fungi in agar tests for 3 to 12 weeks.

According to Ammer (1964), the moisture increases in decayed wood can be caused by fungal respiration that produces water (i.e., in metabolizing cell wall material), increasing the amount of free water present in the wood. The increasing moisture content may also be influenced by hyphae transport of water (Muller et al., 2001). Some fungi can transport water from high humidity media into drier specimens (WPC), creating conditions that are more suitable for fungal attack.

In our test, WPC's submerged in liquid media and water for three months reached moisture contents approaching 40% (Silva et al., 2003). These values were over the fiber saturation point of the wood (30%), and indicate that moisture conditions were generally suitable for fungal attack.

3.6.3 Conclusions

G. trabeum produced greater weight losses on MEA than in PDA regardless of media concentration. *P. placenta* produced high weight losses on both MEA and PDA at all three concentrations, while *T. versicolor* caused greater weight losses when grown on 1.5% PDA than on any other media combination tested. Both MEA and PDA were associated with enhanced moisture uptakes. The wood weight losses on either MEA or PDA for all three fungi show that both media are suitable for accelerated WPC decay tests.

3.7 Soil block tests

These tests were performed to provide results from a standard test that could be used for comparison with the results obtained in the screening and optimization tests.

3.7.1 Standard procedures and methods

These tests were carried out following ASTM Standard D1413 (ASTM, 1999) except that the samples were 10 x 20 x 0.5 mm thick, and the weight loss was measured after 3, 6, 9, and 12 weeks of exposure. In contrast, the specimen size described in the standard is 19 x 19 x 19 mm, and the incubation period is 12 weeks.

Thirty-two decay chambers were prepared, each one consisting of a 454-ml glass jars half-filled with 145 g of garden soil. The moisture content of the soil was adjusted to about 70% by adding distilled water. Small sapwood feeder strips (3 x 15 x 15 mm) cut from either western hemlock (*Tsuga heterophylla* [Raf.] Sarg), for the brown-rot fungi (*G. trabeum* or *P. placenta*), or red alder (*Alnus rubra* Bong) for the white-rot fungus (*T. versicolor*), were placed on the soil. The jars were loosely capped and sterilized for 45 min at 121 °C, allowed to cool overnight, and sterilized again for 20 minutes at 121 °C. After cooling, the feeder strips were inoculated with an agar plug cut from the actively growing edge of the appropriate test fungus. The jars were incubated at 28 °C until the mycelium nearly covered the wood surface. Seven WPC and four maple wafer samples were placed on the surface of the wood feeder strips. Each fungus/time combination was replicated in two incubation chambers. The exposure periods and test fungi for the soil block tests are shown in Figure 3.30.

3.7.2 Results and discussion

3.7.2.1 Standard procedures

Contamination was a serious issue in some of the test jars despite sterilization according to the recommended procedures (ASTM, 1999). Failures during the

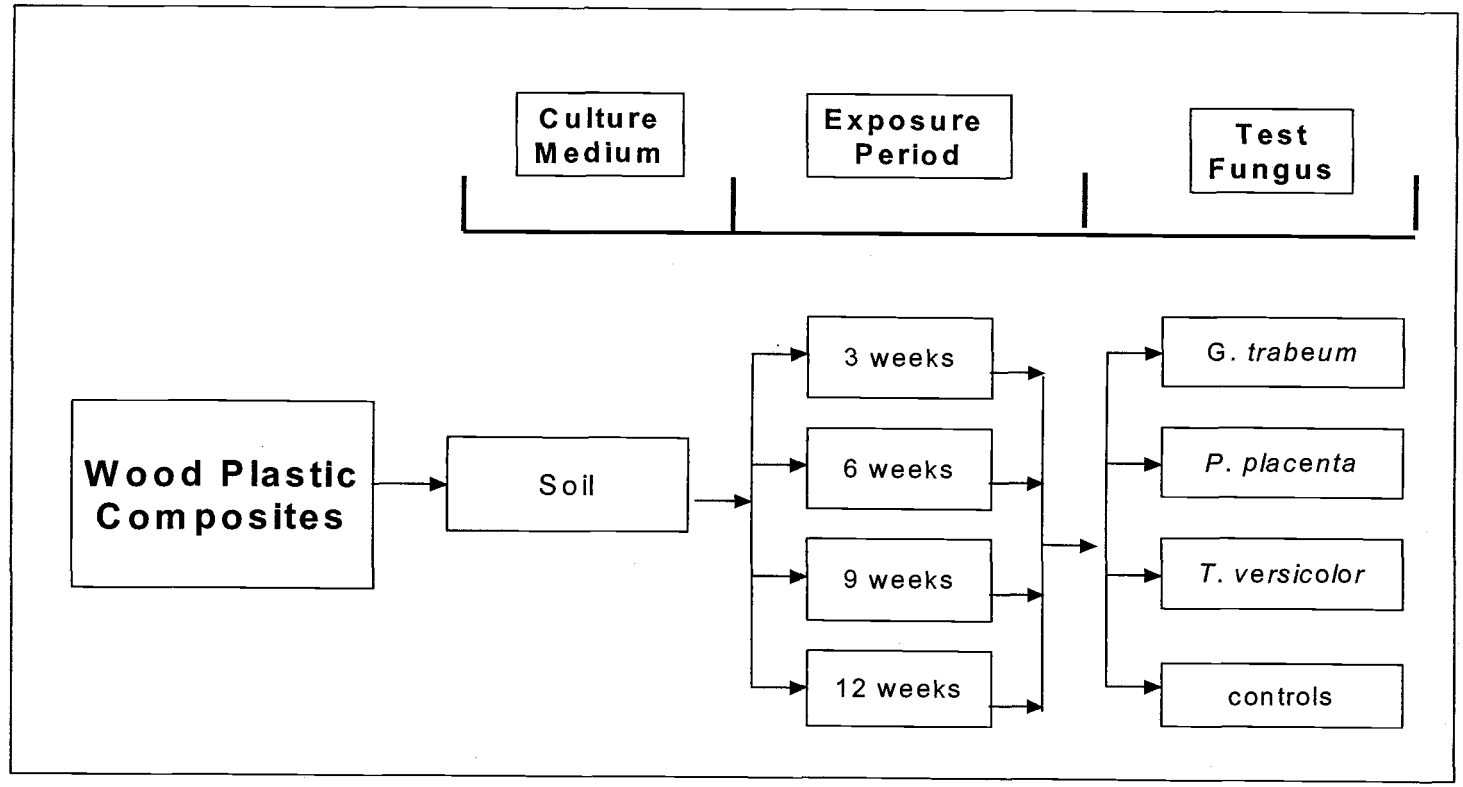

Figure 3.30 Exposure periods and test fungi combinations examined in soil block tests.

sterilization process, the inoculation of the wood feeders (when placing the agar plug next to the wood feeder), or in sample introduction may have caused this contamination

3.7.2.2 Moisture contents and weight losses

Exposure periods and test fungus both influenced the decay rates. Weight losses were nearly 17% in samples exposed for six weeks to *G. trabeum*, suggesting that the soil block test could also be a rapid screening method if thin samples are used.

Moisture contents and weight losses of the WPC's increased steadily over the 12-week test when exposed to any of the test fungi (Table 3.4, Figures 3.31 and 3.32). Moisture contents for controls were nearly 30%, while weight losses were less than 2% (Table 3.4). Brown-rot fungi were associated with significantly higher moisture contents and weight losses than the white-rot fungus; however, there were no significant differences between *G. trabeum* and *P. placenta* (Table 3.5). Solid maple wafers (serving as controls) exposed to fungal attack reached higher moisture contents (>80%) and also experienced larger weight losses (>75%) than WPC specimens.

Most previous reports indicate that WPC's reached low moisture contents in decay tests. Ibach et al., (2003) reported moisture contents of nearly 25% for 3 mm thick WPC's made of pine (40 mesh) and HDPE (50:50 wood/plastic ratio).

Simonsen and Morrell (2002) and Clemons et al. (2002) reported weight losses ranging from 5% to 10% for 2 mm thick WPC's exposed in soil block tests. Pendleton et al. (2002) reported similar results for 10 mm thick specimens made with maple exposed to *G. trabeum, P. placenta* or *T. versicolor.*

Table 3.4 Moisture contents (MC) and weight losses (WL) of the wood component in a WPC exposed for 3 to 12 weeks to *G. trabeum, P. placenta* or *T. versicolor* in soil block tests.

Test Fungus	Exposure Period [a]							
	3 wk		6 wk		9 wk		12 wk	
	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)
G. *trabeum*	34.4 (3.5)	4.9 (2.9)	44.5 (6.1)	17.1 (5.7)	57.9 (10.1)	30.0 (6.9)	64.6 (8.3)	36.1 (6.3)
P. placenta	31.6 (1.4)	2.5 (0.5)	37.9 (3.1)	9.6 (3.3)	46.1 (5.7)	18.7 (4.6)	64.8 (6.6)	35.9 (6.2)
T. versicolor	30.3 (3.5)	3.2 (1.1)	34.9 (2.8)	7.5 (1.8)	42.6 (3.9)	15.7 (3.6)	54.7 (9.5)	25.2 (5.9)
Controls	29.4 (1.5)	1.4 (0.5)	29.7 (1.9)	0.9 (0.7)	30.2 (2.3)	1.7 (1.1)	29.8 (5.7)	1.2 (1.1)

[a] Values represent means of seven samples. Values in parentheses represent one standard deviation.

Table 3.5. Moisture contents (MC) and weight losses (WL) of WPC's exposed to *G. trabeum, P. placenta,* or *T. versicolor* on agar (MEA and PDA) on soil block tests.

Fungi	Culture media	MC (%)	WL (%)
G. trabeum	soil block tests [a]	64.6 (8.3) D	36.1 (6.3) CDE
	MEA (0.5%) [b]	69.9 11.0) CD	38.3 (8) CD
	MEA (1%)	68.3 (10.1) CD	36.7 (8) CDE
	MEA (1.5%)	68.9 (7.3) CD	35.4 (5.6) DE
	PDA (0.5%) [b]	35.0 (3.6) LMNOP	6.9 (3.6) PQRS
	PDA (1%)	36.6 (2.1) KLMN	2.2 (1.6) STUVW
	PDA (1.5%)	43.3 (12.4) HIJK	12.0 (12.4) LMNOPQ
P. placenta	soil block tests	64.8 (6.6) D	35.9 (6.2) CDE
	MEA (0.5%)	67.5 (9.8) CD	37.3 (7.1) CDE
	MEA (1%)	70.8 (7.2) CD	39.8 (5.8) CD
	MEA (1.5%)	67.8 (7.5) CD	36.9 (3.9) CDE
	PDA (0.5%)	74.3 (12.7) C	41.7 (8.6) BC
	PDA (1%)	87.2 (4.4) B	47.7 (4.6) A
	PDA (1.5%)	81.2 (8.3) B	45.4 (6.3) AB
T. versicolor	soil block tests	54.7 (9.5) EF	25.2 (5.9) GH
	MEA (0.5%)	52.4 (8.7) EFG	17.2 (5.8) JKLM
	MEA (1%)	45.0 (4.2) HIJ	15.1 (2.7) KLMNO
	MEA (1.5%)	44.3 (5.0) HIJK	12.2 (3.9) LMNOP
	PDA (0.5%)	56.9 (16.1) E	20.5 (12.5) IJK
	PDA (1%)	64.8 (16.3) D	29.3 (12.5) FG
	PDA (1.5%)	95.6 (21.2) A	49.1 (11.5) A

[a] Values of the soil block tests represent means of 14 samples (7 samples x 2 replicas).
[b] MEA = malt extract agar, PDA = potato dextrose agar, n= 7. Values of the MEA or PDA tests represent means of 7 samples. Figures in parentheses represent one standard deviation. Values followed by the same letter(s) do not differ significantly by Duncan's multiple-range test ($\alpha = 0.5$).

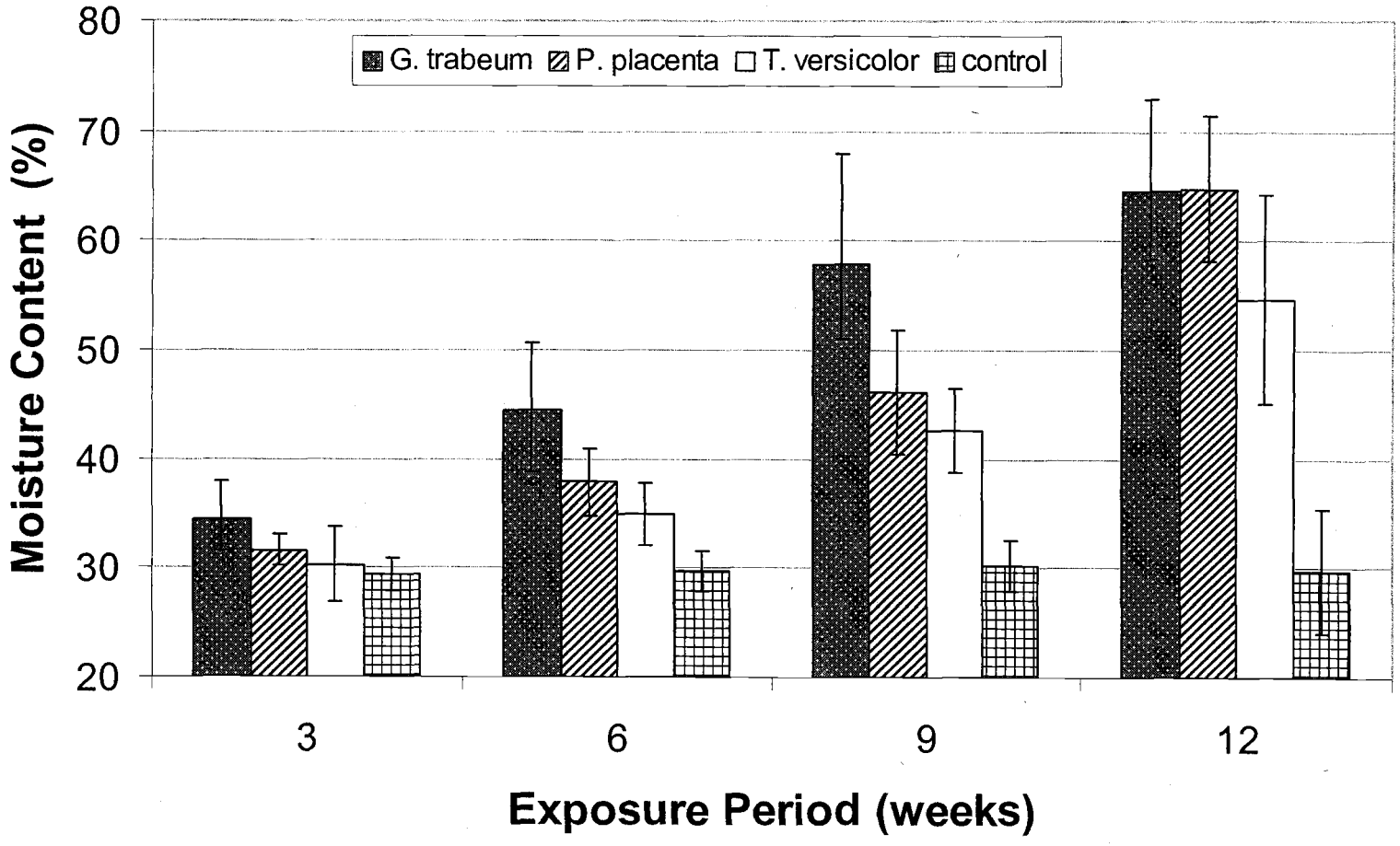

Figure 3.31 Moisture contents of the wood component in a WPC following exposure for 3 to 12 weeks to decay fungi in a soil block test. (Error bars represent one standard deviation).

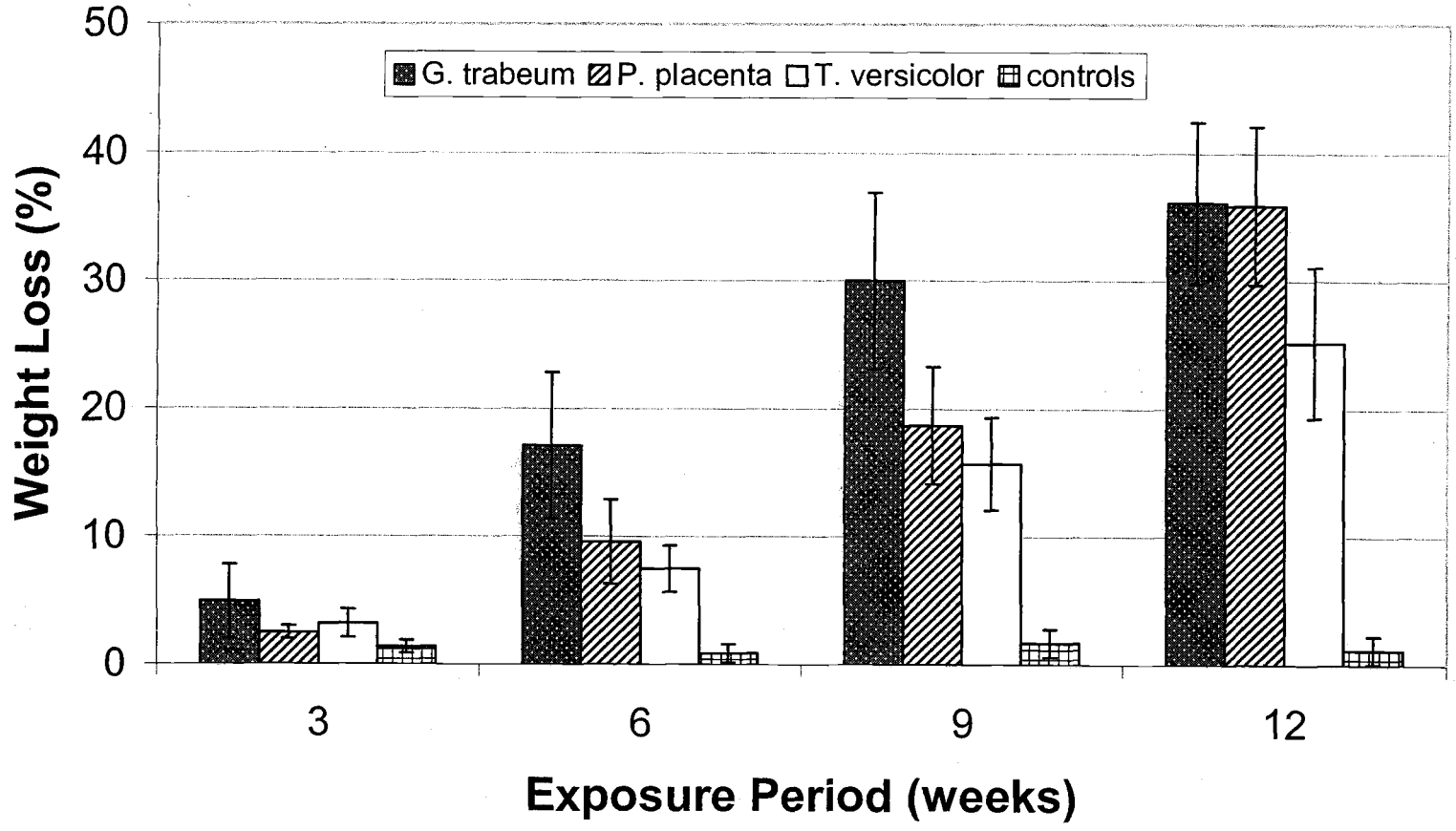

Figure 3.32 Weight losses of the wood component in a WPC following exposure for 3 to 12 weeks to decay fungi in a soil block test. (Error bars represent one standard deviation).

