AN ABSTRACT OF THE THESIS OF

Shih-Wei Ho for the degree of Master of Science in Chemical Engineering presented on December 14, 1998. Title: Preparation of Interpenetrating Polymer Networks for Improved Cellulose Ester Plastics.

Abstract approved:  

W. E. (Skip) Rochefort

Interpenetrating polymer networks are novel polymeric materials with many advantageous properties, including ease of processing, excellent mechanical strength and good chemical resistance. The formation of an interpenetrating polymer network involves a primary chemical interaction in at least one of the constituent polymers, such as crosslinking, and a secondary chemical bonding between two component polymers, such as hydrogen bonding. In this study a series of semi-interpenetrating polymer networks (semi-IPNs) from cellulose acetate butyrate (CAB) and Novolac resins were prepared and characterized. The goal of this work was to develop an understanding of how the type and frequency of chemical bonds effect the mechanical properties of the cellulose ester polymer blend. The materials were prepared in a Brabender mixer by the addition of Hexamethylenetetramine (Hexa) to CAB/Novolac blends. Hexa acts as a crosslinker for the Novolac resin, thus creating the IPN. Blends composed of CAB (number average molecular weight $M_n = 70,000$), Novolac ($M_n = 900-1,000$; weight average molecular weight $M_w = 5,000-10,000$) and four concentration levels of Hexa were studied. The blends were characterized using chemical analysis, pyrolysis molecular beam mass spectrometry (p-MBMS) and swelling measurement, thermal analysis, differential scanning calorimetry (DSC), rheological analysis, dynamic oscillatory shear, and mechanical analysis, three point bending.

All blends exhibited a single glass transition temperature (Tg) in the DSC measurement and the Tg’s increased with Hexa (crosslinker) content, which suggested that the blends
were fully compatible and that the molecular weight of the polymers increased with the addition of Hexa. From the rheological analysis, the blends containing higher content of Hexa exhibited larger dynamic modulus ($G'$ and $G''$) and higher maximum relaxation time (lower crossover frequency). The blends containing more Novolac showed greater improvement in the rheological properties since they were capable of more effective reactions between Novolac and Hexa. The combined results from DSC and rheology demonstrate that the molecular weight of the blend increase with the increase in Hexa content.

All blends dissolved in methyl ethyl ketone in the swelling measurement, which indicated that none of them were highly crosslinked. However, the solutions of those containing more Hexa were more viscous than those containing no Hexa. The results of p-MBMS analysis demonstrated that the blends consisting of more Hexa showed a significant increase in the number of methylene linkages inside the blend. These results proved that Novolac formed a more branched network with the addition of Hexa but were not able to form sufficient intermolecular methylene bridges to crosslink the system since the reactive Novolac chains were diluted by a large excess of CAB chains, limiting the number of effective reactions between Novolac and Hexa. Mechanical measurements indicated decreased modulus of elasticity and modulus of rupture with the addition of Hexa. This result has not yet been fully explained but is under further investigation.

It was concluded that the blends exhibited higher molecular weight with the increase in the Hexa content by chain extension but were not yet fully crosslinked. The replacement of Novolac with a higher molecular weight phenolic polymer, such as polyvinylphenol will be attempted in an effort to increase crosslinking and achieve the desired increase in mechanical properties.
Preparation of Interpenetrating Polymer Networks
for Improved Cellulose Ester Plastics

by

Shih-Wei Ho

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Master of Science thesis of Shih-Wei Ho presented on December 14, 1998

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Dean of Graduate School

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Shih-Wei Ho, author
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Preparation of Interpenetrating Polymer Networks for Improved Cellulose Ester Plastics

CHAPTER 1
INTRODUCTION

Cellulose esters are high value thermoplastic polymers produced by the reactions between high quality cellulose and organic acids, anhydrides or acid chlorides. Annual domestic production of cellulose esters exceeded billions pounds and more than 150 million pounds are for molded plastic products. These biobased polymers exhibit many advantageous properties including easy processing, outstanding clarity and good mechanical properties. They are widely used in fabric coatings and injection molded products. However, cellulose esters will either deform or break when subjected to a long-term load, which means that these materials are limited in creep resistance and stiffness. One route to improving the strength and creep resistance is through the formation of polymer blends, sometimes with compromises in decreasing mechanical strengths and losing clarity if the blends are not fully compatible.

The mechanical properties of a polymer blend are greatly affected by the extent of mixing and interactions between the two polymers in the blend. Typically two polymers, even very similar ones, do not mix well, which leads to the poor performance in strength, stiffness and impact properties. However, if certain chemical interaction between the functional groups on two polymer chains can be formed, it is possible to obtain a miscible blend with satisfying properties. Recently a series of studies have been done on the polymer blends based on cellulose esters and phenolic polymers. These polymer blends are fully compatible with the help of secondary chemical bonding. The cellulose ester/phenolic blends possess good clarity and excellent properties while the improvement in creep resistance and stiffness is little.
To further improve the creep resistance and stiffness, chemical bonds can be formed within the cellulose ester/phenolic blends. One example is to crosslink the polymers, which will prevent the slippage of polymer chains as they pass over one another and thus will dramatically improve their creep resistance and stiffness. These crosslinked polymer blends, with secondary chemical interaction between the two constituent polymers, are called interpenetrating polymer networks. The resulting cellulose ester/phenolic interpenetrating polymer networks should exhibit better performance in mechanical properties and can be used to form strong molded plastic products. Their applications include molded sheets, molded products such as screw driver handles and toothbrush handles, and coatings of wood furniture and lacquer coatings.