Defects in the wood/polymer interface induced by wetting (sample conditioning) and sterilization that increased the amount of wood directly exposed to fungal attack could account for the higher weight losses observed in this test. The thinner samples (0.5 mm) tested in our study may have enhanced the decay rate and, as a consequence, increased the moisture content. It should be noted that the higher weight losses observed in our study occurred despite the smaller particle size of the WPC's tested (80 mesh). Smaller wood particles (>60 mesh) have been associated with higher resistance to fungal attack (Verhey et al. 2001).

The soil block test was suitable for testing resistance to fungal attack of the wood in the WPC's. Observed moisture contents and weight losses suggest that conditions in the soil block test were favorable for fungal attack by both brown rot fungi, but they were less favorable for the white-rot fungus. Clearly, the moisture sorptions and weight losses observed demonstrate the susceptibility of the WPC to fungal attack.

In general, WPC specimens exposed to fungal attack in either MEA and PDA experienced significantly greater weight losses compared to those exposed in the soil block test (Table 3.5).

3.7.2.3 Moisture content and weight loss relationship

As with previous tests on agar, moisture content and weight loss were closely related for all fungi/exposure periods. Higher weight losses were directly associated with elevated moisture contents (Figure 3.33).

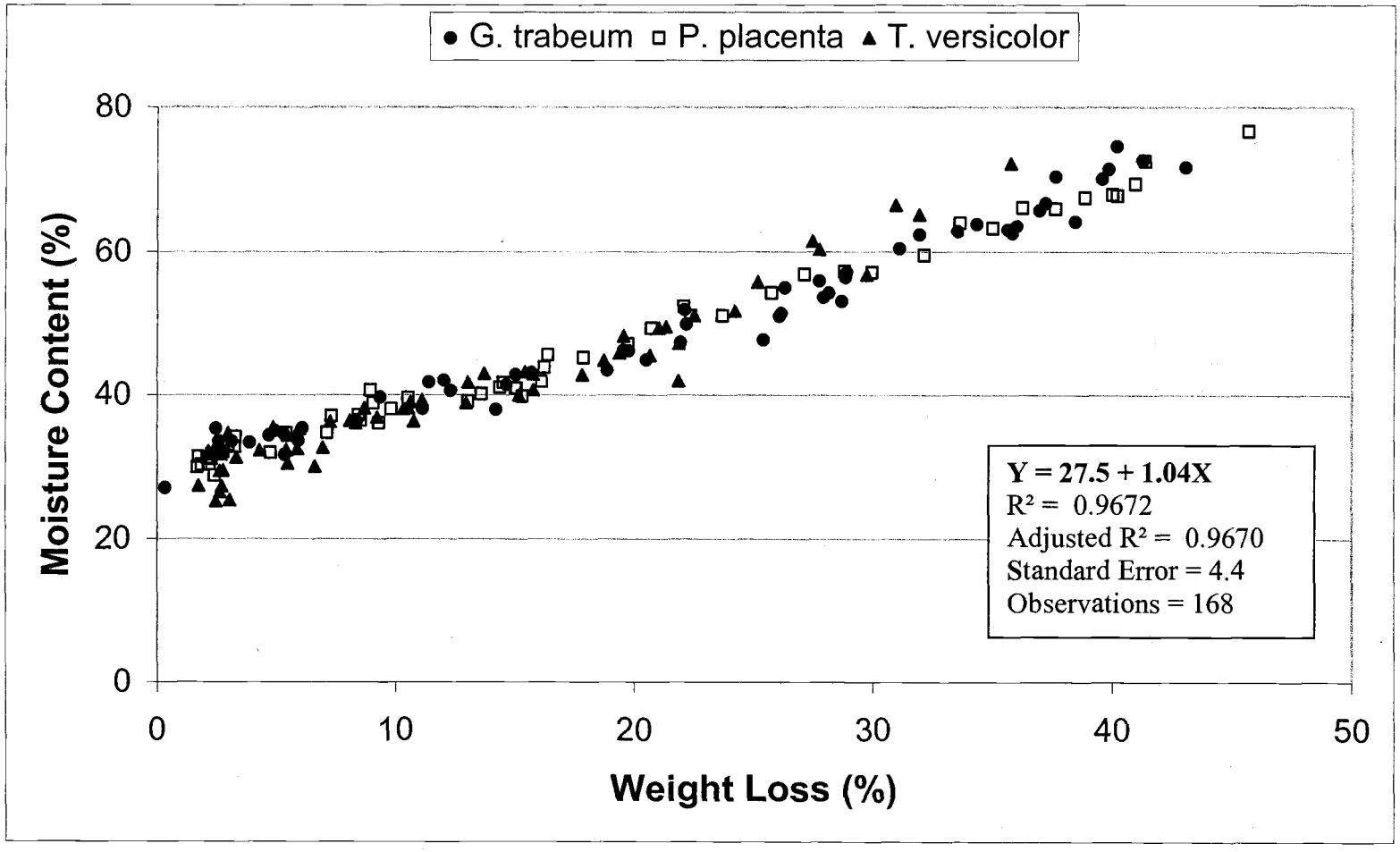

Figure 3.33 Relationship between moisture content and weight loss in WPC specimens exposed to three different decay fungi for 3 to 12 weeks in a soil block test.

3.7.3 Conclusions

Based on the rate of decay observed in the WPC (>20%) exposed to either test fungi, the soil block test was a suitable method for assessing decay resistance of WPC, although contamination was a serious issue.

3.8 Proposed criteria for classifying natural resistance to decay of WPC

Currently, there is no classification for assessing the decay resistance of WPC's. Criteria for classifying natural resistance of WPC's to fungal attack would provide a reference for discussion of the values obtained in this and other studies (Table 3.6).

The proposed classification system was based on the current classifications for natural durability of solid wood as described in ASTM Standard D 2017 (ASTM, 1999) and Standard EN 350-1 (1994). The scales were modified for WPC's because these materials exhibit different decay resistance than solid wood. The ranges for weight losses in this classification were based on a combination of values reported in previous studies (i.e., Naghipour, 1996; Mankowski and Morrell, 2000; Verhey et al., 2001; Pendleton et al., 2002, Clemons et al., 2002; Ibach et al., 2003), and also on the results from the current study.

Lower weight losses (<5%) should be expected of the wood component in a WPC when exposed to fungal attack due to the slower water uptake and plastic protection, regardless of the wood species, type of polymer, wood particle size, and wood/plastic ratios used. In our tests, a maximum weight loss of nearly 50% was observed in maple WPC specimens exposed to *T. versicolor* in 1.5% PDA for 12 weeks.

Table 3.6 Proposed criteria for classifying natural resistance of WPC's to decay fungi compared with the classification for the solid wood according standards.

Indicated Class of Resistance to a Specified Test Fungus	Durability Class	Weight loss (%)[1]	Weight loss (%)[2]	Weight loss (%)[3]
Highly resistant	I	$1 \leq 5$	$0 \leq 10$	$0 \leq 5$
Resistant	II	$6 \leq 10$	$11 \leq 24$	$5 \leq 10$
Moderately resistant	III	$11 \leq 19$	$25 \leq 44$	$10 \leq 20$
Slightly resistant or nonresistant	IV	≥ 20	≥ 45	$20 \geq 30$[4]
	V	*]	*]	> 30[5]

[1] Classification for natural resistance of wood component in the wood/plastic composite based upon this research.
[2] Classification for natural resistance of the solid wood according to American Society for Testing and Materials (ASTM) ASTM D standard designation 2017 (ASTM, 1999).
[3] Classification for natural resistance of solid wood according to European Norms (EN) designation EN 350-1 (EN, 1994).
[4] Slightly resistant
[5] Non resistant
*] Durability class (V) not considered in the classification.

Verhey et al. (2001) reported weight losses of nearly 40%, while Mankowski and Morrell (2000) reported about 20% weight loss for thicker WPC's.

The classification using maximum weight loss was based on the weight losses observed in this and in previous studies. WPC's exhibiting weight losses over 20% should be considered non-resistant to fungal attack. When designing the proposed classification it was assumed that grouping all the weight losses in values below 15% might make it difficult to separate more than two or three durability classes, while weight losses over 20% may be difficult to achieve over a 12 week exposure. As a consequence, most WPC's would be classified as very resistant to fungal attack. Also, it was assumed that the class V durability (EN, 1994) category was unnecessary because of the limited range of the scale (0% to 20%) where practical limits between "slightly resistant" or "nonresistant" would be difficult to assess and meaningless in practical terms.

Neither classification is perfect or fits all needs of the users (scientists, industry, and end users). It is evident that further work will be required to refine and standardize the method.

Despite the strong relationship between weight loss and moisture content, it was decided not to include moisture content as a parameter in the classification because moisture uptake may be less useful for assessing decay resistance if the WPC contains a biocide.

3.9 Natural durability of the WPC's tested on the agar and soil block tests

The 0.5 mm thick WPC's made of maple could be classified in durability class IV as non-resistant materials when exposed in MEA, PDA or soil block tests. All tested specimens exhibited weight losses from 20% to 49% when exposed in agar tests for 12 weeks, and 25% to 36% when exposed in soil block test regardless of the fungus or concentration media tested.

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Chapter 4

Suitability of liquid medium culture for enhancing decay of wood plastics composites

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4.1 Abstract

This study examined the suitability of malt extract broth (stationary and rotary shaker conditions) for enhancing decay of wood in a WPC exposed to brown rot and white rot fungi using Ultimate Tensile Strength (UTS), moisture content and weight loss as parameters for assessing extent of decay.

Liquid media was unsuitable for enhancing decay of wood in a WPC despite reasonable fungal growth in liquid media. UTS was a poor parameter for assessing the early stages of WPC decay.

4.2 Introduction

Wood/plastic composites (WPC's) are commodity-engineered composites that blend wood with plastic. These composites are one of the fastest growing plastics and building products market segments (Morton et al., 2003). The use of smaller wood particles, new additives, and better processing technologies allow WPC manufacturers to produce an ever increasing array of materials, often without developing a full understanding of how these materials will behave under conditions conducive to fungal attack.

Evidence suggests that wood in WPC's remains susceptible to decay (Morris et al., 1998). However, these materials exhibit slower water uptake because of the protection provided by the plastic. As a result, traditional tests for assessing decay resistance of solid wood are less suitable for testing resistance of the wood in WPC's to fungal attack.

Long exposure periods and low weight losses are major concerns when testing resistance of WPC's to fungal attack. These issues have created considerable interest in developing an accelerated method for assessing decay resistance of WPC's. Liquid media have long been used for preparing fungal cultures. Given the slow moisture sorption of WPC's, even when fully-immersed in water, we wondered if decay tests in liquid media might help to accelerate wetting and thereby increase fungal attack. Liquid media could also allow the addition of specific nutrients that encourage enzyme production, and might help transport enzymes from the medium into wood particles in the WPC. Stationary and shaker incubation phases may also be used to enhance oxygenation and the production of secondary metabolites (Carlile and Watkinson, 1994) that could enhance the decay of the wood in the WPC.

In this report, we describe tests to assess the effect of fungal attack on ultimate tensile strength (UTS), moisture content, and weight loss of a WPC exposed in liquid cultures.

4.3 Materials and Methods

4.3.1 WPC's samples

Wood/plastic pellets used to make WPC samples contained 60% (wt/wt) sugar maple (*Acer saccharum*) (ground to pass a 80 mesh screen) and 40% polypropylene. Samples were made using a 150 x 150 x 0.5 mm mold. The mold containing the pellets was heated to nearly 180 °C (350 °F) and pressed for 10 minutes at 1500 KPa, then cooled at room temperature to about 100 °C (180 °F). The resulting sheets were cut in

to 10 x 40 mm specimens using a paper cutter. These pieces were used to make dog-bone shaped tensile testing specimens, 6.6 mm (nominal) wide at the mid-point (Figure 4.1). Several sets containing 40 WPC specimens (previously cut) were assembled and cut using a drill press for making the dog-bone shaped tensile required for testing.

The dimensions of the tension specimens were selected based on the results obtained in a preliminary test (not discussed in this chapter) in which specimens measuring 0.5, 1.0 or 1.5 mm thick by 30, 40 or 50 mm long were tested in tension to determine which dimensions produced the most consistent results. The 0.5 mm thick by 40 mm length specimens produced the most consistent results. Thicker specimens tended to have knife-induced defects in the dog-bone area that increased variability in tensile strength.

All WPC samples were oven-dried at 104 °C for 24 hours, weighed to the nearest 0.0001 grams, and submerged in distilled water for 48 hours until weight gains reached nearly 30% (wt/wt), creating conditions suitable for fungal attack.

After soaking in water, the specimens were grouped in sets of ten specimens. The ends of the specimens were separated from each other with small pieces of WPC (3 mm x 10 mm). Both the specimens and separators were then secured with a rubber band (Figure 4.2). The purpose of this assembly was to provide enough open space among specimens for fungal colonization, and also to avoid overlapping among specimen surfaces, which eventually could interfere with the fungal growth.

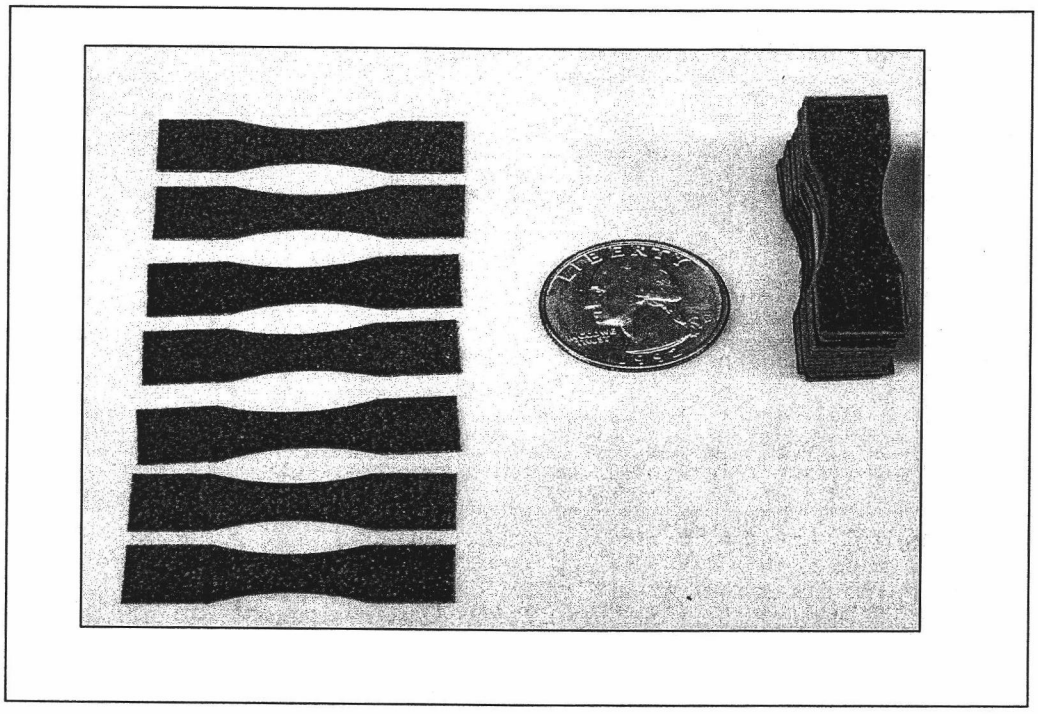

Figure 4.1 Shape of the WPC's specimens used for tension tests.

The moistened assemblies containing the WPC specimens were sterilized (immersed in containers with a small amount of water to avoid moisture loss) by heating at 121 °C for 20 minutes in an autoclave.

4.3.2 Test fungi

The brown-rot fungi *Coniophora puteana* (Schum.:Fr.) Karst (Isolate Madison 515), *Gloeophyllum trabeum* (Pers. ex. Fr.) Murr. (Isolate Madison 617) and *Postia placenta* (Fr.) M. Larsen & Lomb. (Isolate Madison 698), and the white-rot fungi *Irpex lacteus* (Fr.: Fr.) Fr. (HBB– 7328) and *Trametes versicolor* (L:Fr) Pilat (Isolate R105) were grown on 1% malt extract agar (MEA) until needed.

4.3.3 Standard procedures and methods

Ten sets of six incubation chambers were prepared by placing two 4 mm-diameter discs of each test fungus in Erlenmeyer flasks containing 50 ml of 1% malt extract broth. Two additional sets of six incubation decay chambers were left uninoculated to serve as controls. Five sets of six incubation chambers inoculated with a given fungus (one half of the total incubation chambers), and one non-fungal exposed control set were incubated under either stationary conditions or in a rotary shaker at laboratory room temperature (22-25 °C) for 21 days.

Once the fungus had grown for 21 days, one set of ten assembled WPC's samples was placed in each flask and incubated at room temperature (22°C to 25°C).

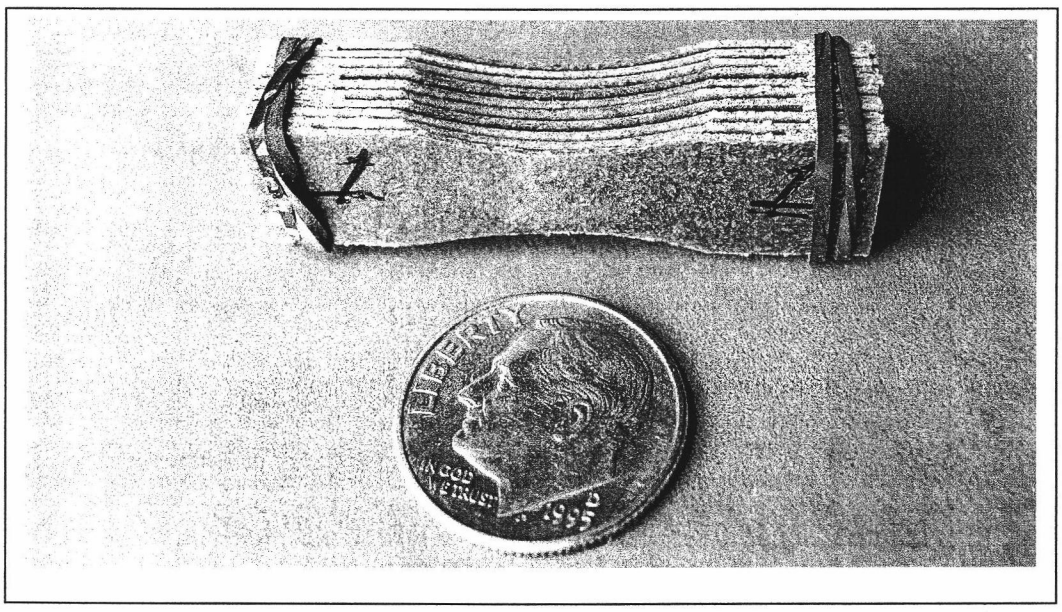

Figure 4. 2 Example of 10 WPC test samples assembled with spaces and banded together for fungal exposure.

4.3.3.1 Exposure periods

Specimens were incubated for 7, 14, 21, 31, 41 or 88 days. Sample removal was initially scheduled every seven days; however, the time intervals were extended due to the lack of decay of the wood component. At each interval, one flask with ten specimens inoculated with one of the five test fungi, and one flask containing the non-fungal exposed specimens were removed. The treatment combinations examined are shown in Figure 4.3.

4.3.3.2 Specimen sampling

The adhering mycelium and remaining malt broth were carefully removed from WPC surfaces with a dry paper towel. The specimens were weighed to the nearest 0.0001 g and then, while still wet, were immediately tested in tension until total rupture. The tensile tests were carried out according to the procedures described in the ASTM Standard D 143 (ASTM, 1999) for testing small clear specimens of timber with the following modifications: the specimens were a WPC instead of solid wood, and the dimensions were smaller (10 x 40 x 0.5 mm, 6.6 mm (nominal) wide at the mid-point of the specimen) than those listed in the standard for solid wood. The specimens were loaded in tension at a rate of 2 mm/min. The load was recorded continuously and load at failure was reported as ultimate tensile strength (UTS).

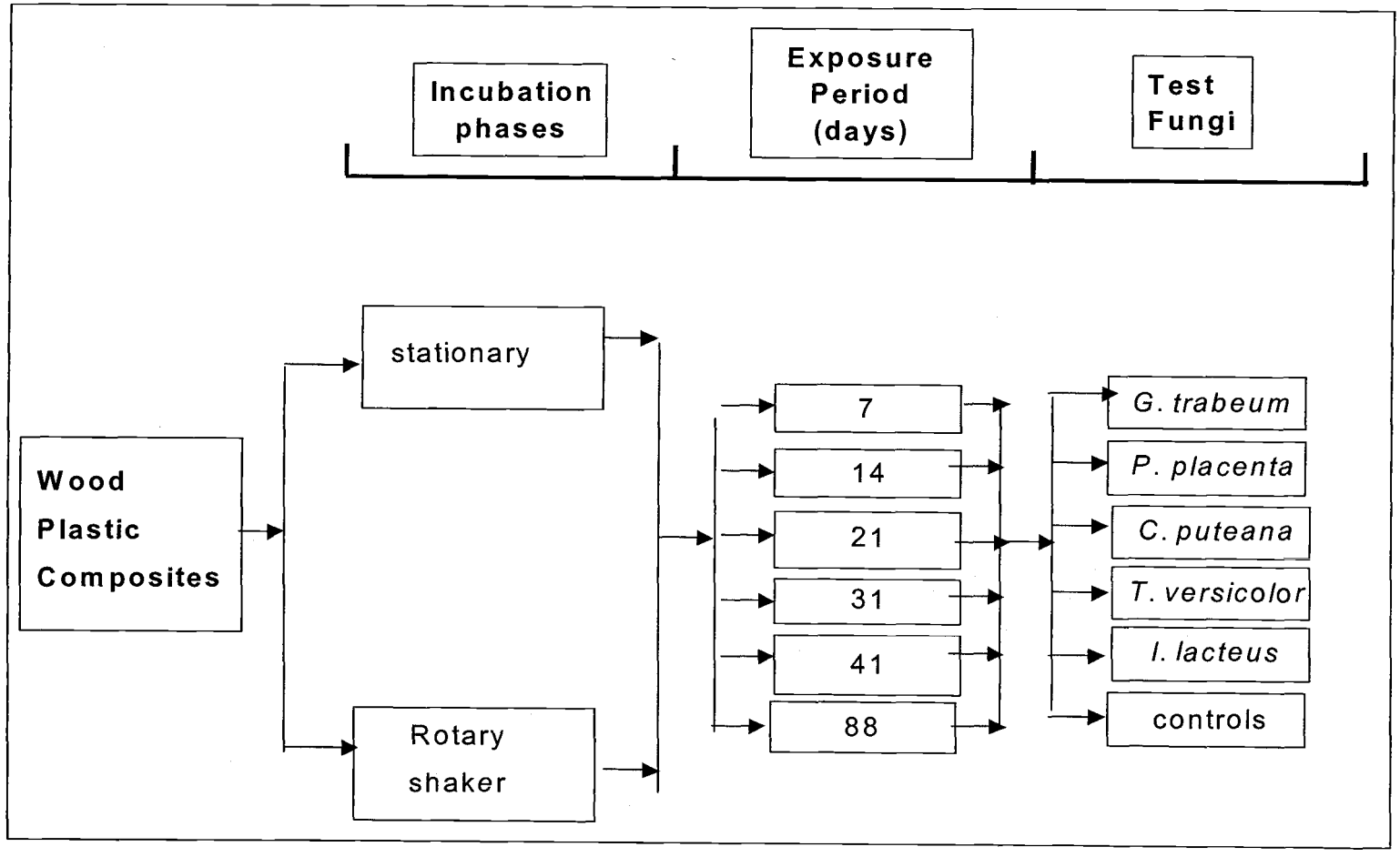

Figure 4.3 Variables employed to evaluate the ability of a liquid media to accelerate fungal attack of a WPC.

After testing, specimens were oven-dried for 24 hrs at 104 °C and weighed again to determine both moisture content and mass loss over the exposure period. Finally, a microscopic observation was performed on selected specimens for evidence of fungal attack on the WPC surface.

4.3.4 Parameters used to estimate extent of decay

Changes in ultimate tensile strength (UTS), moisture content, and weight loss of the wood in the WPC's were used to estimate the extent of decay.

Moisture content and weight loss of the WPC's were calculated based on the estimated amount of wood in the WPC's. It was assumed that the moisture uptake and fungal attack would occur in the wood, because the plastic is hydrophobic and not susceptible to attack by the test fungi (Naghipour, 1996). As a result, all moisture content and weight loss calculations were expressed in terms of the wood component in the WPC.

4.3.5 Statistical analysis

Statistical analyses were performed using Statistical Analysis System (SAS Institute Inc., 2002). The data were subject to an Analysis of Variance (ANOVA), and a General Linear Models (GLM) was used to analyze the data. Mean values were compared using Duncan's multiple-range test ($\alpha = 0.5$).

4.4 Results and discussion

Except for the WPC specimens exposed to *P. placenta* under stationary conditions, none of the WPC specimens experienced reductions in tensile strength after

being exposed to any of the five decay fungi at any time period or in either shaker or stationary incubation phase (Table 4.1 and Figure 4.4).

WPC specimens exposed to *P. placenta* for 88 days under stationary conditions exhibited incipient weight losses (Table 4.3). This result suggests that the weight loss detected might be attributed to fungal attack. No evidence of fungal attack was observed in the remaining samples regardless of the exposure period, incubation conditions, or test fungi (Tables 4.1, 4.2 and 4.3, and Figures 4.5 and 4.6).

4.4.1 Procedures and methods

No major problems were found with the procedures with regard to experiment set up or culture contamination. An initial weight loss of nearly 4% was detected after WPC sample sterilization and prior to fungal exposure. This initial weight loss may be explained by extractive losses in the water in which they were immersed during sterilization. The initial weight losses observed in our test were similar to the amount of extractives reported for maple (Koelling et al., 1996).

4.4.2 Ultimate tensile strength (UTS)

None of the test fungi nor the control treatment induced detectable changes in the UTS of WPC specimens (Table 4.1). Using strength loss instead of weight loss in a field test, Verhey et al. (2001) showed that modulus of rupture (MOR) decreased with exposure time. However, this decrease was totally attributed to moisture uptake by the WPC. Despite ideal moisture levels for fungal attack (nearly 60%), no evidence of decay was detected on WPC stakes (Verhey et al., 2003).

It appears that the liquid media were unable to support fungal attack of the specimen. The lack of fungal effects on tensile strength may have several causes. First, nutrient solutions may have limited fungal growth, but this appears unlikely because fungal mats were evident in stationary cultures. Second, low oxygen availability may have limited fungal growth or conditions may have been unsuitable production of wood degrading enzymes.

According to Carlile and Watkinson (1994), the growth of filamentous fungi cultivated in liquid media could be improved by agitation of the media, which provides oxygen for fungal metabolic activity, such as production of secondary metabolites. Regardless of the benefits of the shaker cultures for improving aeration and increased fungal growth, in our test we found no evidence of fungal attack in any specimen exposed in rotary shaker conditions.

4.4.3 Moisture content and weight loss

Except for the specimens exposed to *P. placenta* under stationary conditions, neither incubation condition (stationary or rotary shaker) nor test fungi were associated with sifnificant changes in the moisture content or weight losses of the WPC (Tables 4.2 and 4.3, and Figures 4.5 and 4.6). Fungal mycelium covered almost all exposed WPC's and induced color changes of the colonized surfaces after 41 days exposure. However, fungal colonization did not enhance moisture content or weight loss. It appears that fungi only used the specimens as a matrix upon which to grow in the liquid media.

Table 4.1 Ultimate tensile strength (UTS) of WPC's exposed to 5 decay fungi for 7 to 88 days in malt extract broth in shake or stationary cultures.

Fungus	Culture conditions	Ultimate Tensile Strength (N/mm^2) [a]					
		7 days	14 days	21 days	31 days	41 days	88 days
G trabeum	stationary	13 (1.8)	15 (2.4)	14.5 (1.9)	13.7 (1.2)	14.8 (1.7)	13.5 (2.5) AB
G. trabeum	rotary shaker	12.8 (1.9)	13 (1.9)	13.4 (2.2)	14.3 (1.1)	14.4 (0.9)	15.4 (1.9) A
P. placenta	stationary	13.8 (1.8)	13.8 (1.6)	13.8 (2.4)	13.8 (1.3)	13.4 (1.9)	11.2 (1.4) C
P. placenta	rotary shaker	13.5 (2.2)	12.9 (1.7)	13.8 (2.1)	14.0 (1.6)	14.2 (1.8)	13.9 (2.5) AB
T. versicolor	stationary	12.3 (3.2)	12.1 (3.4)	14.6 (1.4)	14.0 (1.5)	13.9 (1.5)	14.2 (1.9) AB
T. versicolor	rotary shaker	11.7 (2.2)	12.9 (2.4)	13.5 (1.0)	13.2 (1.8)	14.5 (1.9)	13.3 (2.2) B
C. puteana	stationary	12.6 (1.7)	15.1 (1.6)	13.7 (1.8)	14.1 (2.7)	ND [b]	13.6 (1.4) AB
C. puteana	rotary shaker	14.6 (2.0)	13.9 (1.9)	14.3 (1.2)	14.4 (1.2)	ND [b]	14.1 (3.3) AB
I. lacteus	stationary	14.7 (1.7)	14 (1.4)	13.9 (1.1)	ND [b]	ND [b]	14.3 (1.1) AB
I. lacteus	rotary shaker	13.0 (1.4)	14.3 (1.4)	13.6 (1.5)	ND [b]	ND [b]	15.1 (1.2) AB
Controls	stationary	11.7 (3.6)	14.2 (1.0)	13.4 (1.5)	14.3 (1.5)	14.6 (1.3)	14.6 (1.2) AB
Controls	rotary shaker	12.7 (1.8)	13.4 (1.7)	14.0 (2.0)	13.9 (1.9)	14.2 (1.6)	15.1 (2.1) AB

[a] Values represent means of ten samples per treatment. Values in parentheses represent one standard deviation.
[b] ND = Not determined. Value followed by the same letter(s) do not differ significantly by Duncan's multiple-range test ($\alpha = 0.5$).

Surface deterioration and delamination in WPC's have been associated with moisture weathering (Naghipour, 1996; Peyer and Wolcott, 2002). Separation of wood and plastics, and induced cracks in the wood-plastic interface provide pathways for water penetration and fungal colonization. Previous studies demonstrated that the fungal activity in decayed WPC's was concentrated on the surfaces, a process that gradually roughens the composite surface (Mankowski and Morrell, 2000). This decay pattern was not observed in our test. In our test, microscopic observation of the surfaces of selected WPC's before and after fungal exposure showed physical deterioration and delamination. These factors apparently did not influence the decay of WPC's.

Immersion in fungal inoculated 1% malt broth for three months did not result in significant increases in moisture content of the samples (>30%) when compared to those immersed in liquid without the test fungi, although moisture levels in all WPC specimens (nearly 30%) were favorable for fungal attack.

WPC specimens tended to experience low weight losses after being exposed to fungal attack regardless of the wood/plastic ratio, wood species, and composite thickness. Naghipour (1996) reported weight loss below 5% for 3 mm thick WPC's made with maple and polypropylene (60:40 wood plastic ratio) exposed to *G. trabeum* in MEA. Clemons and Ibach (2002) and Ibach et al. (2003) reported weight losses ranging from 6% to 10% when exposed to *G. trabeum*. These losses appear to be due to leaching rather than to any biological effect.

Table 4.2 Moisture contents (MC) of WPC's exposed to 5 decay fungi for 7 to 88 days in malt extract broth in shake or stationary cultures.

Fungus	Culture conditions	Moisture content (%) [a]					
		7 days	14 days	21 days	31 days	41 days	88 days
G trabeum	stationary	30.7 (1.3)	29.7 (2.1)	31.8 (1.6)	32.6 (1.4)	30.7 (1.3)	30.6 (1.2) AB
G. trabeum	rotary shaker	31.0 (0.8)	30.4 (1.5)	32.6 (1.5)	30.6 (1.3)	31.0 (0.8)	31.0 (1.7) AB
P. placenta	stationary	31.3 (1.2)	30.9 (1.6)	32.4 (1.4)	31.3 (1.4)	31.3 (1.2)	28.7 (3.1) C
P. placenta	rotary shaker	30.8 (1.5)	33.3 (1.5)	32.5 (1.2)	30.8 (1.1)	30.8 (1.5)	31.2 (1.9) A
T. versicolor	stationary	31.0 (1.1)	32.8 (1.1)	31.7 (1.1)	31.6 (1.3)	31.0 (1.1)	30.8 (1.8) AB
T. versicolor	rotary shaker	32.0 (1.1)	33.0 (1.4)	33.6 (1.5)	31.9 (1.2)	32.0 (1.1)	31.5 (1.5) A
C. puteana	stationary	31.6 (1.1)	32.8 (0.6)	31.6 (1.7)	ND [b]	31.6 (1.1)	30.4 (1.5) AB
C. puteana	rotary shaker	31.2 (1.0)	33.7 (2.2)	31.4 (1.4)	ND [b]	31.2 (1.0)	30.3 (1.3) AB
I. lacteus	stationary	33.8 (1.8)	31.8 (1.0)	ND [b]	ND [b]	33.8 (1.8)	30.8 (1.2) AB
I. lacteus	rotary shaker	35 (1.8)	32.1 (1.6)	ND [b]	ND [b]	35 (1.8)	31.0 (1.2) AB
Controls	stationary	30.7 (1.8)	31.4 (1.5)	32.0 (1.9)	32.0 (1.0)	30.7 (1.8)	29.3 (1.0) BC
Controls	rotary shaker	31 (1.2)	31.3 (1.1)	30.3 (1.5)	30.7 (1.4)	31 (1.2)	30.6 (1.7) AB

[a] Values represent means of ten samples per treatment. Values in parentheses represent one standard deviation.
[b] ND = Not determined. Value followed by the same letter(s) do not differ significantly by Duncan's multiple-range test ($\alpha = 0.5$).

Table 4.3 Weight losses (WL) of WPC's exposed to 5 decay fungi for 7 to 88 days in malt extract broth in shake or stationary cultures.