The objectives of this work were to develop enhanced viscoelastic and mechanical properties of cellulose esters by forming interpenetrating polymer networks and to determine the relationship between the chemical compositions and the extent of crosslinking.
CHAPTER 2
BACKGROUND

2.1 Literature Review

2.1.1 Interpenetrating Polymer Networks -- General

An interpenetrating polymer network (IPN) is an intimate combination of two polymers, where at least one of which is synthesized, or crosslinked in the immediate presence of the other\(^1\). Unlike usual polymer blends, by forming networks with chemical bonds or permanent entanglements, IPNs exhibit high interfacial strength between the two polymers and have fewer phase separation problems. It also provides a way of combining two crosslinked thermosetting polymers since the mixing of two polymers occurs or before polymerization. The purpose of forming an IPN is to meet specific needs. The chemical and physical combination of two or more structurally dissimilar polymers provides a feasible way to modify the blend properties. Usually an IPN is easy to process and allows for modifications in mechanical properties, including flexibility, tensile and impact strength, and physical properties, such as chemical resistance, weatherability and flammability resistance.

There are three types of IPNs: simultaneous, sequential and semi. Simultaneous IPNs are synthesized by combining two monomers or linear prepolymers together with their respective crosslinkers and catalysts in melt state or solution. The individual monomers are polymerized by chain or stepwise polymerization. Reactions between the polymers are usually prevented due to different polymerization modes. In sequential IPN, monomer I is combined with the crosslinker to form network I. Network I is then swollen with monomer II and another crosslinker is added to form network II. The resulting mixture contains network I and II. If only one of the two polymers is crosslinked and the other remains linear, a semi-IPN results. Generally, simultaneous IPNs are found to have fewer phase separation problems and improved mechanical and
physical properties. Sequential and semi-IPNs exhibit improved properties, but the majority are phase-separated systems. The potential for improved properties offered by IPNs has led to much recent interest in studying these materials. The studies have focused on two areas: morphology and miscibility studies to determine the characteristics of the IPNs, and measurements of mechanical properties of the resulting IPNs. Several recent examples of each of the IPN types will be discussed.

2.1.2 Examples of simultaneous IPN

Lee and his coworkers 2-6 had examined a series of simultaneous IPNs based on polyurethane and polyester. In a polyurethane (PU)/unsaturated polyester (UPE) system, the morphology and phase mixing were found to strongly depend upon the curing temperature and the reaction sequence of PU and UPE while the mechanical properties showed a strong correlation with the morphology4,5. Improved morphology, impact strength and tensile strength were observed when both the reactions of PU and UPE occurred at the same time5. The driving force for phase separations was found to be the PU reaction4. The study of rheological properties was conducted on IPNs with different concentrations of PU and UPE 6 and the rheological behavior was found to be affected by the amount of polyester in the system. The viscosity and gel formation increased as more UPE was added to in the system.

Simultaneous IPNs prepared from polyphenyleneoxide and polyurethane were studied by Frisch and Zhou 8. Completely miscible, one phase IPNs were made from components whose linear polymers were wholly immiscible. These IPNs had superior properties such as ultimate mechanical strength compared to the pure crosslinked networks or any blend of the corresponding linear polymers.

Fox et al. 32 had studied two types of polyurethane-acrylic copolymer IPNs, both simultaneous and sequential. The IPNs formed simultaneously were found to have larger
crosslink density since they exhibited higher glass transition temperature, better miscibility and greater modulus than the IPNs prepared sequentially.

A series of simultaneous IPNs prepared from PU and epoxy was investigated by Zhang and Hourston. All IPNs showed a single, broad glass transition that shifted to a lower temperature as the content of epoxy increased. No phase separation was found and good compatibility was obtained.

### 2.1.3 Examples of sequential IPNs

Sequential IPNs based on a nitrile-phenolic blend and poly(alkyl methacrylate) was prepared and characterized by Samui et al. The IPNs were not fully compatible but exhibited higher tensile strength compared to corresponding nitrile-phenolic blends. The strength increased with the increase in the concentration of poly(alkyl methacrylate).

Gong and Zhang studied a polyurethane/elaeostearin IPN coatings on regenerated cellulose film. In a PU-rich system, the interpenetration increased with an increase of elaeostearin content. The regenerated cellulose films coated with IPNs showed improved mechanical properties, water resistivity and increased biodegradability.

Rheological behavior of IPNs based on polyurethane and poly(butyl methacrylate) was studied by Lipatov et al. IPNs possessed much greater sets of relaxation times than pure constituent networks based on the dynamic mechanical measurement on the elastic modulus $G'$. The relaxation behavior was found to be dependent on the phase composition and morphological features. The existence of a two-phase structure was also observed.
2.1.4 Examples of semi IPN

Sperling et al.\textsuperscript{12} had investigated semi-IPNs composed of poly(ethylene terephthalate) and caster oil. They found that bond interchange between these two materials played a major role in initial miscibility and morphology. The semi-IPNs displayed much better mechanical properties than the individual component materials did.

A sequence of semi-IPNs based on an addition type thermosetting polyimide and a condensation type thermoplastic polyimide had been investigated by Pater\textsuperscript{14,15}. These high performance networks were synthesized from commercial Thermid-600 and LARC-TPI. Notable improvements were observed in toughness, microcracking resistance and high temperature mechanical performance. These property improvements were achieved without significantly compromising the ease of processing and cost effectiveness.