Fungus	Culture conditions	Wood weight loss (%) [a]					
		7 days	14 days	21 days	31 days	41 days	88
G trabeum	stationary	4.1 (0.6)	4 (0.9)	5.3 (0.6)	4.0 (0.9)	4.1 (0.6)	4.5 (0.6) C
G. trabeum	rotary shaker	3.9 (0.4)	4.2 (0.9)	4.9 (0.8)	4.0 (0.8)	3.9 (0.4)	4.1 (0.7) CD
P. placenta	stationary	4.1 (0.5)	4.6 (0.5)	5.0 (0.4)	4.0 (0.6)	4.1 (0.5)	9.4 (2.2) A
P. placenta	rotary shaker	4.3 (0.6)	3.9 (0.7)	3.6 (0.3)	3.7 (0.4)	4.3 (0.6)	3.6 (0.9) D
T. versicolor	stationary	4.3 (0.7)	4.4 (0.4)	4.6 (0.6)	4.1 (0.6)	4.3 (0.7)	5.8 (0.7) B
T. versicolor	rotary shaker	3.8 (0.4)	4.2 (0.7)	4.2 (0.5)	4.4 (0.7)	3.8 (0.4)	5.7 (0.8) B
C. puteana	stationary	4.4 (0.5)	4.6 (0.3)	4.2 (0.5)	ND [b]	4.4 (0.5)	4.3 (0.5) CD
C. puteana	rotary shaker	4.6 (0.3)	3.7 (0.3)	3.8 (0.4)	ND [b]	4.6 (0.3)	4.0 (0.5) CD
I. lacteus	stationary	3.5 (0.5)	3.8 (0.3)	ND [b]	ND [b]	3.5 (0.5)	4.1 (0.6) CD
I. lacteus	rotary shaker	3.7 (0.5)	4.4 (1.4)	ND [b]	ND [b]	3.7 (0.5)	3.9 (0.7) CD
Controls	stationary	3.8 (0.7)	4.4 (0.6)	4.5 (0.7)	3.9 (0.4)	3.8 (0.7)	4.0 (0.7) CD
Controls	rotary shaker	4.3 (0.4)	4.2 (0.4)	4.9 (0.7)	4.6 (0.4)	4.3 (0.4)	3.9 (0.5) CD

[a] Values represent means of ten samples per treatment. Values in parentheses represent one standard deviation.
[b] ND = Not determined. Value followed by the same letter(s) do not differ significantly by Duncan's multiple-range test ($\alpha = 0.5$).

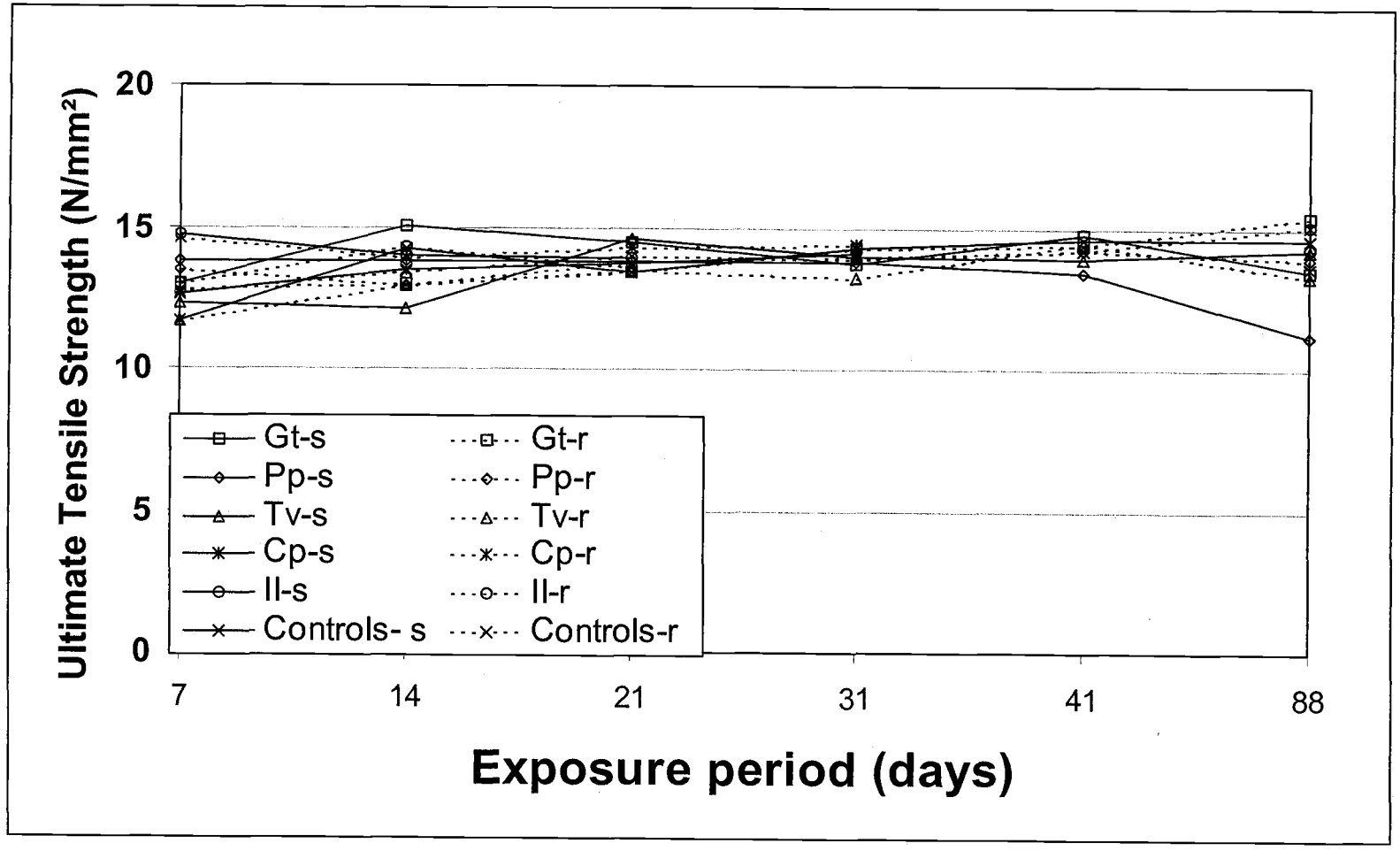

Figure 4.4 Effect of exposure to five different decay fungi for 7 to 88 days in malt extract in shake or stationary culture on ultimate tensile strength of a WPC.

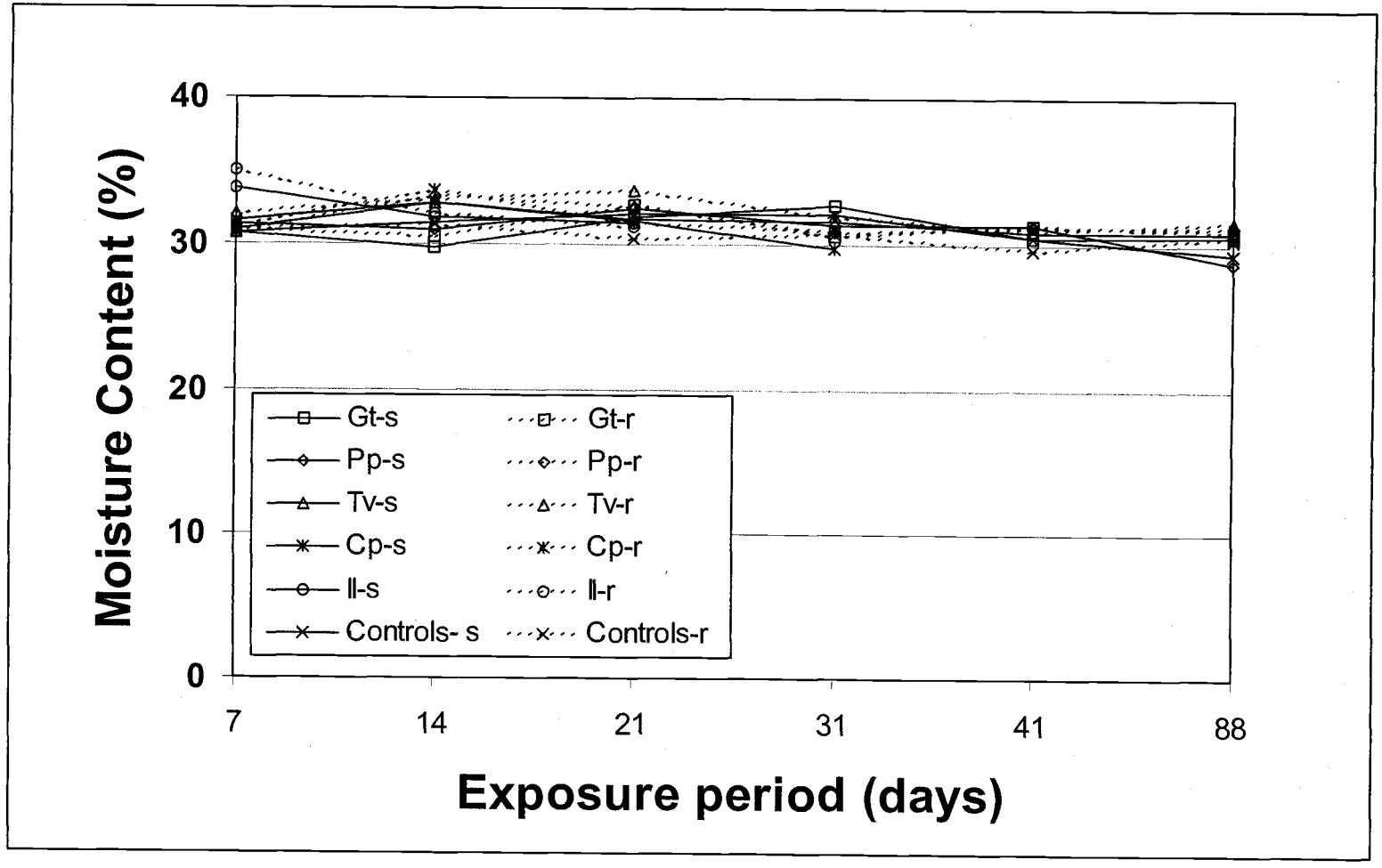

Figure 4.5 Effect of exposure to five different decay fungi for 7 to 88 days in malt extract in shake or stationary cultures on moisture content of a WPC.

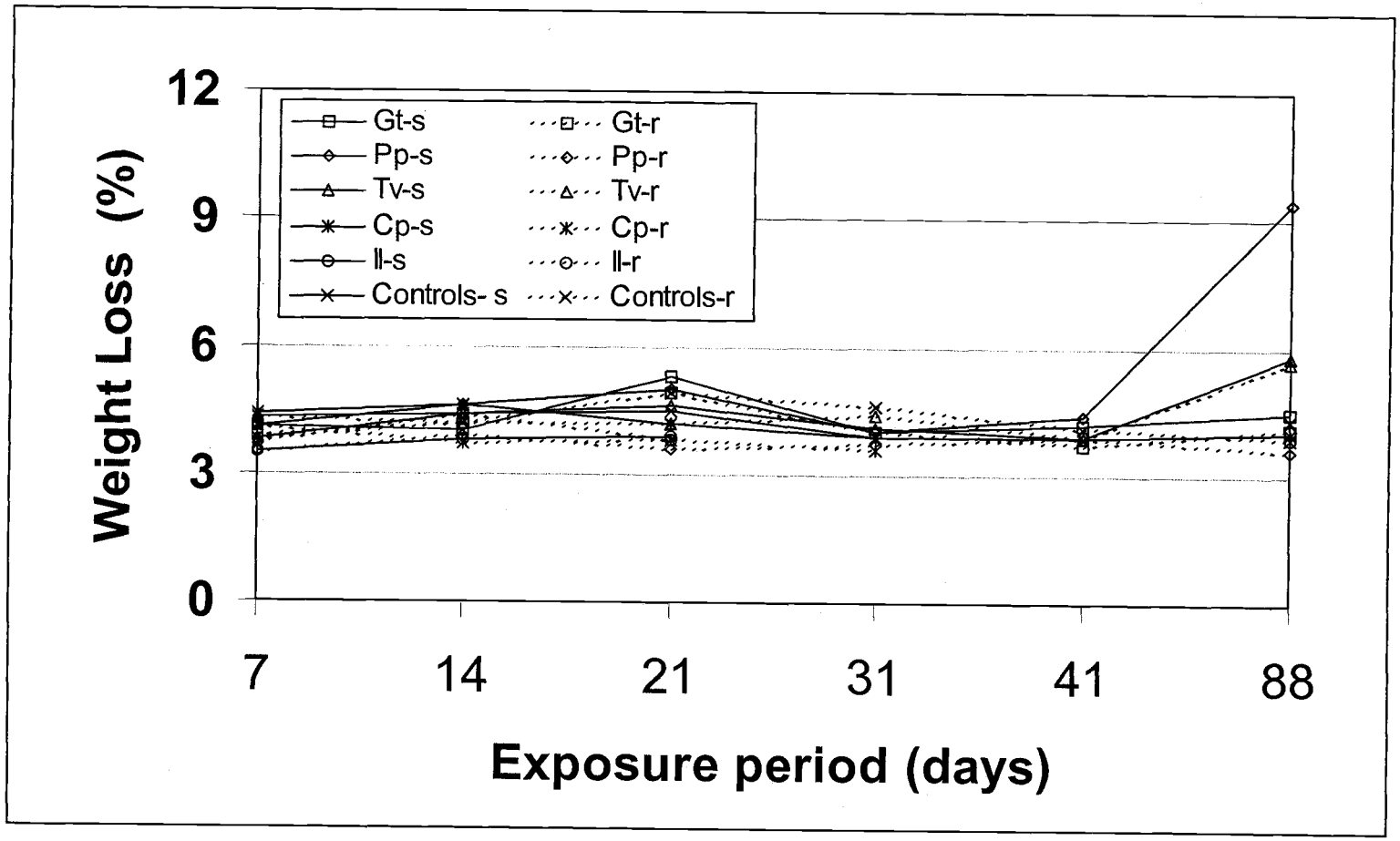

Figure 4.6 Effect of exposure to five different decay fungi for 7 to 88 days in malt extract in shake or stationary cultures on weight loss of a WPC.

According to Scheffer and Livingston (1937), wood decay is more rapid under aerated conditions. Decay stops in very wet wood regardless of nutrient availability, temperature, and favorable conditions for fungal attack due to oxygen deficiency (Scheffer and Livingston, 1937). Most wood-inhabiting fungi are unable to grow effectively in water-saturated wood because they are obligate aerobic organisms (Zabel and Morrell, 1992). Decay development ceases when the volume of air in the wood reaches 20% of the original void volume (Zabel and Morrell, 1992).

In our test, the moisture content of all the WPC specimens was slightly over the fiber saturation point (FSP) (>30%). This moisture level in the WPC's indicates that the wood fibers may not have reduced the original level of air to a level closer to 20%, level required for inhibiting the decay process (Zabel and Morrell, 1992). However, it appears that lack of oxygen or other environmental factors in the liquid media severely limited fungal growth.

According to Highley (1973), the following factors may be responsible for the inability of culture filtrates of brown-rot fungi to degrade highly ordered cellulose: culture conditions did not permit induction of the appropriate enzyme or fungal secretions inhibited fungal attack.

We originally thought that enzymes secreted in liquid culture might enhance decay of the WPC surface. Additionally, it was assumed that liquid media might help transport enzymes from the medium into the unexposed wood particles in the WPC. No evidence of fungal attack was detected in any WPC specimen tested, except for the

specimens exposed to *P. placenta* under stationary conditions for 88 days. It appears that conditions in the liquid medium were unsuitable to produce decay of WPC's. These results suggest that other media and exposure conditions should be used to assess fungal attack on the WPC's.

4.5 Conclusions

Evidence from this study suggests that liquid medium was unsuitable for enhancing decay of the WPC. The liquid media conditions tested failed to produce an environment that was conducive to enzyme production by the selected test fungi. Malt extract broth is a relatively nutrient rich media and it is possible that excess nutrients repressed enzyme activity by some fungi. However, it is doubtful that the effect was universal and it appears more likely that oxygen conditions in the media were not suitable for extensive decay of WPC's.

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Chapter 5

Effect of wood species and composite thickness on decay rate of wood polypropylene composites (WPC's)

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5.1 Abstract

The effect of wood species (maple and pine) and composite thickness on decay resistance of the wood in a WPC was assessed using brown rot and white rot fungi on 1% malt extract agar.

The results indicated that WPC's made with maple were more susceptible to fungal attack than those made with pine, regardless of the test fungus. Increases in composite thickness from 0.5 mm to 2.5 mm sharply reduced rates of decay. Thicker WPC specimens experienced slow moisture uptake and lower weight losses.

The results indicated that WPC specimens with a high surface to volume ratio would be more susceptible to fungal attack.

5.2 Introduction

The resistance of wood/plastic composites (WPC's) to fungal attack is currently the subject of much debate due to emerging reports showing that the wood in these materials is susceptible to fungal attack, especially when exposed to adverse environments or moisture sources. Previous studies have demonstrated that decay fungi can colonize and degrade wood at the composite surface regardless of wood species, particle size, or polymer type present in the composite (Mankowski and Morrell, 2000; Miller, 2001; Pendleton et al., 2002; Morris et al., 1998). In most cases, however, the fungi have been unable to penetrate the composite matrix because the plastic provides a protective barrier that totally or partially covered the wood particles. This barrier restricts moisture movement, diminishing or eliminating paths for fungal colonization of the composite.

Another factor that may affect overall susceptibility of WPC's to fungal attack is the dimension of the composite. A variety of softwoods or hardwoods can be used to manufacture WPC's. The natural resistance of wood to fungal attack varies among species depending on their density, permeability, and chemical composition, and importantly, their content and toxicity of heartwood extractives. The potential resistance to fungal attack of the WPC will also depend upon these factors.

The thickness of the composite may influence wetting rates and, potentially, could allow formation of layers with a range of densities that could permit differential fungal penetration. WPC's that maximize surface to volume ratios will result in conditions more suitable for decay development (Naghipour, 1996; Wang and Morrell, 2003). Large moisture gradients from the surface to the core of the composite would largely restrict fungal attack to the surface layer (Mankowski and Morrell, 2000).