Kwei and Pearce\textsuperscript{16-18} had conducted studies on semi-IPNs. Networks of phenolic resins and polyamides were prepared and control of miscibility was achieved\textsuperscript{16}. A new route to prepare IPNs was also discovered without the presence of any external crosslinkers or catalysts\textsuperscript{17}. This special semi-IPN was prepared from the interpolymer complexes by condensation of the self-associated dimethylsilanol groups on one of the constituent polymers, for example, poly(styrene-co-4-vinylphenyldimethylsilanol). The system of poly(styrene-co-4-vinylphenyldimethylsilanol) and poly(N-vinylpyrrolidone) was found to attain good miscibility.

Semi-IPNs of poly(vinyl chloride) and polymethacrylates were prepared and characterized by Abu-Isa\textsuperscript{13}. The crosslinker used in this study was peroxide that initiated in situ polymerization of the polyfunctional methacrylate monomers in the PVC matrix. The IPNs exhibited viscous behavior and had diminished elasticity. The morphology was strongly dependent on the curing temperature. PVC and polymethacrylates formed two-phase system when cured at room temperature. When cured at 100 °C, a one-phase system was formed with a single glass transition temperature. The one-phase IPN was stable during cooling and could be stored at room temperature.
2.2 Cellulose Derivatives and Novolac in IPN Applications

2.2.1 IPNs from Cellulose Derivatives

Recently cellulose derivatives have been used for preparation of interpenetrating polymer networks because cellulose exhibits superior mechanical properties and ready availability from natural resources. IPNs containing cellulose derivatives have a distinct potential for yielding light weight and high strength materials. Several examples of IPNs composed of cellulose derivatives were described as followed.

Natchimuthu et al.\textsuperscript{28} has investigated semi-IPNs composed of cellulose nitrate and caster oil based polyurethane. Partial miscibility was observed in the semi-IPNs. Enhanced compatibility was achieved by partially replacing cellulose nitrate with vinyl chloride-vinyl acetate copolymer and forming a ternary IPNs. Improved mechanical properties and flame resistance were noticed in the ternary IPNs.

A series of IPNs prepared from two cellulose derivatives, one of which contained cinnamate and the other containing randomly substituted cinnamate and allyl groups, was studied by Kamath et al\textsuperscript{19}. Both cellulose derivatives used in this study were photocrosslinkable and were used to produce IPNs with common polymers, polystyrene, poly(methyl methacrylate) and poly(vinyl acetate). The IPNs containing cinnamate and allyl groups showed better thermal stability and compatibility than the IPNs of cellulose cinnamate.

The study of the preparation and characterization of high water content semi-IPNs from combinations of cellulose esters (cellulose acetate and cellulose acetate butyrate) and nitrogen-containing vinyl monomers had been conducted by Corkhill and Tighe\textsuperscript{20}. The properties of semi-IPNs were compared with those of copolymers of the same nitrogen containing monomers with methacrylate esters. The IPNs produced from the cellulose esters had higher equilibrium water content and also displayed better performance in stiffness and tensile strength at the expense of elasticity. The surface properties of IPNs,
however, were different from those of copolymers of comparable composition for having appreciably lower polar components of surface energy.

### 2.2.2 IPNs from Novolac type Phenolic Resins

Novolac prepolymer are branched, low molecular weight materials and thus are too brittle to be useful. When crosslinked Novolacs can be used in industry for their good heat resistance, electrical insulation, thermal stability, flame resistance and chemical resistance\(^{21}\). Forming IPNs based on Novolac was a simple, economic and effective way to modify and extend the properties of this phenolic resin. Several Novolac-based IPNs are described here.

The study on cured Novolac/poly(methyl methacrylate) blends was conducted by Zhang and Solomon\(^{27}\). The phase structure of crosslinked Novolac blends showed composition dependence. Novolac-rich blends resulted in semi-interpenetrating polymer networks where Novolac formed a highly crosslinked network through the whole blend while PMMA distributed uniformly. PMMA-rich systems showed partially miscible or phase-separated. Glass transition disappeared in the Novolac-rich system since segmental motion of chains was frozen by crosslinking.

Ma and his co-workers\(^{24-26}\) studied the thermodynamic properties of IPNs containing Novolac. The IPNs were prepared from Novolac and several commercial polymers such as poly(adipic ester) and poly(hydroxyl ether of bisphenol A). All systems exhibited complete miscibility within moderate temperature range. The intermolecular hydrogen bonding presented within the phenolic resin led to the improvement in miscibility. The thermodynamic properties predicted from Painter-Coleman association model were consistent with the experimental results.

Zhong and Guo\(^{22}\) have conducted a series of experiments on semi-IPNs based on Novolac and poly(ethylene oxide). Novolac was cured with hexamethylenetetramine at
100 °C, 160 °C and 190 °C. Hydrogen bonding interaction between the hydroxyl groups on Novolac and the ether oxygens of poly(ethylene oxide) were found in the blends. All IPNs displayed satisfying miscibility over the entire composition range while Tg disappeared in the Novolac-rich blends. Another series of Novolac/poly(e-caprolactone) blends was also investigated by Zhong and Guo. The miscibility and morphology of the blends were significantly altered by curing. Partial miscibility was observed in the Novolac-rich system while phase separation was found in blends with lower Novolac contents. Crosslinking the Novolac again resulted in the disappearance of the glass transition temperature, which is attributed to less mobility of the Novolac chain segments following crosslinking.

2.3 Chemical Background

In a semi-interpenetrating polymer network system, two linear polymers are mixed and then the crosslinker and catalyst are added so that one of the components is crosslinked. The polymer blend first formed by the two uncrosslinked polymers has to be a miscible mixture to assure a certain degree of mechanical strength. A number of studies have shown that some specific interactions between two polymers, such as hydrogen bonding, acid-base interactions and bipolar interactions, can lead to the production of miscible blends. The two polymers used in this study are cellulose acetate butyrate (CAB) and Novolac. Figure 2-1 is a schematic showing the interaction between these two chemicals. CAB has acetyl and butyryl ester groups and hydroxyls all attached to the cellulose backbone. The acetyl and butyryl ester groups are strong electron acceptors. Novolac has hydroxyl groups which are electron donors and can readily form bonds with ester, amide or ether groups. Therefore CAB can form hydrogen bonds with the phenolic hydroxyl groups on Novolac, producing a miscible blend.

After forming hydrogen bonding with CAB, Novolac can be reacted with hexamethylenetetramine (Hexa), the crosslinker, to form a highly branched or crosslinked network. Figure 2-2 displays the reaction mechanism between Novolac and
hexamethylenetetramine. Since Novolac is a low molecular weight polymer, the crosslinker first extends the chains on Novolac leading to increased branching. As Novolac continues to chain extended, it will start to crosslink, and if there is sufficient Novolac and Hexa in the system, a fully crosslinked polymer network can be formed. When Novolac and Hexa react, there is a large amount of ammonia evolved (see Figure 2-2). The evolved ammonia acts as the catalyst for reaction.

Figure 2-1 A schematic of the hydrogen bonding between CAB and Novolac
Figure 2-2 A schematic of the reaction mechanism between hexamethylenetetramine (Hexa) and Novolac
2.4 Experimental Background

2.4.1 Swelling Measurement

The swelling measurement is widely used technique to determine the degree of crosslinking. When a crosslinked polymer is placed in a suitable solvent, the polymer imbibes the solvent and expands to a swollen gel. Unlike linear or branched polymers, the swollen gel formed by the crosslinked polymer does not break down, i.e., a crosslinked polymer never dissolves in the solvent unless the crosslinks are broken. The amount of swelling depends on the density of crosslinking and the strength of the interaction between polymer and solvent.

2.4.2 Thermal Analysis: Differential Scanning Calorimetry

2.4.2.1 Morphological Changes in Polymers

Polymers show a characteristic sequence of changes as they are heated. Linear polymers are glassy at low temperatures. As the temperature is raised, small scale molecular motions increase until they undergo cooperative motion known as the glass transition. The temperature where the glass transition occurs is called glass transition temperature ($T_g$). When heated above $T_g$, amorphous polymers will pass through a rubbery state and eventually enter the liquid state. Crystalline polymers remain in the rubbery state above $T_g$ until the temperature is further raised to the melting temperature ($T_m$). At this point, the crystalline regions within the polymer melt to a viscous fluid at a sharply defined temperature.

$T_g$ and $T_m$ are two important properties of polymers. The detection of $T_g$ and $T_m$ of a polymer enables the identification of the polymer and determination of appropriate processing conditions. Moreover, the detection of $T_g$ of a blend composed of two constituent polymers can provide information on the miscibility of the two polymers. A
miscible blend shows a single Tg in between those of the two polymers while a
immiscible blend shows two distinct glass transitions, each corresponding to the
constituent polymer.

2.4.2.2 Differential Scanning Calorimetry (DSC)

DSC is one of the most popular techniques for thermal analysis. The word differential
emphasizes that the measurement involves both the measured sample itself and a blank
reference material. DSC is also regarded as a quantitative technique in thermal analysis.
The area enclosed by the thermo-curve recorded against time is directly proportional to
the energy change.

A cell containing a small amount of sample and a reference cell are mounted in a block
with a heater for each cell and thermocouples to monitor the temperatures of both cells.
The thermodynamic property monitored is enthalpy. A control system adjusts and
measures the power to the heaters to maintain the same temperature for the sample and
the reference temperatures as the sample is heated at a pre-programmed rate. At Tg, the
heat capacity of the sample suddenly increases, requiring more power relative to the
reference in order to maintain the same temperatures. This differential heat flow to the
sample causes a drop in the DSC curve. At Tm, the sample crystals melt at a constant
temperature, requiring a sudden input of large amounts of heat to keep the sample at the
same temperature with the reference. This big input of heat results in an endothermic
melting peak.

2.4.3 Rheological Analysis: Dynamic Oscillatory Shear

Rheology is the science of the deformation and flow of materials. A body is said to be
deformed when the application of an appropriate force system alters the shape or size of
the body. A body is said to flow if its degree of deformation changes continuously with time. Rheology predicts the force necessary to cause a given deformation or flow of the body, or conversely, to predict the deformation or flow resulting from the given force. If the body is a viscous fluid, the application of forces will result in flow but the fluid will not return to its original state after releasing the forces. If the body is an elastic solid, it will deform but not flow under the given forces and will return to its undeformed state after releasing the forces. Polymers are viscoelastic materials, exhibiting both “fluid-like” and “solid-like” behaviors. Since the classical theory of mechanics (Hooke's Law) only handles properties of elastic solids while the classical theory of hydrodynamics (Newton’s Law) merely deals with properties of viscous liquids, the viscoelastic behavior of a polymer, which does not obey either Hooke’s law of elasticity or Newton’s law of viscosity, is studied through rheology.