The objective of this study was to evaluate the effect of two wood species (maple and pine) and variable specimen thicknesses on decay resistance of the wood in a WPC.

5.3 Materials and methods

5.3.1 WPC and solid wood samples

Wood/plastic pellets used to make WPC samples contained either 60% pine (*Pinus* sp., ground to pass a 60 mesh screen) or 60% sugar maple (*Acer saccharum*, ground to pass a 80 mesh screen) and 40% polypropylene (Northwood Plastic, Inc., Chavoyan, WI). Wood/plastic composites with thicknesses of 0.5, 2.5, 5.0, and 8.0 mm were fabricated in the laboratory using a 150 x 150 mm mold. The mold containing the

pellets was heated to nearly 180 °C (350 °F), and pressed for 10 minutes at 1500 psi, then cooled at room temperature to about 100 °C (180 °F). The resulting samples were cut using a paper cutter to 10 x 20 mm x composite thickness. Initial moisture content of the wood in the WPC ranged from 1% to 2%.

Solid wood specimens, dimensions 10 x 20 mm, thickness of 0.5, 2.5, 5.0 or 8.0 mm were made from red alder (*Alnus rubra* Bong). This wood species is very susceptible to decay and it was used to confirm that conditions in the incubation chambers were suitable for fungal attack.

All samples (WPC's and solid wood) were then oven-dried at 104 °C for 24 hours, and then weighed to the nearest 0.0001 grams. The specimens were soaked in distilled water for 48 hours to introduce enough water to allow fungal attack (about 30% in WPC's made with maple, nearly 20% in those WPC's made with pine, and over 100% in the solid wood samples), then all moistened specimens were sterilized by heating at 121 °C for 20 minutes in an autoclave.

5.3.2 Test fungi

The brown-rot fungi *Gloeophyllum trabeum* (Pers. ex. Fr.) Murr. (Isolate Madison 617) and *Postia placenta* (Fr.) M. Larsen & Lomb. (Isolate Madison 698), and the white-rot fungus *Trametes versicolor* (L:Fr) Pilat (Isolate R105) were maintained on 1% malt extract agar (MEA) at room temperature (22 to 25 °C) until needed. These fungi were selected because they are aggressive wood decayers, and commonly damage the wood used in structural applications.

5.3.3 Standard procedures and methods

Previous tests showed that 1% MEA was the most effective medium for enhancing the decay of the wood component in a WPC (Silva et al., 2003). Forty ml of sterile 1% MEA were poured into 115 mm diameter petri dishes. Sets of forty petri dishes were center inoculated with placing a 4 mm diameter plug cut from the actively growing edge of cultures of one of the three test fungi. Forty petri dishes were left uninoculated to serve as controls. Eight sets of five petri dishes were prepared for each combination of wood species (maple or red alder), thickness (0.5, 2.5, 5.0 or 8.0 mm) and fungus evaluated (*G. trabeum, P. placenta* or *T. versicolor)* (i.e., one set of five petri dishes, inoculated with *G. trabeum*, was used for testing 0.5 mm thick WPC's made with maple). Eight additional sets of five petri dishes were left uninoculated to serve as controls, one for each combination. The petri dishes were sealed with paraffin film and incubated at 28°C for two to three weeks until the fungal mycelia reached the edges of the petri dish, then one sterile glass rod was placed in the center on the agar surface. Seven sterilized WPC's and four red alder specimens (serving as controls) were placed so that one end of each specimen was on the glass rod and the other end was in direct contact with the agar. Direct contact of all specimens was intended to allow moisture to wick up the ends of these samples to create a moisture gradient. Petri dishes were incubated at 28 °C for 3 to 15 weeks. At 3 weeks intervals, one petri dish containing a given combination of wood species, thickness and fungus was removed. Both the WPC's and red alder specimens were removed from the petri dishes, and adhering mycelium or agar was carefully scraped off. The specimens were weighed to

the nearest 0.0001 g, oven-dried for 24 h at 104 °C, and then weighed again to determine moisture content and weight loss. The combinations of wood species, specimen thickness, exposure period and test fungi used to assess the WPC decay resistance are shown in Figure 5.1.

5.3.4 Parameters used to estimate the extent of decay

The moisture content and weight lost by samples were used to estimate the extent of decay. The moisture content and weight loss of the WPC's were calculated based on the estimated amount of wood in the WPC's. It was assumed that the "moisture content" changes would occur in the wood not in the plastic and that the plastic was not subject to decay (Naghipour, 1996). As a result, all moisture content and weight loss calculations were expressed on a wood basis.

5.3.5 Statistical analyses

Statistical analyses were performed using Statistical Analysis System (SAS Release 8.02, 2002). The procedures Analysis of Variance (ANOVA) and General Linear Models (GLM) were used to analyze the moisture content and weight loss data. Mean values were compared using Duncan's multiple-range test ($\alpha = 0.5$).

Statistical analyses were performed for the moisture content and weight loss obtained at 12 weeks to allow comparison with results obtained in a previous optimization test (Silva et al., 2003).

5.4 Results and discussion

The moisture contents and weight losses of the maple and pine in the polypropylene composites exposed in 1% MEA to test fungi for various exposure

periods are summarized in Tables 5.1 and 5.2 and Figures 5.2 to 5.9. The statistical analysis comparing means for moisture contents and weight losses of both woods at variable composites thicknesses are shown in Table 5.3.

5.4.1 Procedures and methods

A potential moisture problem was detected in the medium in some incubation chambers after 12 weeks of exposure. We noticed that the MEA was partially liquefied in all petri dishes inoculated with *G. trabeum.* Liquification may have disrupted the decay process due to inhibition of the fungal activity because of the presence of excess water.

According to Freitag (personal communication, 2003), *G. trabeum* can degrade the agar due to water production during its metabolism. The absence of substantial weight loss increases between 12 and 15 weeks of exposure may reflect this liquification. Incubation chambers inoculated with either *P. placenta* or *T. versicolor* exhibited only large amounts of condensed water droplets over the mycelia, but no liquification.

5.4.2 Moisture content and weight loss

5.4.2.1 Influence of wood species

Water absorption and weight losses of the WPC differed widely with wood species. Moisture content and weight loss of the maple in the 0.5 mm WPC specimens increased steadily over the 15-week exposure to *G. trabeum* (Figures 5.2 and 5.3), *P. placenta* (Figures 5.4. and 5.5), or *T. versicolor* (Figures 5.6 and 5.7).

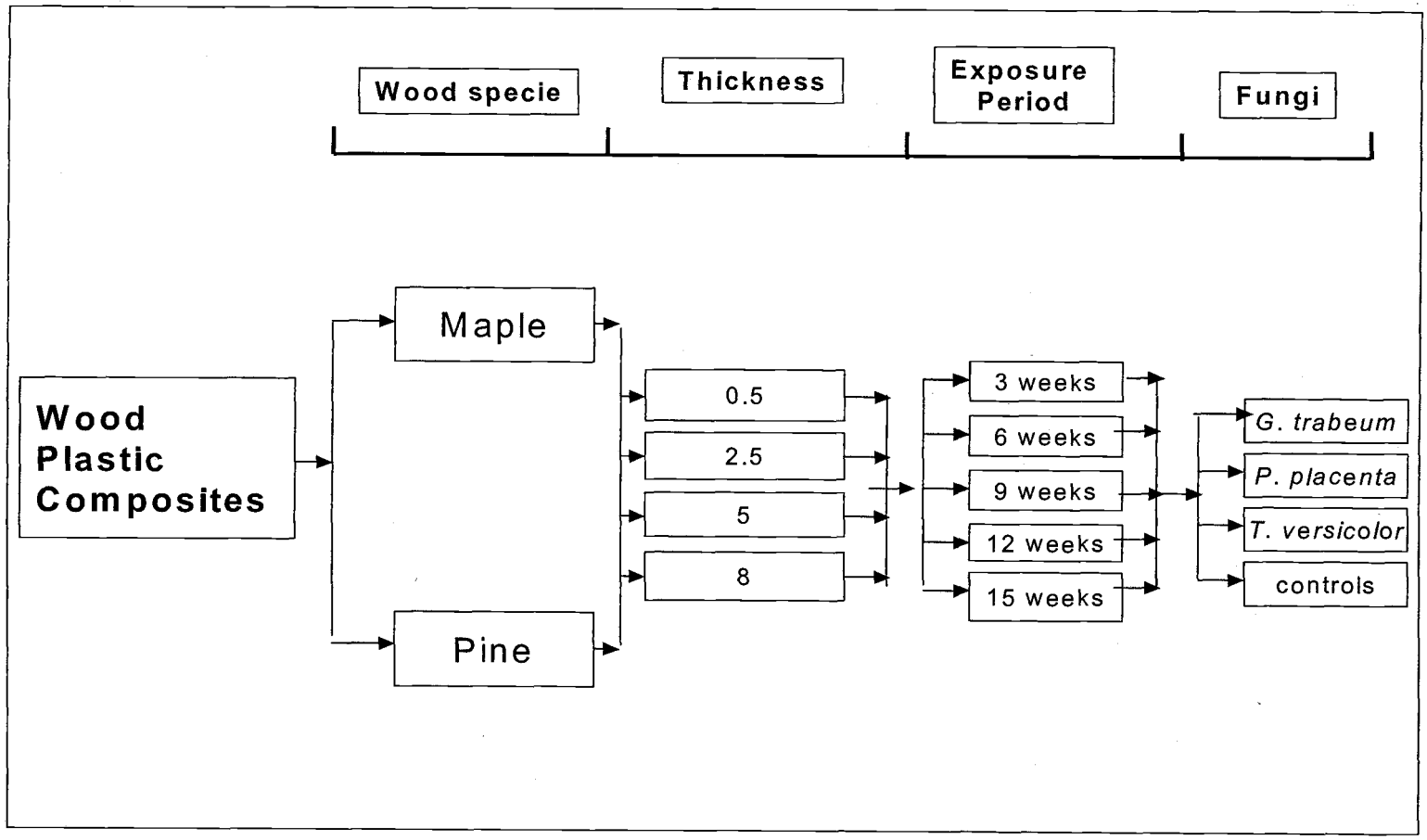

Figure 5. 1 Combinations of wood species, specimen thicknesses, exposure periods and test fungi used for assessing moisture content and weight loss of the wood component in a WPC.

Table 5.1 Moisture contents (MC) and weight losses (WL) of the maple component of a WPC exposed on 1% MEA to *G. trabeum, P. placenta* or *T. versicolor* for 3 to 15 weeks.

Test Fungus	Wood species and thickness (mm)		Exposure Period (weeks)[a]									
			3		6		9		12		15	
			MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)
G. trabeum	Maple	0.5	28.8 (3.9)	9.3 (1.6)	44.3 (6.7)	17.0 (4.1)	48.9 (3.7)	21.5 (2.5)	57.2 (7.6)	27.1 (6.6)	51.4 (7.1)	21.0 (5.2)
		2.5	27.9 (0.8)	2.9 (0.4)	32.8 (1.5)	5.9 (1.1)	32.1 (1.7)	5.2 (1.0)	36.3 (2.8)	8.4 (1.1)	35.1 (1.1)	8.0 (0.7)
		5	29.0 (0.9)	2.1 (0.3)	31.7 (1.4)	3.8 (0.4)	34.4 (1.4)	5.7 (0.3)	32.9 (1.2)	5.4 (0.4)	36.1 (1.2)	6.9 (0.4)
		8	30.5 (2.0)	1.9 (0.2)	32.1 (2.0)	3.8 (0.8)	34.5 (2.2)	5.2 (0.5)	33.8 (0.9)	6.0 (1.0)	36.7 (3.4)	6.4 (0.9)
P. placenta	Maple	0.5	29.8 (3.4)	3.4 (1.1)	35.5 (2.2)	5.8 (2.0)	40.7 (3.7)	11.9 (3.1)	50.3 (6.2)	19.3 (3.8)	56.5 (15.1)	24.0 (14.0)
		2.5	27.2 (1.0)	1.2 (0.3)	29.2 (0.8)	1.8 (0.5)	30.1 (1.1)	3.7 (1.2)	33.1 (2.2)	6.0 (1.9)	38.9 (2.1)	12.7 (2.0)
		5	27.9 (1.1)	1.3 (0.3)	31.1 (1.5)	1.6 (0.3)	31.6 (1.1)	1.9 (0.3)	31.2 (1.1)	2.7 (0.8)	34.8 (1.5)	5.1 (1.0)
		8	29.0 (1.4)	0.9 (0.2)	31.8 (2.8)	1.2 (0.3)	32.5 (2.0)	3.2 (0.7)	32.7 (2.4)	3.8 (0.8)	32.9 (1.9)	3.7 (1.7)
T. versicolor	Maple	0.5	30.7 (1.6)	2.8 (0.8)	38.3 (3.2)	5.4 (2.1)	43.0 (3.0)	11.1 (2.9)	47.5 (9.8)	12.5 (8.2)	54.0 (10.3)	18.5 (7.2)
		2.5	26.7 (0.7)	1.2 (0.3)	29.8 (0.7)	1.5 (0.5)	29.9 (0.9)	2.4 (0.5)	31.6 (1.2)	2.6 (1.4)	35.5 (2.4)	5.5 (2.2)
		5	28.7 (0.8)	1.4 (0.2)	30.7 (0.9)	1.5 (0.2)	32.0 (1.0)	2.9 (0.5)	31.2 (0.7)	1.6 (0.4)	34.4 (1.6)	4.5 (0.8)
		8	29.1 (0.9)	1.2 (0.1)	30.7 (0.8)	1.6 (0.2)	31.6 (0.6)	1.8 (0.3)	32.8 (1.7)	1.3 (0.4)	37.6 (1.5)	4.6 (0.8)
controls	Maple	0.5	32.6 (1.9)	2.8 (1.4)	31.7 (1.5)	0.6 (0.7)	31.2 (1.2)	0.9 (0.5)	33.7 (2.5)	1.4 (0.9)	33.8 (2.6)	2.2 (0.9)
		2.5	28.3 (0.7)	1.3 (0.3)	28.6 (0.9)	1.0 (0.2)	28.9 (0.7)	0.9 (0.2)	28.6 (1.0)	0.6 (0.3)	29.9 (0.9)	1.7 (0.1)
		5	29.6 (1.2)	1.3 (0.1)	29.8 (1.1)	0.9 (0.0)	30.0 (0.8)	1.1 (0.1)	29.6 (0.9)	0.5 (0.1)	31.1 (1.1)	1.9 (0.5)
		8	29.5 (0.8)	0.9 (0.1)	31.0 (2.6)	0.6 (4.9)	30.2 (0.5)	0.9 (0.1)	28.9 (0.7)	0.0 (0.1)	32.1 (1.6)	1.5 (0.1)

[a] MC and WL values estimated based on the wood component of the WPC. Average moisture content and weight loss are the means of 7 specimens per treatment. Values in the parenthesis are standard deviations.

Table 5.2 Moisture contents (MC) and weight losses (WL) of the pine component of a WPC exposed on 1% MEA to *G. trabeum, P. placenta* or *T. versicolor* for 3 to 15 weeks.

Test Fungus	Wood species and thickness (mm)		Exposure Period (weeks)[a]									
			3		6		9		12		15	
			MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)
G. trabeum	Pine	0.5	15.2 (1.4)	1.3 (0.8)	21.0 (0.8)	3.4 (0.6)	23.5 (1.0)	7.4 (0.9)	21.0 (1.4)	3.8 (1.1)	22.2 (0.7)	5.9 (0.7)
		2.5	16.5 (0.8)	1.6 (0.2)	18.4 (1.0)	2.1 (0.4)	18.7 (2.2)	3.3 (0.6)	18.3 (0.9)	2.2 (0.4)	19.7 (1.1)	3.5 (0.6)
		5	16.7 (1.1)	1.4 (0.4)	18.5 (1.2)	2.5 (0.4)	19.1 (1.3)	2.7 (0.6)	18.0 (1.1)	1.8 (0.6)	19.5 (1.8)	2.9 (0.7)
		8	17.1 (1.5)	1.0 (0.2)	19.9 (1.4)	2.0 (0.1)	20.0 (1.6)	2.6 (0.3)	19.2 (1.8)	1.7 (0.5)	20.4 (1.6)	3.0 (0.4)
P. placenta	Pine	0.5	16.8 (1.1)	1.4 (1.2)	19.3 (0.8)	2.5 (1.1)	21.1 (1.0)	4.4 (1.0)	21.0 (2.3)	5.1 (2.3)	24.3 (2.2)	8.8 (3.7)
		2.5	16.7 (2.3)	1.2 (0.3)	17.5 (1.0)	0.8 (0.2)	19.2 (0.7)	2.7 (0.2)	18.1 (1.0)	1.8 (0.3)	20.4 (1.0)	4.8 (0.5)
		5	15.5 (1.4)	0.5 (0.4)	17.7 (0.7)	1.0 (0.2)	19.7 (1.3)	3.1 (0.7)	19.5 (0.4)	3.0 (0.4)	19.0 (1.6)	2.4 (0.7)
		8	15.4 (1.6)	0.4 (0.5)	18.8 (1.4)	1.1 (0.2)	21.1 (4.2)	2.5 (0.4)	19.9 (1.5)	1.7 (0.4)	20.6 (1.7)	2.9 (0.5)
T. versicolor	Pine	0.5	17.9 (1.5)	2.3 (1.5)	19.9 (0.5)	1.4 (0.7)	22.0 (1.1)	2.1 (1.2)	21.1 (2.3)	1.8 (1.0)	21.2 (4.7)	2.3 (0.9)
		2.5	17.1 (0.9)	0.8 (0.2)	17.7 (0.7)	0.6 (0.2)	18.3 (0.6)	1.1 (0.3)	17.2 (0.9)	0.3 (0.2)	19.2 (0.8)	1.4 (0.3)
		5	17.0 (3.0)	0.7 (0.2)	18.3 (0.7)	1.2 (0.2)	18.2 (1.2)	1.2 (0.3)	17.6 (1.0)	0.5 (0.3)	18.5 (0.7)	1.1 (0.1)
		8	17.1 (1.4)	0.5 (0.1)	20.0 (2.0)	0.8 (0.3)	19.0 (1.4)	0.9 (0.1)	18.9 (1.3)	0.2 (0.1)	20.8 (1.3)	1.1 (0.1)
controls	Pine	0.5	18.3 (0.9)	2.2 (0.6)	18.6 (1.4)	0.9 (0.9)	20.1 (0.8)	3.8 (0.7)	16.8 (0.9)	0.2 (0.9)	18.3 (2.5)	1.3 (0.7)
		2.5	17.8 (0.7)	0.9 (0.2)	17.2 (0.8)	0.5 (0.1)	18.2 (0.9)	1.1 (0.1)	16.8 (0.8)	-0.2 (0.1)	16.4 (3.2)	-0.3 (3.2)
		5	17.7 (1.7)	0.6 (0.2)	17.9 (0.6)	0.8 (0.1)	17.9 (0.8)	0.9 (0.2)	17.6 (0.5)	0.2 (0.1)	18.0 (1.0)	0.8 (0.2)
		8	18.1 (3.1)	0.4 (0.2)	20.7 (3.0)	0.6 (0.1)	19.5 (2.0)	0.7 (0.0)	20.1 (3.5)	-0.3 (0.2)	19.0 (1.6)	0.7 (0.1)

[a] MC and WL values estimated based on the wood component of the WPC. Average moisture content and weight loss are the means of 7 specimens per treatment. Values in the parenthesis are standard deviations.