Understanding the rheological properties of a material is a prerequisite to its processing and eventual application in a product. There are several methods to measure the rheological properties of the wide variety of polymer materials ranging from polymer solids, melts and solutions. Dynamic oscillatory shear is most often used to study the viscoelastic behavior of a polymer melts or solutions. In this technique, elastic (storage) modulus ($G'$) and viscous (loss) modulus ($G''$) are measured as the polymer undergoes an oscillatory shear at various frequencies and strains. The dynamic modulus ($G', G''$) can be used to calculate the complex viscosity. Rheological data over a wide frequency range can be obtained by performing experiments at various temperatures and then employing time-temperature superposition to obtain “master curves” at some reference temperature. The master curves can be used to compare the rheological behavior of the prepared semi-IPNs. They provide useful information such as maximum relaxation time, molecular weight and extent of crosslinking, which can be used to characterize the polymers.
2.4.3.1 Dynamic Oscillatory Shear

In a dynamic oscillatory shear experiment, the thin polymer sample is placed between two parallel plates (parallel plate geometry). The sample is subjected to a harmonic shear stress with controlled amplitude \( \sigma_0 \) and angular frequency \( \omega \) described by

\[
\sigma = \sigma_0 \cos \omega t
\]

Eq. 2-1

If the viscoelastic behavior is linear, the strain will be out of phase with the stress and is given by

\[
\gamma = \gamma_0 \sin \omega t
\]

Eq. 2-2

The shear rate \( \dot{\gamma} \) is derived by differentiating Eq.2-2.

\[
\dot{\gamma} = \omega \gamma_0 \cos \omega t
\]

Eq. 2-3

To express the relationship between the shear stress and the shear rate, a constitutive equation for linear viscoelasticity has to be used.

\[
\sigma(t) = \int_{-\infty}^{t} G(t-t') \dot{\gamma}(t') dt'
\]

Eq. 2-4

where \( G(t) \) is called the relaxation modulus.

By Inserting Eq. 2-3 into Eq. 2-4 and replacing \( (t-t') \) with \( (s) \), Eq. 2-4 becomes

\[
\sigma(t) = \int_{0}^{\infty} G(s) \omega \gamma_0 \cos(\omega(t-s)) ds
\]

Eq. 2-5

\[
= \gamma_0 \left[ \frac{\omega}{2} \int_{0}^{\infty} G(s) \sin(\omega s) ds \right] \sin(\omega t) + \gamma_0 \left[ \frac{\omega}{2} \int_{0}^{\infty} G(s) \cos(\omega s) ds \right] \cos(\omega t)
\]

Defining two frequency-dependant functions – the storage modulus \( G'(\omega) \) and the loss modulus \( G''(\omega) \), Eq. 2-5 can be simplified into
\[ \sigma(t) = \gamma_0 \left( G' \sin \omega t + G'' \cos \omega t \right) \quad \text{Eq. 2-6} \]

where

\[ G' = \omega \int_0^\infty G(s) \sin(\omega s) \, ds \]

\[ G'' = \omega \int_0^\infty G(s) \cos(\omega s) \, ds \]

Defining \( \delta(\omega) \) to be the phase angle between the stress and strain, apply trigonometric relations into Eq. 2-1:

\[ \sigma = \sigma_0 \cos \omega t = \sigma_0 \sin(\omega t + \delta) = \sigma_0 \cos \delta \sin \omega t + \sigma_0 \sin \delta \cos \omega t \]

Comparison of the expanding Eq. 2-1 and Eq. 2-6 shows that

\[ G' = \left( \frac{\sigma_0}{\gamma_0} \right) \cos \delta \quad \text{Eq. 2-7} \]

\[ G'' = \left( \frac{\sigma_0}{\gamma_0} \right) \sin \delta \]

### 2.4.3.2 Time-Temperature Superposition

Rheological properties are usually highly temperature dependent. This means that to obtain a complete picture of the material behavior, experiments must be carried out over a wide temperature range. However, this is always restricted by instrumental limits. Time-temperature superposition is a method to bring together moduli and viscosity measured at various temperatures on a single master plot. This technique can greatly simplify the experiments and make it possible to display the rheological properties on a single curve covering a much broader range of frequency than ever can be measured at a single temperature.
The basic concept of time-temperature superposition is that there is a relationship between the relaxation time and both time and temperature. In the linear viscoelastic region, the relaxation time distribution of the polymer melt remains unchanged over frequencies and temperatures. So the $G'$ and $G''$ curves can be shifted along the time domain (frequency axis) without affecting the shapes of the curves. The shift of curves is based on the following equation.

$$E(T, \omega) = E(T_0, \omega_{T_0} a_T)$$  \hspace{1cm} Eq. 2-9

where $E$ is the modulus, $T_0$ is the reference temperature, $\omega$ is the frequency and $a_T = \omega_{T_0}/\omega$ is the shifting factor. The superposition of data at various temperatures is a very useful technique of extending the curves of $G'$ and $G''$ to many decades of time or frequency, which is well beyond the range of frequencies accessible using standard lab rheometers.

### 2.4.3.3 Interpretation of rheological data

The effect of molecular weight of a polymer can be observed from several places. One of the most important and the simplest indications is the maximum relaxation time $\tau_R$. From the reptation theory first discussed by Edwards and de Gennes and then developed in detail by Doi and Edwards, the relationship between the maximum relaxation time and the molecular weight is given by

$$\tau_R \propto M^3$$  \hspace{1cm} Eq. 2-10

The polymer exhibiting a higher maximum relaxation time has a larger molecular weight. The value of the maximum relaxation time can be approximately determined from the $G'$ and $G''$ vs. frequency diagram. The $G'$ and $G''$ curves will cross at certain frequency called the crossover frequency $\omega_c$. The relationship between the maximum relaxation time and crossover frequency is displayed as follows.

$$\tau_R \propto \frac{1}{\omega_c}$$  \hspace{1cm} Eq. 2-11
Thus, the polymer with a lower crossover frequency has a higher maximum relaxation time and higher molecular weight.

Figure 2-3 displays the modulus of a crosslinked polymer. At short times, the polymer is in the glassy state and the value of the modulus starts to drop off. The modulus will then approach an equilibrium value at long times for the crosslinked network. Figure 2-4 shows the modulus of two uncrosslinked polymers. Polymer A has a molecular weight below that at which there is a significant level of entanglement and polymer B has a molecular weight which is sufficiently high to attain a degree of entanglement. Both curves behave similarly to that of the crosslinked polymer at short times. At longer times, both curves decline instead of approaching a constant. A plateau zone can be roughly observed from curve B.

The shape of the curve of the modulus is an important indication of crosslinking. The modulus of a crosslinked polymer will reach an equilibrium value at long times (in low frequency regime) while the modulus of an entangled, uncrosslinked polymer will show a plateau region and then move downward to a terminal zone. If there is no significant change in the chemical nature of the polymer, the effect of crosslinking shows only a slight difference from the uncrosslinked polymer at short times.
Figure 2-3 Relaxation modulus of a crosslinked polymer

Figure 2-4 Relaxation modulus of uncrosslinked polymers
2.4.4 Mechanical Analysis: Modulus of Elasticity and Modulus of Rupture

Both the modulus of elasticity and the modulus of rupture can be observed in the stress-strain behavior of a polymeric material. Modulus of elasticity is the ratio of stress to strain when deformation is totally elastic; it is also a measurement of the stiffness of a material. Modulus of rupture is the stress at rupture from a bend test, from which the brittleness of a material can be compared.

When a test specimen is under an axial load, the relationship between the load and the displacement can be plotted as a stress-strain diagram. Figure 2-5 and Figure 2-6 display two types of stress-strain diagrams that are usually seen among polymeric materials. The maximum stress on the curve is the modulus of rupture and the slope of the linear region is the modulus of elasticity.

Figure 2-5 Sample remains unbroken after reaching the maximum load.
In this study, a three-point bending test is performed to obtain the modulus of elasticity and the modulus of rupture. Figure 2-7 is a simple schematic of the three point bending test. A rectangular bar was used as the test specimen. L is the length between two support points, d is the thickness of the bar and b is the width, F is the axial load applied on the specimen and \( \Delta d \) is the displacement caused by the axial load.

![Diagram of three point bending test](image)

Figure 2-7 A schematic of the three point bending test
Modulus of elasticity (MOE) and modulus of rupture (MOR) can be calculated from the following equations where $F_{\text{Max}}$ is the maximum load where the bar breaks or the stress-strain curve starts to drop off.

$$MOE = \frac{\Delta F L^3}{4 b d^3 \Delta d}$$

$$MOR = \frac{3 F_{\text{Max}} L}{2 b d^2}$$

Eq. 2-14

2.5 References:

1. Kirk-Othmer Encyclopedia of Chemical Technology
A series of semi-interpenetrating polymer networks were prepared from cellulose acetate butyrate (CAB) and Novolac resin. Cellulose acetate butyrate is a linear polymer. Novolac is a phenolic polymer that can be crosslinked with the addition of Hexamethylenetetramine (Hexa). The polymers were melt blended, ground into powders with beach sand size grains and molded at 190 °C into desired shapes for characterizations. Molecular beam mass spectrometry, swelling measurement, differential scanning calorimetry, dynamic oscillatory shear and three-point bending tests were performed on the CAB/Novolac blends. The details of the sample preparations and characterizations are described in the following sections.

3.1 Materials

3.1.1 Sample Matrix

Table 3.1 showed the composition of the blends of CAB, Novolac and Hexa. Two series of blends were investigated, 90 Wt% CAB/10 Wt% Novolac (90/10) and 80 Wt% CAB/20 Wt% Novolac (80/20). The blends with 70 Wt% CAB/30 Wt% Novolac and 50 Wt% CAB/50 Wt% Novolac were prepared but not characterized due to poor flow properties at high temperatures. The concentration of the crosslinker, Hexa, was added relative to the concentration of Novolac. The maximum ratio of Novolac to Hexa used in this study was 0.2, which is comparable to the concentrations used in the industry.
### 3.1.2 Chemicals

Cellulose acetate butyrate was purchased from Eastman Chemical Company, Kingsport, Tennessee. The number average molecular weight was reported by the manufacturer to be 70,000. High molecular weight Novolac resin (Mn=900-1,000; Mw=5,000-10,000) was obtained from Plastics Engineering Company (Plenco), Sheboygan, Wisconsin. Hexamethylenetetramine was purchased from Aldrich Chemical Company, Milwaukee, Wisconsin. All chemicals were used without modifications.

#### Table 3.1 Sample matrix

<table>
<thead>
<tr>
<th>Series</th>
<th>CAB* (Wt%)</th>
<th>Novolac (Wt%)</th>
<th>Hexa** (Wt%)*</th>
<th>Novolac/Hexa</th>
<th>Notation</th>
</tr>
</thead>
<tbody>
<tr>
<td>90/10</td>
<td>90</td>
<td>10</td>
<td>0</td>
<td>0</td>
<td>90/10/0</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>10</td>
<td>0.5</td>
<td>0.05</td>
<td>90/10/0.05</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>10</td>
<td>1</td>
<td>0.1</td>
<td>90/10/0.1</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>10</td>
<td>2</td>
<td>0.2</td>
<td>90/10/0.2</td>
</tr>
<tr>
<td>80/20</td>
<td>80</td>
<td>20</td>
<td>0</td>
<td>0</td>
<td>80/20/0</td>
</tr>
<tr>
<td></td>
<td>80</td>
<td>20</td>
<td>1</td>
<td>0.05</td>
<td>80/20/0.05</td>
</tr>
<tr>
<td></td>
<td>80</td>
<td>20</td>
<td>2</td>
<td>0.1</td>
<td>80/20/0.1</td>
</tr>
<tr>
<td></td>
<td>80</td>
<td>20</td>
<td>4</td>
<td>0.2</td>
<td>80/20/0.2</td>
</tr>
</tbody>
</table>