Table 5.3 Effect of wood species and specimen thickness on moisture content and weight loss of maple and pine in wood/plastic specimens following exposure to *G. trabeum, P. placenta* or *T. versicolor* for 12 weeks on 1% MEA [a].

Test Fungus	Wood species	Composite thickness (mm)	Wood moisture content (%)	Wood weight loss (%)
G. trabeum	Maple	0.5	57.2 (7.6) A	27.1 (6.6) A
		2.5	36.3 (2.8) C	8.4 (1.1) DC
		5	32.9 (1.2) DE	5.4 (0.4) FE
		8	33.8 (0.9) DC	6.0 (1.0) DE
	Pine	0.5	21.0 (1.4) G	3.8 (1.1) FHEG
		2.5	18.3 (0.9) HG	2.2 (0.4) JHI
		5	18.0 (1.1) HG	1.8 (0.6) JHI
		8	19.2 (1.8) HG	1.7 (0.5) JHI
P. placenta	Maple	0.5	50.3 (6.2) B	19.3 (3.8) B
		2.5	33.1 (2.2) CDE	6.0 (1.9) DE
		5	31.2 (1.1) DEF	2.7 (0.8) JHIG
		8	32.7 (2.4) DE	3.8 (0.8) FHEG
	Pine	0.5	21.0 (2.3) G	5.1 (2.3) FEG
		2.5	18.1 (1.0) HG	1.8 (0.3) JHI
		5	19.5 (0.4) HG	3.0 (0.4) FHIG
		8	19.9 (1.5) HG	1.7 (0.4) JHI
T. versicolor	Maple	0.5	47.5 (9.8) B	12.5 (8.2) C
		2.5	31.6 (1.2) DEF	2.6 (1.4) JHIG
		5	31.2 (0.7) DEF	1.6 (0.4) JHI
		8	32.8 (1.7) DE	1.3 (0.4) JHI
	Pine	0.5	21.1 (2.3) G	1.8 (1.0) JHI
		2.5	17.2 (0.9) H	0.3 (0.2) JI
		5	17.6 (1.0) HG	0.5 (0.3) JI
		8	18.9 (1.3) HG	0.2 (0.1) JI
control	Maple	0.5	33.7 (2.5) DC	1.4 (0.9) JHI
		2.5	28.6 (1.0) F	0.6 (0.3) JI
		5	29.6 (0.9) EF	0.5 (0.1) JI
		8	28.9 (0.7) F	0.0 (0.1) J
	Pine	0.5	16.8 (0.9) H	0.2 (0.9) JI
		2.5	16.8 (0.8) H	-0.2 (0.1) J
		5	17.6 (0.5) HG	0.2 (0.1) JI
		8	20.1 (3.5) HG	-0.3 (0.2) J

[a] Moisture contents and weight losses represent the mean of 7 specimens per treatment. Means with the same letter are not significantly different using Duncan's multiple-range test ($\alpha = 0.5$).

The 0.5 mm thick sections exceeded the fiber saturation point (FSP) almost immediately for maple samples, but only approached the FSP after 15 weeks for pine. Moisture contents in the thicker WPC's made with maple were below 40% after 15 weeks of exposure.

Weight losses of the maple in the 2.5 mm thick specimens ranged from 5% to 13% after 15 weeks, reflecting the lower moisture levels. Weight losses below 5% were found in the 5 and 8 mm thick maple WPC's after 15 weeks of exposure, regardless of the test fungus.

The moisture content of the pine in the WPC's was nearly 20% regardless of composite thickness, exposure period and test fungus. The FSP of pine is normally between 25% and 30% (Forest Products Laboratory, 1999). Clearly, the plastic altered the moisture sorption capability of the wood. Weight losses of the pine component were below 9% for the 0.5 mm thick WPC's exposed to *G. trabeum* (Figures 5.2 and 5.3) or *P. placenta* (Figures 5.4 and 5.5) and below 5% for the thicker specimens. Moisture contents and weight losses for the WPC specimens made with pine exposed to *T. versicolor* (Figures 5.6 and 5.7) were similar to those found with the controls (<2%) at any given thickness (Figures 5.8 and 5.9). There was convincing statistical evidence (Table 5.3) that at similar composite thicknesses, WPC's made with maple were more susceptible to fungal attack than those made with pine. WPC's made with maple exhibited moisture contents and weight losses that were nearly double those for pine after 15 weeks of exposure period, regardless of the test fungi.

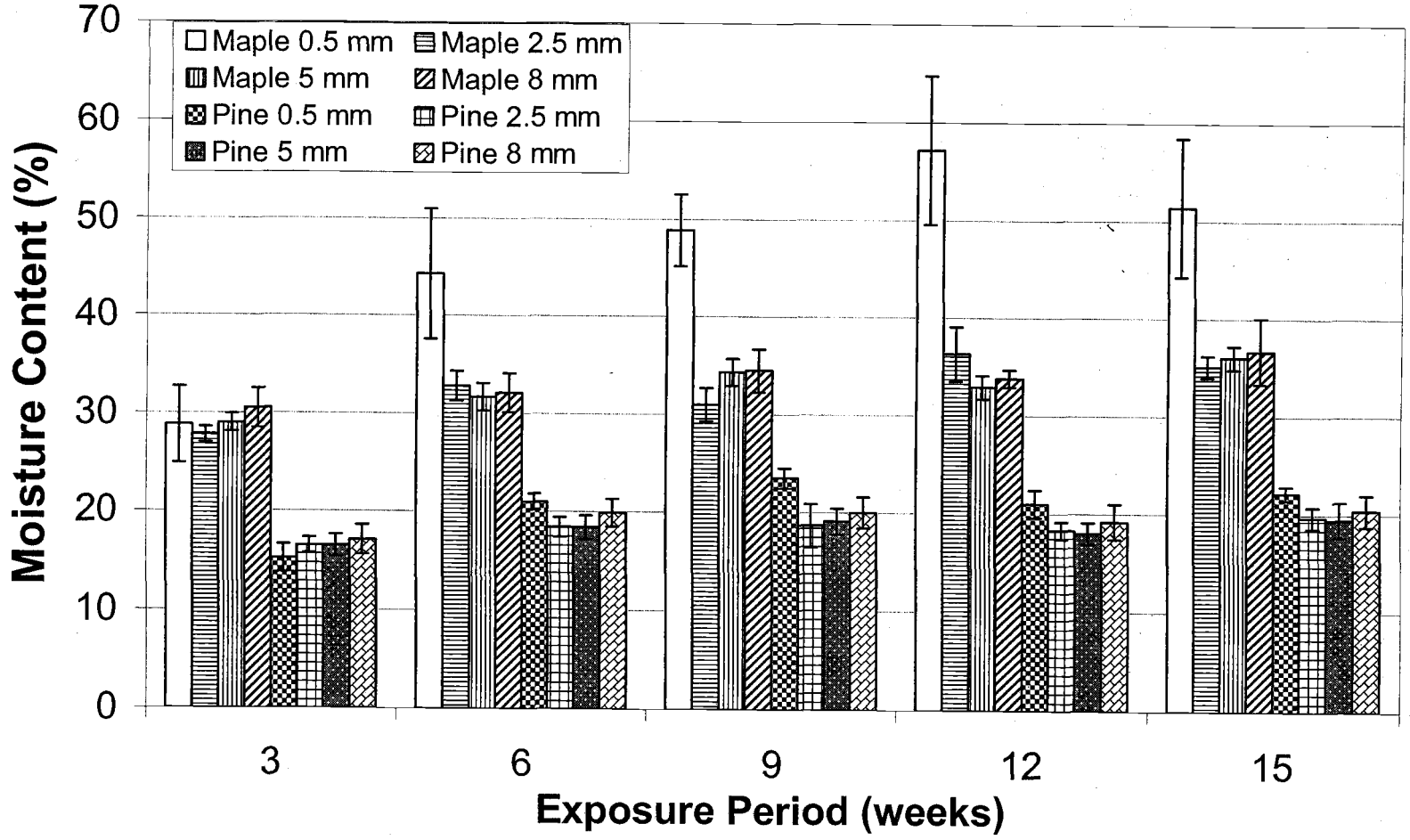

Figure 5.2 Influence of wood species and composite thickness on moisture contents of the wood component of a WPC following exposure to *G. trabeum* for 3 to 15 weeks in agar decay chambers.

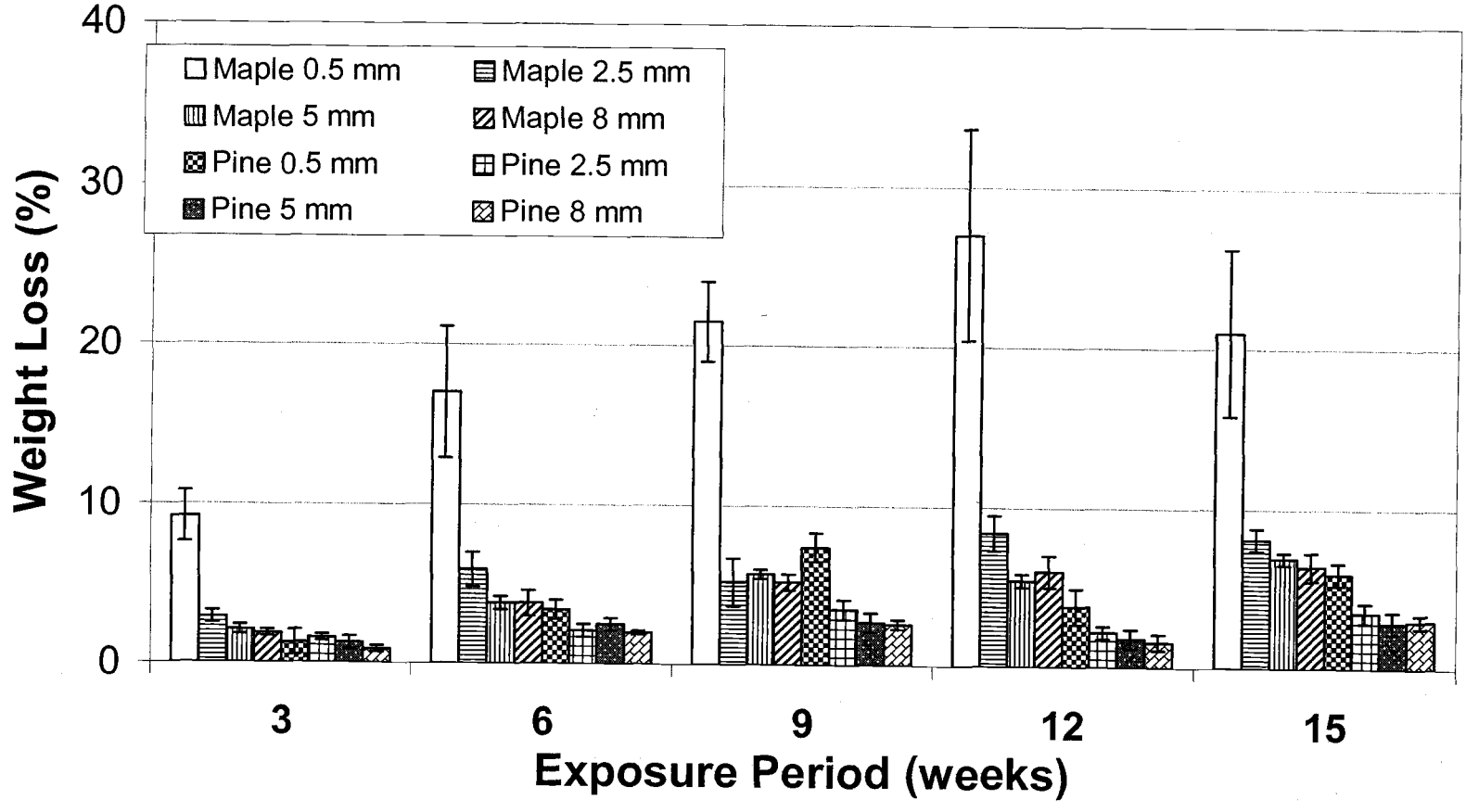

Figure 5.3 Influence of wood species and composite thickness on weight losses of the wood component of a WPC following exposure to *G. trabeum* for 3 to 15 weeks in agar decay chambers.

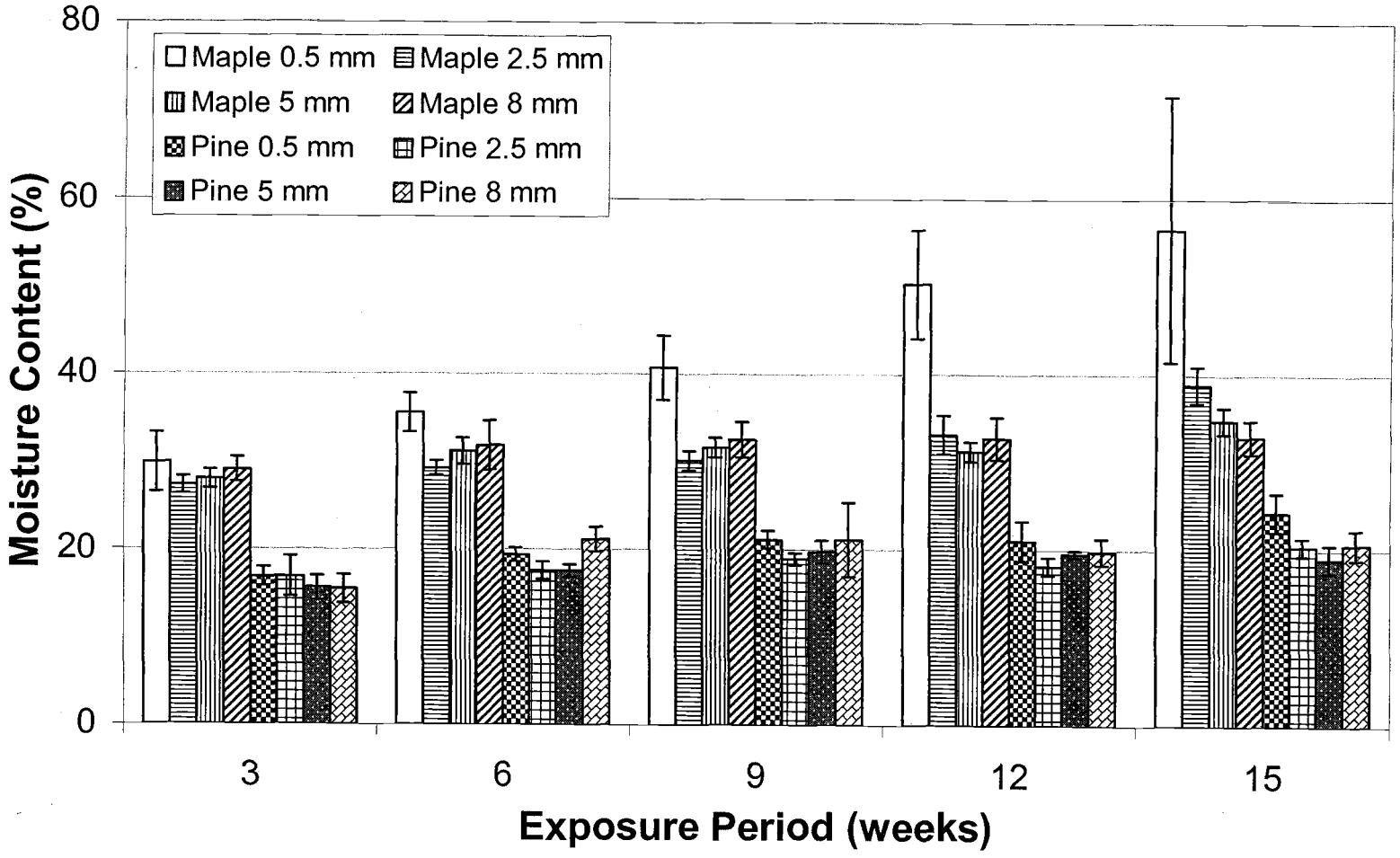

Figure 5.4 Influence of wood species and composite thickness on moisture contents of the wood component of a WPC following exposure to *P. placenta* for 3 to 15 weeks in agar decay chambers.