* CAB: cellulose acetate butyrate  
** Hexa: hexamethylenetetramine  
*** The concentration of hexamethylenetetramine is relative to that of Novolac.
3.2 Sample Preparations

3.2.1 Blending

A Brabender plasticorder with two cam blades and a bowl mixer (standard) was used to blend the polymers. The mixer was heated with a temperature-controlled oil circulating system while the temperature was monitored by a K-type thermocouple. The cam blades were rotated at a fixed speed of 30 rpm.

The pre-weighed CAB and Novolac powders were placed in the mixer before heating. As the mixer temperature was raised to 160 °C, the polymer mixture started to melt and became transparent with a light yellow color. The mixture was kept at the maximum blending temperature of 190 °C, for 10 minutes to ensure a homogeneous product.

Hexa crosslinker was then added at 190 °C. At this temperature Hexa reacted with Novolac immediately upon contact, which resulted in a large amount of ammonia released during the reaction. The mixture became opaque and bright yellow. After the addition of Hexa, the blend was mixed for another 10 minutes before it was removed from the mixer. The polymer blend was then air-cooled to the room temperature outside the mixer.

3.2.2 Powder Formation

The crude polymer blends were cracked into small chips less than 1 cm in size with a hammer. The chips were then ground into sandlike powders in a grinding mill, and the powders were collected and stored in glass jars for further studies.
3.2.3 Molding

Two types of vacuum molds were used in this study. A disk mold was used to prepare samples for the rheometer and is shown in Figure 3-1. Bar samples for the three-point bending test were made in the mold shown in Figure 3-2. The mold assembly consisted of three parts: the bottom plate, the main body and the plunger. The bottom plate was attached to the main body with two or four bolts. The plunger was inserted from the top and glided down the cylindrical or rectangular hole by gravity and the force created by the vacuum. For sealing purpose, Viton O-rings were used on the bottom plate and the plunger, respectively. The temperature in the mold was monitored by two K-type thermocouples: one was inserted into the small hole in the plunger and the other into the hole in the bottom plate. The mold was vented and connected to a vacuum pump to minimize bubble formation.

The disk mold was used to make a 25-mm diameter by 2-mm thick disk. Approximately 1 g of sample powder was placed in the mold and the plunger was inserted. The mold was sitting on a heating plate and wrapped with a heating tape. The mold was slowly heated up to the maximum molding temperature, 190 °C, in a process of approximately 25 minutes, and stayed for another 30 minutes. After reaching the desired temperature and time, the mold was air-cooled to the room temperature before the disk was removed in approximately 2 hours.

The bar mold was used to make a 2x12x50-mm bar. The bars were molded using basically the same process as the disk molding, except that the ammonia off-gassing was much more severe during this process. The molding temperature of the bar was similarly between 180 and 190 °C but the heating schedule was more complicated than that of the disk. Table 3-2 demonstrated the best heating condition of the successfully-molded bars.
Table 3-2 The best heating schedule of molding bars

<table>
<thead>
<tr>
<th>Time [min]</th>
<th>0 - 30</th>
<th>30 - 45</th>
<th>45 - 60</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heating plate</td>
<td>Level 2</td>
<td>Off</td>
<td>Off</td>
</tr>
<tr>
<td>Heating tape</td>
<td>90% Power</td>
<td>60% Power</td>
<td>75% Power</td>
</tr>
</tbody>
</table>
Figure 3-1 A schematic of the disk mold assembly
Figure 3-2 A schematic of the bar mold assembly
3.3 Chemical, Thermal, Rheological and Mechanical Characterizations:

3.3.1 Chemical Analysis

3.3.1.1 Pyrolysis Molecular Beam Mass Spectrometry (p-MBMS)

The chemical composition of Novolac following the reaction with Hexa was examined with pyrolysis molecular beam mass spectroscopy (p-MBMS). The p-MBMS is described in detail elsewhere \(^1\), \(^2\) and will be only briefly described here. The experimental system consists of a pyrolysis furnace coupled to a molecular beam mass spectrometer. Between 10 and 20 mg of the CAB/Novolac sample was inserted into the pyrolysis furnace held at 550 °C. The sample was rapidly decomposed to yield primary pyrolysis vapors that were carried into the orifice of the p-MBMS by the hot nitrogen stream that has been preheated to 500 C. Residence time in the pyrolysis furnace was less than 0.5 seconds. The vapors were rapidly expanded in a nearly adiabatic and isentropic manner, which resulted in a very rapid quenching. With a low pressure the vacuum stage and a very low pressure second stage the molecular beam collimated, focused, and introduced into the ion source of the mass spectrometer. The molecular beam collided with a low energy electron beam providing a positive ion mass spectrum. Similar systems had been used for the characterization of phenolic polymers including phenolic resins \(^3\)-\(^5\) and lignin\(^1\).