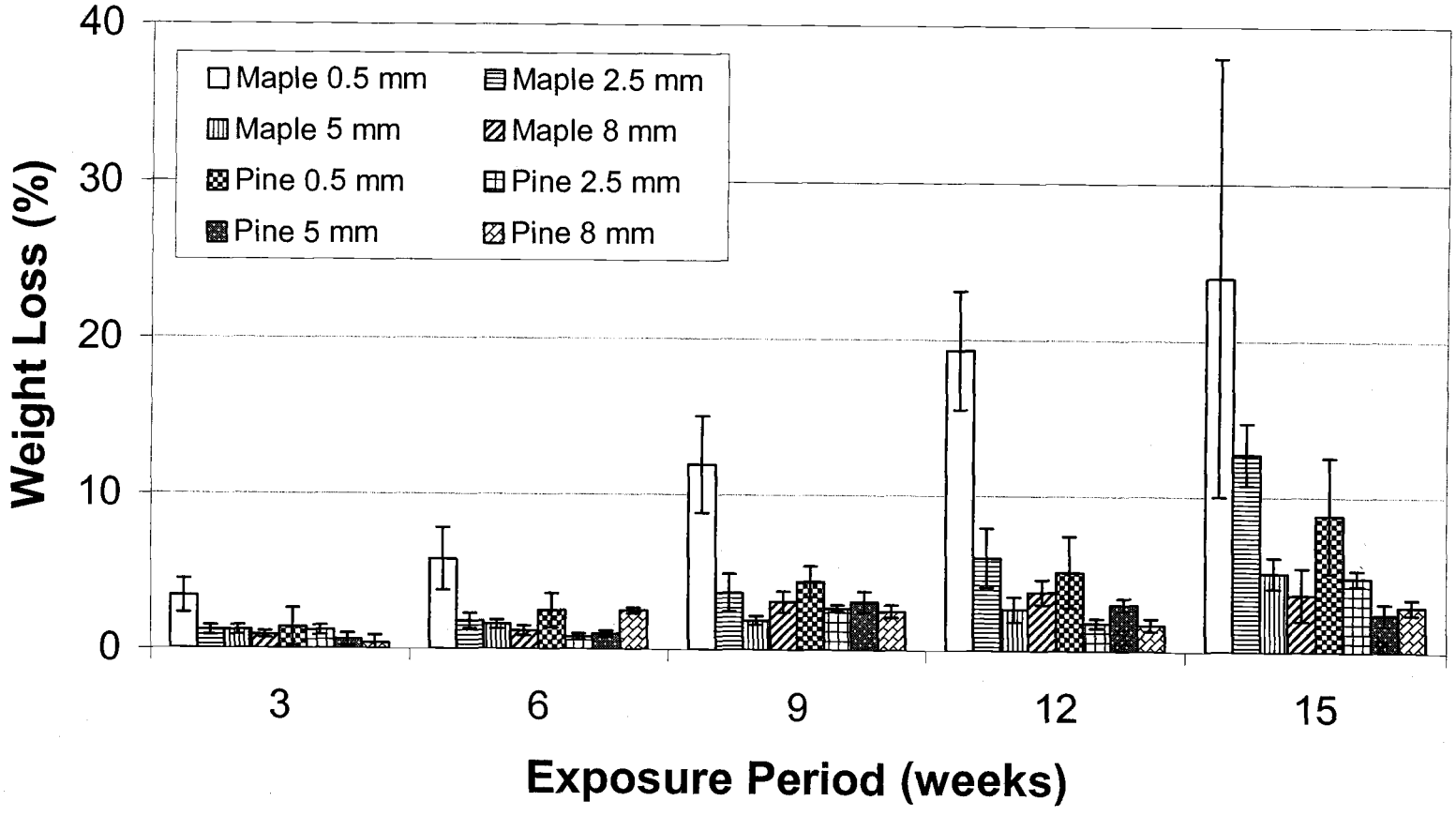

Figure 5.5 Influence of wood species and composite thickness on weight losses of the wood component of a WPC following exposure to *P. placenta* for 3 to 15 weeks in agar decay chambers.

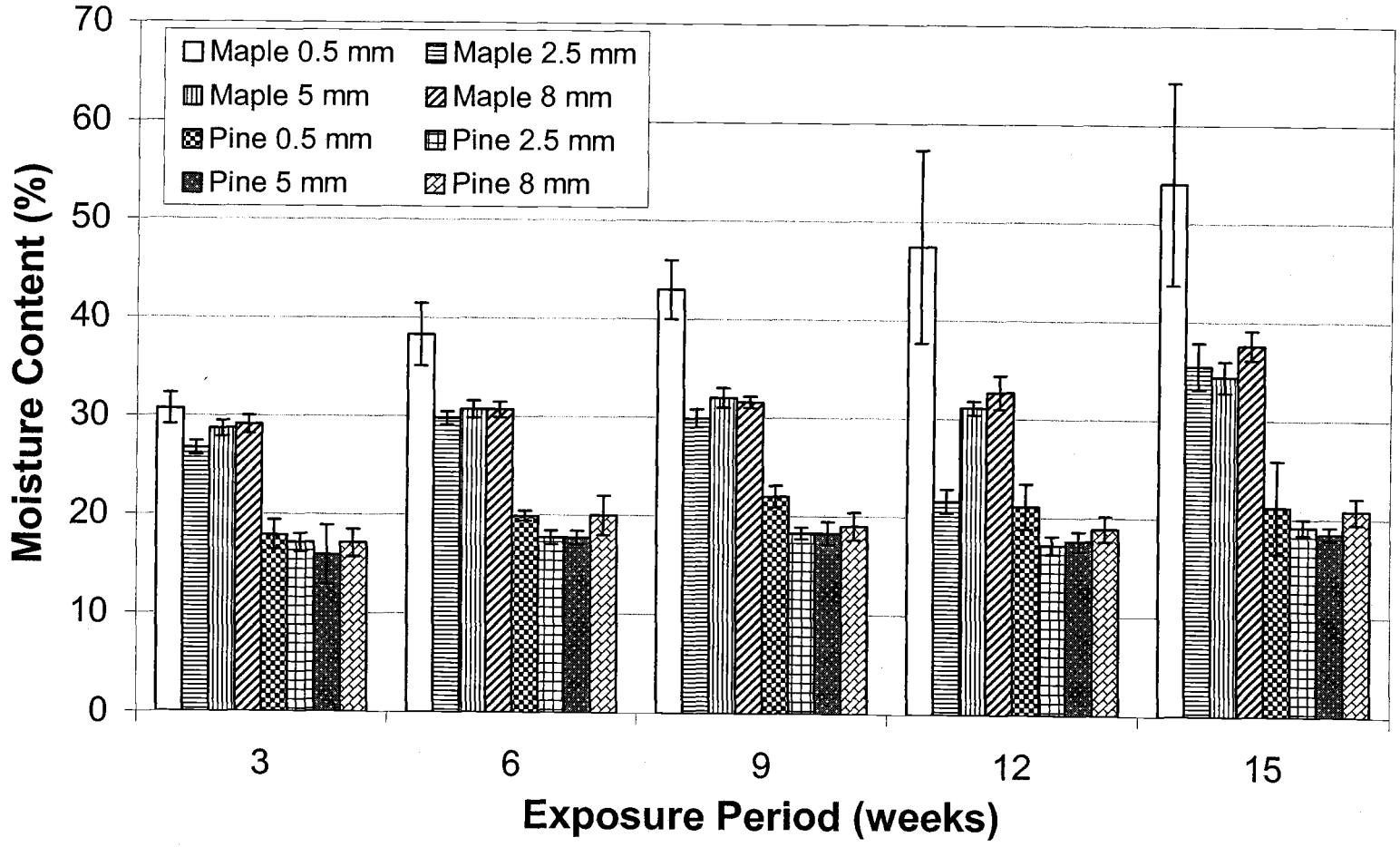

Figure 5.6 Influence of wood species and composite thickness on moisture contents of the wood component of a WPC following exposure to *T. versicolor* for 3 to 15 weeks in agar decay chambers.

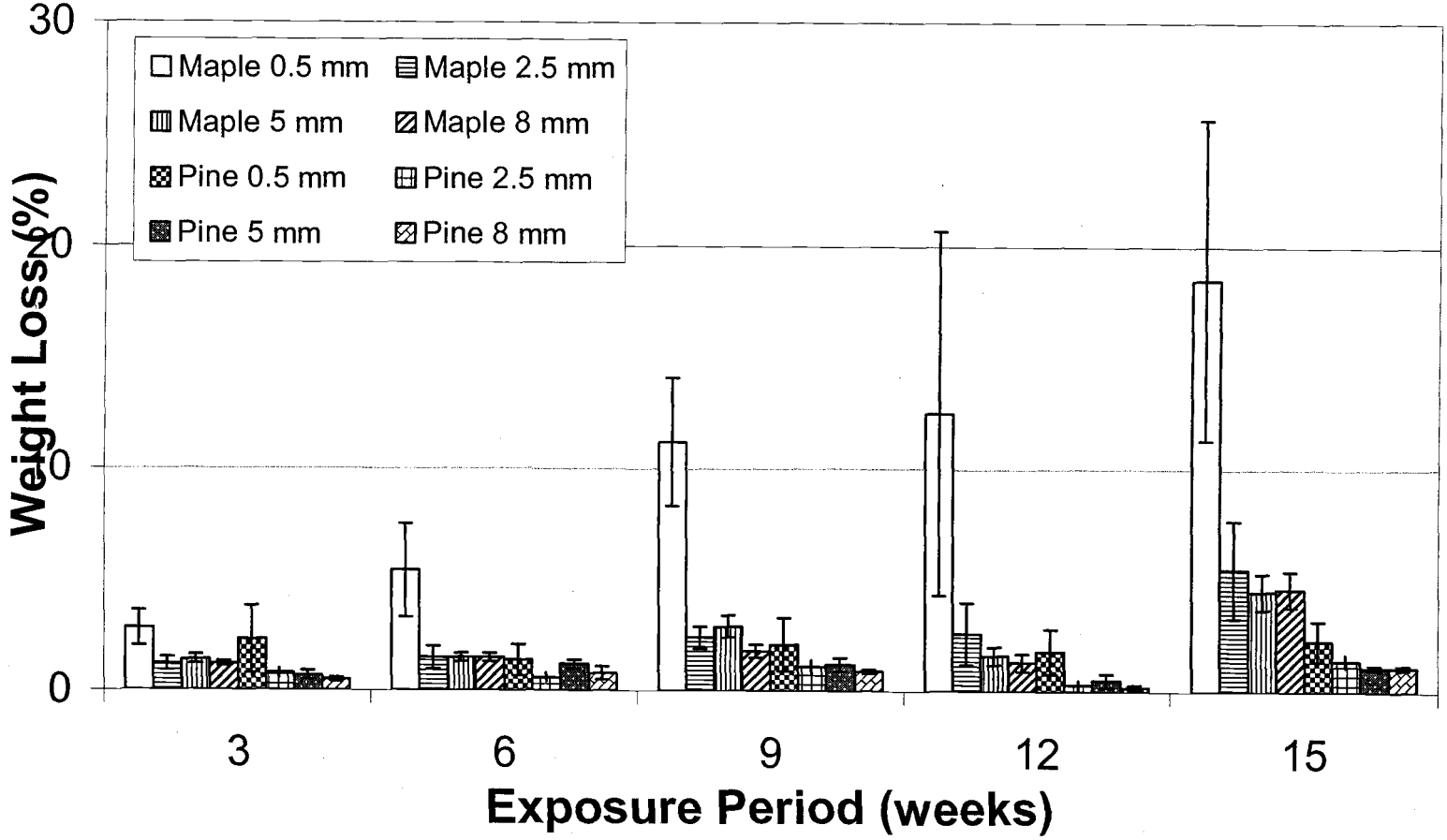

Figure 5.7 Influence of wood species and composite thickness on weight losses of the wood component of a WPC following exposure to *T. versicolor* for 3 to 15 weeks in agar decay chambers.

Moisture contents of the 0.5 mm thick control solid wood samples varied widely, but were over 150% in all specimens exposed to the test fungi, and around 100% for unexposed wood controls. In general, moisture contents for the 2.5, 5 and 8 mm thick solid wood controls were over 150% when exposed to *G. trabeum* or *P. placenta,* but were higher in some totally decayed 8 mm thick solid wood control specimens exposed to *T. versicolor*. In contrast, moisture contents in the solid wood controls were nearly 180%, regardless of thickness (see Appendix B for additional data on moisture content in the wood control specimens exposed with the wood/plastic specimens).

Neither the affinity of water to maple and pine nor their longitudinal air permeability explains the differences in water uptake by the WPC's. The initial moisture content of freshly cut maple is nearly 70% for both sapwood and heartwood, while pine ranges from 33% (heartwood) to 110% (sapwood; Forest Products Laboratory, 1999). These values suggest that pine WPC's should have higher moisture contents and decay rates should be similar for both species, because neither is very decay resistant. However, WPC's made of maple exhibited higher moisture contents and decay rates than those made with pine.

Longitudinal gas permeability of maple and pine are equal (100 cm^3 (gas)/cm atm sec) (Siau, 1984), so differences in moisture uptake observed in our experiment could not be explained on the basis of wood permeability. However, the anatomical features may have influenced rates of moisture content and weight loss. Pine is composed of tracheids while maple contains vessels and fibers. These cells may have

different water sorption properties. In addition, the amount of extractives, nearly 4% in maple (Koelling and Heiligmann, 1996) and about 2% in pine (Panshin and deZeeuw, 1980), may influence longitudinal water permeability and, as a consequence, amount of water uptake.

Another factor that may affect moisture uptake was the high temperature used (about 180 °C) during both pellet compounding and WPC manufacture. This high temperature may affect the hygroscopicity of maple to a greater extent than pine due to thermal degradation of the wood structural components. Reduced moisture uptake by pine may reflect inactivation of the ¯OH groups due to high temperatures during processing that severely degraded the wood hemicellulose. Surface inactivation is known to decrease the ability of wood to absorb water. In our study, it appears that more surface inactivation of the ¯OH groups occurred in pine than maple during pressing.

Another potential factor that may have been affected the rate of water uptake of wood species may be the wood particle size. Particle size could also affect susceptibility to decay since larger wood particles have been associated with higher rates of decay in WPC (Miller, 2001). Verhey et al. (2001) reported nearly 40% weight losses of the wood component in 6 mm thick WPC's made with pine (20 mesh) and polypropylene exposed to *G. trabeum*. In our tests, the particle size of the pine (60 mesh) was larger than maple (80 mesh), so better encapsulation of the individual fibers in the continuous plastic matrix and therefore higher decay resistance would be expected for the maple not the pine.

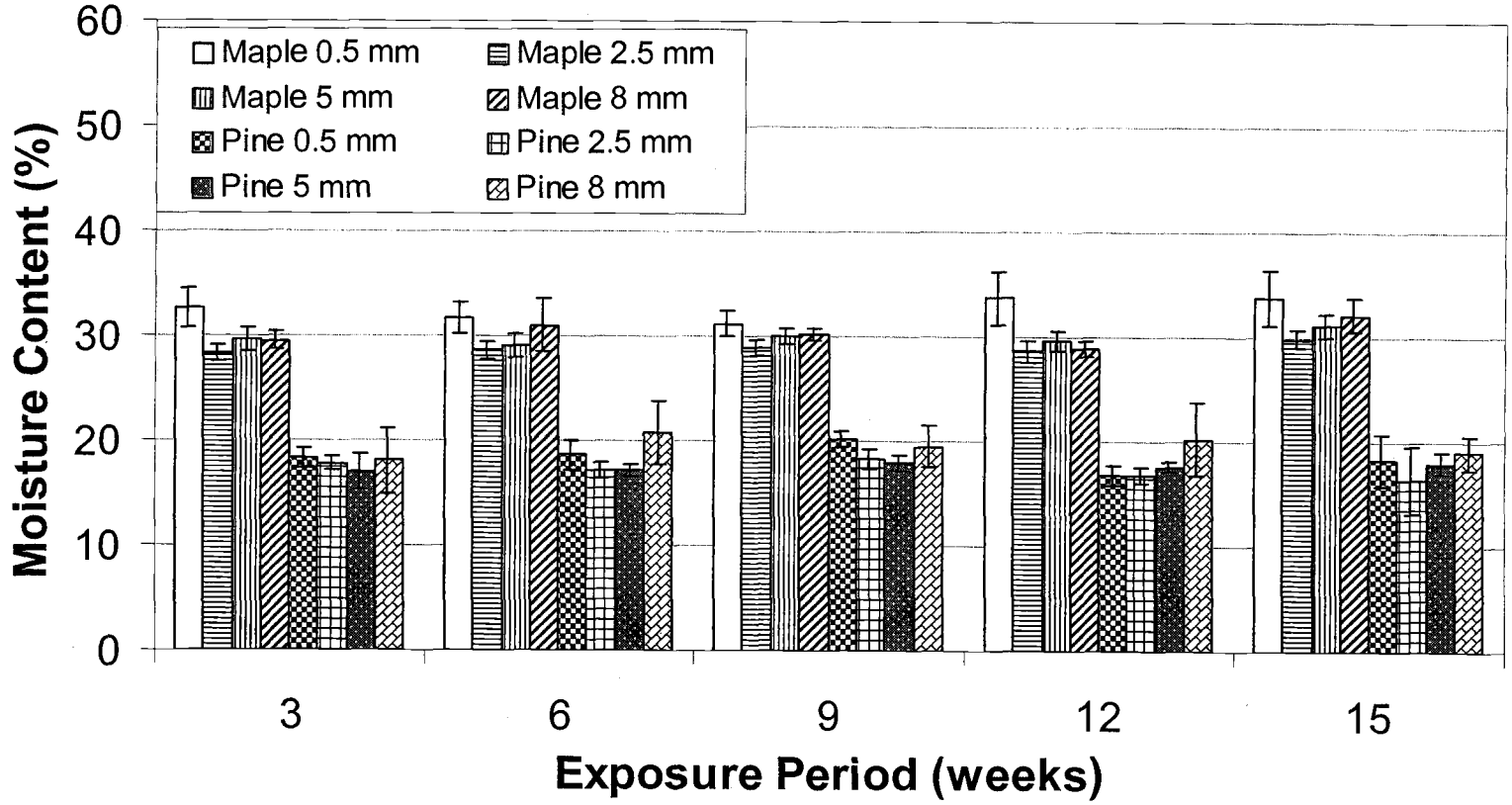

Figure 5.8 Influence of wood species and composite thickness on moisture contents of the wood component of non-fungal exposed WPC controls.

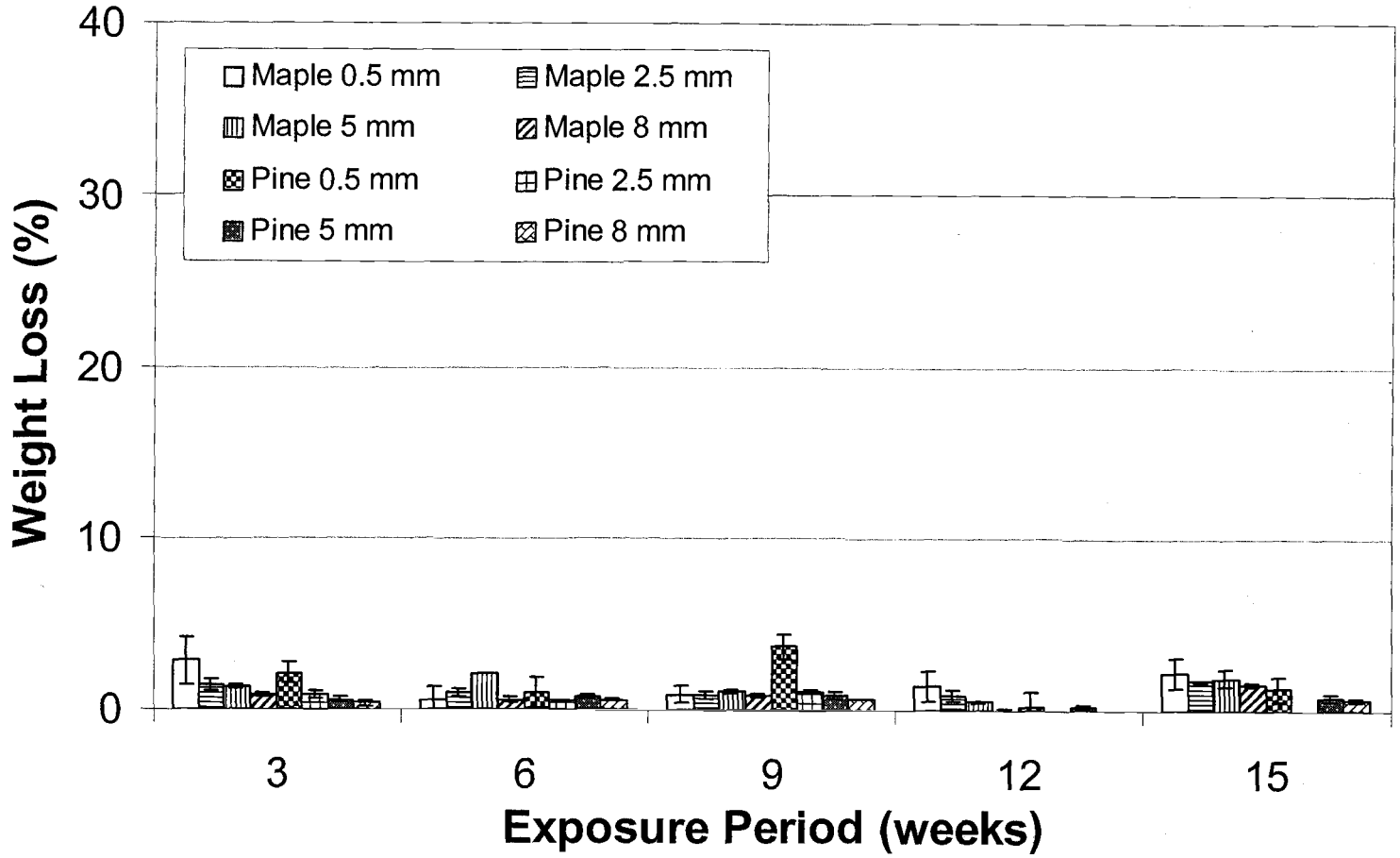

Figure 5.9 Influence of wood species and composite thickness on weight losses of the wood component of non-fungal exposed WPC controls.

Water absorption on the WPC surface is a key parameter for decay since this is the zone where fungal attack is initiated. According to Mankowski and Morrell (2000) and Naghipour (1996), the wood fiber encapsulation by the polymer in a WPC may be incomplete, especially near the surface. As a result, the wood component in these materials reaches moisture levels suitable for fungal attack (Mankowski and Morrell, 2000; Naghipour, 1996). Greater water uptake in the WPC samples containing maple could be attributed to a weak interface between the wood and the polypropylene in the composite. In contrast, it appears that in the pine WPC specimens, the pine/polymer interface was stronger than in those WPC specimens made with maple. Naghipour (1996) showed that WPC's had slower moisture uptake, but were permeable and, as a consequence, subject to fungal decay, particularly at high wood–polymer ratios (> 50% of wood). Surface deterioration and delamination in WPC's was also associated with moisture weathering (Naghipour, 1996). In the current study, microscopic observation of the WPC surfaces after weathering showed a surface deterioration and delamination in WPC's made with maple. This problem was more severe after sterilization because of the effect of the heating. Peyer and Wolcott (2002) reported void formation at the interface of the wood particles and polymer following exposure to water. Void and crack frequencies both increased after water exposure. Separation of wood and plastic and induced cracks in the wood-plastic interface not only reduce the integrity of the composite, but also provide pathways for water penetration and fungal colonization. Previous studies have demonstrated that fungal activity in decayed WPC's was concentrated on the exterior surfaces of the composite, a process that gradually

roughens the composite surface (Mankowski and Morrell, 2000). In the current study, maple particles on the WPC surfaces exposed directly to fungal attack were partially degraded after 15 weeks exposure.

It is interesting to note that higher weight losses found during this research for the 0.5 mm thick maple specimens exposed to *G. trabeum* (nearly 27%) were about half of those found in a previous tests using similar specimens exposed under similar conditions (Silva et al., 2003). Lower weight losses of the maple in the 0.5 mm thick composites exposed to *G. trabeum* suggest that decay process disruption that may have resulted when the MEA liquified. However, it should be noted that 0.5 mm thick WPC's specimens made with maple exposed to either *P. placenta* or *T. versicolor* also exhibited lower weight losses when compared to those obtained in previous studies for similar WPC's (Silva et al., 2003). The incubation chambers inoculated with either *P. placenta* or *T. versicolor* contained large amounts of water condensation over the mycelia after 12 weeks of exposure. This water condensation could eventually influence the decay process. Similar WPC specimens exposed in previous experiments did not exhibit this apparent moisture problem when exposed under similar conditions (Silva et al., 2003).

5.4.2.2 Influence of specimen thickness

Specimen thickness had a strong effect on both water uptake and decay rate of the WPC. Thin WPC specimens reached higher moisture contents and experienced larger weight losses than thicker WPC's regardless of the wood species. Thin specimens made with maple reached higher moisture contents and experienced greater weight

losses than thicker ones. Increasing maple WPC specimen thickness above 0.5 mm was associated with sharp decreases in moisture content and weight loss.

Moisture contents and weight losses of the maple in the 0.5 mm thick WPC specimens exposed to *G. trabeum* were significantly higher than those in specimens exposed to *P. placenta* or *T. versicolor* and controls (Table 5.3). No statistical differences were found in moisture contents and weight losses of the thicker maple WPC's when compared with the controls regardless of the composite thickness tested (Table 5.3). Moisture contents in the 0.5 mm thick WPC's made with pine were significantly higher than those observed in the thicker specimens, but no statistical differences in moisture contents were found among the fungi tested for the 0.5 mm thick specimens (Table 5.3). Thicker WPC samples (>5 mm) did not experience decay. Moisture contents in 5 or 8 mm thick specimens were close to those found with the 2.5 mm thick specimens, regardless of the fungus tested.

Moisture levels in the 2.5, 5 and 8 mm thick WPC specimens made with either maple or pine, and in the 0.5 mm thick pine samples were too low for fungal attack at the end of the test period (i.e <30% in composites made with maple and <20% in those made with pine). Wang and Morrell (2003) found that conditions on the surfaces of commercial WPC's, after 200 days of water immersion were suitable for fungal attack, while moisture levels 5 mm below the surface had changed only slightly. Clearly, test specimen sizes that maximize surface to volume ratios will result in conditions more suitable for decay development. Ohmi et al. (1996) reported that wetting of WPC's

might be influenced by the amount of the material per volume. This would explain the low moisture contents and weight losses found with the 5 and 8 mm thick WPC's.

In general, when the wood in WPC was subjected to fungal attack, the weight losses found in previous studies were relatively low (below 10%) regardless of the composite thickness, wood/plastic ratios, wood species, or test fungus. Our results for the 2.5, 5, and 8 mm thick WPC's made with maple were in the ranges reported by Naghipour (1996), who reported weight loss below 5% for 3 mm thick WPC's made with maple and polypropylene (60:40 wood plastic ratio) exposed to *G. trabeum* in MEA. Clemons (2002) reported that moisture contents reached nearly 25% in 3 mm thick WPC's containing pine (40 mesh) and HDPE (50:50 wood/plastic ratio). These WPC's exhibited weight losses ranging from 6 to 10% when exposed to *G. trabeum*. Pendleton et al. (2002) reported similar results for 10 mm thick specimens made with maple exposed to *G. trabeum, P. placenta* or *T. versicolor*. Simonsen et al. (2002) reported a 6% weight loss in 2 mm thick pine and polypropylene WPC's exposed to *G. trabeum*. Our tests confirm the high resistance of thicker WPC's to fungal attack, especially in those made with pine. This resistance is probably due to slower moisture uptake because of the thermal degradation during processing of the wood fibers.

In contrast, several studies report high weight loss of the wood in a WPC. Verhey et al. (2001) reported a weight loss of nearly 40% of the wood in a 6 mm thick WPC made with pine (20 mesh) and polypropylene (60:40 wood plastic ratio) when exposed to *G. trabeum*. Mankowski and Morrell (2000) reported a weight loss of nearly 20% in the wood of commercial WPC's made with 70% of an unknown mixture of

wood species (0.25 mm diameter) and 30% high-density polyethylene (HDPE) after being exposed to *G. trabeum*, and about 15% when exposed to *P. placenta* in a soil block test. Larger wood particle sizes (20 mesh), polymer type (HDPE versus PP) higher wood/plastic ratio (70:30 versus 60:40), and method (soil block test versus agar method) may help to explain these higher weight losses.

5.4.2.3 Moisture content and weight loss relationship

A direct relationship was observed between moisture content and weight loss in our decay tests. Moisture contents tended to be higher in fungal exposed specimens, and a portion of this increase may reflect metabolic water produced as a result of respiration as the wood decomposed (Ammer, 1964). Fungi are also able to transport water through the hyphae from places of high humidity (the agar) to dry specimens (Muller et al., 2001), increasing the moisture content of the wood in a WPC composite. A thick fungal mat over the WPC surface can also reduce drying, leading to higher moisture levels.

Higher moisture contents were generally associated with higher weight losses (i.e., Figures 5.2 and 5.3). Fungal attack increased the numbers of voids that provide pathways for water movement. Scanning electronic microscopic (SEM) observation of decayed WPC's exposed to *G. trabeum* showed mycelia concentrated on the wood particles exposed on the WPC surface (Silva et al., 2002). Wood particles on the surface were totally degraded after 12 weeks of fungal exposure. This observation supported the premise that wood particles exposed directly to fungal attack were susceptible to decay regardless of any initial plastic covering. Also, once wood is decayed, there is new void volume that can be filled with water.

Silva et al. (2003) reported a strong relationship between moisture content and wood weight loss in 0.5 mm thick WPC's made with maple. In contrast, moisture contents of the wood in specimens that did not experienced substantial decay were very close to those found in the unexposed controls. Silva et al. (2003) reported that moisture contents or weight losses in wood plastic specimens exposed to decay fungi in 1% malt broth for 3 months were similar to those found with the non-fungal exposed controls.

5.5 Conclusions

1) Moisture contents and weight losses of the maple in the WPC's were higher than those found with the pine in the WPC's.
2) Increasing specimen thickness reduced moisture contents and weight losses of the wood in the composite.
3) Moisture contents of the maple in thicker specimens were near the minimum required for fungal attack regardless of test fungus.
4) Moisture contents of the pine in thicker specimens were below the levels required for fungal attack regardless of test fungus.
5) Weight losses of the maple in 5 and 8 mm thick WPC's were similar to the thicker pine specimens.
6) There was a strong positive relationship between moisture content and weight loss.

The results suggested both moisture uptake and weight losses can be accelerated, but further studies are needed to better understand how these results can be translated to tests using thicker specimens. However, further experiments should be

performed to clarify the effect of specimen thickness and volume on wood weight loss in WPC's exposed in 1% MEA. There is no doubt that fungal resistance of WPC's is strongly influenced by the surface to volume ratio, however, it will be important to determine if the results found in our tests using thinner specimens reflect the risk of decay in thicker material.

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Chapter 6

Conclusions

The wood component in a WPC remains susceptible to fungal attack. Although susceptibility was affected by the wood species. Potato dextrose agar (PDA) and malt extract agar (MEA) were the most suitable media for enhancing WPC decay. Fungal attack was concentrated on the composite surface where wood particles exposed directly to fungal attack were totally or severely degraded. This result indicates that test specimens with a high surface to volume ratio will be more susceptible to fungal attack.

6.1 Recommendations for further work

Further work needs to be done to establish whether the extent of decay obtained in these laboratory decay tests reflects the actual risk of fungal attack for WPC's exposed under natural conditions. Further studies are also needed to test the effectiveness of agar media for assessing decay of thicker WPC's. Also, the effect on WPC decay of other type of plastics and wood species should be evaluated. Finally, it would also be interesting to investigate the effect of fungal attack on appearance of WPC surface since appearance is an important attribute for applications such as decking.

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Appendices

Appendix A Agar media preparation

Agar media and concentrations used during the study: malt extract agar (MEA) and potato dextrose agar (PDA), concentrations 0.5, 1.0 or 1.5%.

0.5% media concentration	
Malt extract or potato dextrose	4 g
Agar[a]	8 g
Distillated water	788 g
TOTAL	800 g
1.0% media concentration	
Malt extract or potato dextrose	8 g
Agar[a]	8 g
Distillated water	784 g
TOTAL	800 g
1.5% media concentration	
Malt extract or potato dextrose	12 g
Agar[a]	8 g
Distillated water	780 g
TOTAL	800 g

[a] The agar was kept constant at 1% to avoid problems during media preparation.

Appendix B Preparation of the amended basal salt solution.

Wilson's nutrient solution

Ammonium nitrate	6
Potassium phosphate (dibasic)	5
Potassium phosphate (monobasic)	4
Magnesium sulfate ($7H_2O$)	5
Thiamine HCL	0.001
Glucose	2.5
Agar	15
Distilled water	962.5
	1000 g

Nitrogen sources and concentrations added to a nutrient solution used to obtain the approximate C:N ratios for the amended basal salts media enhanced with nitrogen and glucose. In the current study the nitrogen source was ammonium nitrate.

Desired C:N ratio	Ammonium nitrate
500:1	1.05 g
100:1	5.1 g

The ammended basal salts experiment was based on the procedures described by Morrell, 1981.

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Appendix C Moisture content and weight loss of wood controls exposed on agar to fungal attack

Table A.1 Moisture contents (MC) and weight losses (WL) of the maple wood wafers serving as controls exposed for 12 weeks on MEA or PDA to *G. trabeum, P. placenta* or *T. versicolor,* or left non-exposed.

	Agar media	Concentration	MC (%)	WL (%)
G. trabeum	MEA[a]	0.5	ND [b]	TD [c]
		1.0	ND	TD
		1.5	ND	TD
	PDA	0.5	202	55
		1.0	111	65
		1.5	ND	TD
P. placenta	MEA	0.5	286	56
		1.0	285	55
		1.5	247	50
	PDA	0.5	ND	TD
		1.0	ND	TD
		1.5	300	72
T. versicolor	MEA	0.5	359	62
		1.0	166	48
		1.5	ND	TD
	PDA	0.5	ND	TD
		1.0	ND	TD
		1.5	ND	TD
controls	MEA	0.5	97	3
		1.0	119	3
		1.5	101	1.3
	PDA	0.5	95	1.8
		1.0	98	2
		1.5	100	1.9

[a] Average moisture content and weight loss are the means of 4 specimens per treatment.

[b] TD = Samples totally decayed.

[c] ND = Values not determined due to severe decay.

Table A.2 Moisture contents (MC) and weight losses (WL) of the red alder wood controls exposed jointly with WPC specimens on 1% MEA to *G. trabeum, P. placenta* or *T. versicolor* for 3 to 15 weeks for assessing thickness influence on decay rate.

Test Fungus	Thick -ness (mm)	Exposure Period (weeks)[a]									
		3		6		9		12		15	
		MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)	MC (%)	WL (%)
G. trabeum	0.5	106	11	216	42	200	70	ND[b]	TD[b]	204	40
	2.5	184	5	193	6	196	8	209	14	210	11
	5	187	5	208	8	155	12	214	14	218	14
	8	188	3	200	9	220	11	220	15	212	13
P. placenta	0.5	102	5	130	11	170	27	173	30	161	24
	2.5	180	2	183	3	199	9	20	85	262	24
	5	185	2	196	5	198	5	188	2	190	2
	8	189	2	194	3	198	7	199	8	204	6
T. versicolor	0.5	110	8	181	27	242	45	194	40	356	59
	2.5	185	5	213	13	221	21	230	38	291	48
	5	193	5	210	12	364	37	203	12	598	58
	8	190	3	197	7	369	48	334	37	597	59
controls	0.5	105	5	102	1	96	2	103	2	102	3
	2.5	183	0.3	171	-1	178	-1	168	-2	182	-1
	5	150	-.2	160	-1	164	-1	173	-2	177	-.8
	8	161	-0.1	166	-1.3	166	-1.1	167	-2	166	-0.7

[a] Average moisture content and weight loss are the means of 4 specimens per treatment.
[b] ND = Values not determined due to severe decay.
[c] TD = Samples totally decayed.