Since the goal of this work was to confirm that the addition of Hexa to the CAB/Novolac blends had resulted in a reaction of the Novolac component, the analysis of the resulting mass spectrum was limited to a study of the phenolic fragments. The four fragments of interest were phenol (m/z = 94), 2- or 4-methyl phenol (ortho or para cresol) (m/z = 108), 2,4- or 2,6-dimethyl phenol (xylensols) (m/z = 122), and 2,4,6-trimethyl phenol (m/z = 136). No attempt was made to resolve the isomers. The response factors for these compounds vary slightly, so simple ratios of the three higher mass fragments to that of phenol was used to monitor the effect of the added Hexa. This analysis allowed for the determination of trends in the data.
Replicated CAB/Novolac samples were prepared for each Hexa concentration and each sample was run in replicate. The four ratios obtained from these samples were averaged. This analysis was conducted by Dr. Steve Kelley at National Renewable Energy Lab, Golden, Colorado.

3.3.1.2 Swelling Measurement

The swelling test was carried out with methyl ethyl ketone. A small chunk of polymer blend weighing around 2 g was placed in a 100-mL beaker with 50-mL solvent. The mixture was stirred to enhance mass transfer. The experiment lasted for 24 hours. Filtration was performed if necessary.

3.3.2 Thermal Analysis: Differential Scanning Calorimetry (DSC)

The thermal property measurement was performed with a differential scanning calorimeter (Shimadzu DSC-50). The sample was sealed in an aluminum pan. A heating rate of 10 °C/min and a nitrogen purge rate of 20 mL/min were used in all experiments. All samples were first scanned from room temperature to 190 °C and then slowly cooled to room temperature. This removed any residual stress and the thermal history related to the blending process. A second scan was performed similarly from room temperature to 190 °C, from which the glass transition temperature was determined as the midpoint of the second order transition(s).

3.3.3 Rheological Analysis: Dynamic Oscillatory Shear

Dynamic oscillatory shear testing was performed using a Bohlin Constant Stress Rheometer (CS-50) with parallel plate geometry (25-mm plate). The temperature range studied was 140 to 190°C, and the frequency was selected from 0.001 to 30 Hz. Two
sample disks of each blend were tested. Time-temperature superposition was performed to obtain master curves of $G'(\omega)$ and $G''(\omega)$ at a reference temperature of 150 °C.

### 3.3.4 Mechanical Analysis: Three Point Bending

A three-point bending test was conducted on an Instron testing machine. At least two samples of each composition were tested and the results were averaged. The results were used to calculate Modulus of Elasticity (MOE) and Modulus of Rupture (MOR).

### 3.4 References

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Chemical Analysis

4.1.1 Molecular beam mass spectrometry (MBMS)

The purpose of this analysis was to measure the formation of methylene bridges on Novolac after reaction with the crosslinker, Hexamethylenetetramine. If the network was in low branching regime, the products detected after pyrolysis would be phenol, cresol and xylenol. If the system was highly branched, the pyrolyzed products would be phenol, cresol, xylenol and trimethylphenol as shown in Figures 4-1 and 4-2. Previous work \(^1\-^3\) had shown that all four phenolic compounds could be found in phenolic resins.

A simple statistical analysis of a hypothetical, perfectly linear phenolic resin showed that there will be a 1:2:1 distribution of phenol, methyl phenol and dimethyl phenol fragments produced by a random cleavage of the phenolic polymer. Adding Hexa to this perfectly linear polymer would result in the formation of methylene bridges (branch points) and eventually a crosslinked system. As the degree of branching increased, the relative amount of unsubstituted and mono-substituted phenolic fragments would decrease, and trimethyl fragments would appear. Again, a simple statistical analysis of a completely crosslinked system, where three reactive sites were occupied by a methylene bridge, would show a ratio of phenol, methyl phenol, dimethyl phenol, trimethyl phenol of 1:3:3:1, respectively. Thus, the ratio of these four primary phenolic compounds was an indication of the degree of branching or crosslinking in the Novolac resin. And an increase in branching or the formation of crosslinks could be monitored by monitoring the increase in the more highly substituted dimethyl and trimethyl phenol fragments.
Figure 4-1 A schematic of low branched Novolac and its pyrolyzed products

Figure 4-2 A schematic of high branched Novolac and its pyrolyzed products
The results of the p-MBMS analysis of 90/10 and 80/20 series of CAB/Novolac blends, before and after their reaction with Hexa, were shown in Figures 4-3 and 4-4. The Y-axis represented the mass ratio of methylene linkages to phenol and the X-axis was the ratio of Hexamethylenetetramine to Novolac. These Figures showed that the relative amount of more highly substituted fragments increased as the Hexa/Novolac ratio increased, consistent with an increase in the number of methylene bridges connecting the phenolic chains.

In Figure 4-3 the distribution of fragments for Novolac with no added Hexa showed that methyl phenol (mass 108) was the most common of the substituted fragments, and there was less of the more highly substituted fragments, dimethyl and trimethyl phenol, (masses 122 and 136). As Hexa was added to the CAB/Novolac blend, the relative amount of all of the more highly substituted fragments increased. And, based on an analysis of the raw data (see appendix) the relative amount of unsubstituted phenol decreased. Comparing the methyl substituted fragments to one another, the amount of dimethyl phenol increased more rapidly than methyl phenol, and at a Hexa/Novolac ratio of 0.2 the relative amounts of these two fragments were similar. This result suggested that most of the available sites on the Novolac polymer have reacted.

Similar trends were seen in Figure 4-4. Again the relative amount of substituted fragments increased as the Hexa/Novolac ratio increased. The relative amount of dimethyl phenol increased more rapidly than that of methyl phenol, suggesting that again the Novolac had been reacted to a large extent, although not as completely as seen for the 90/10 series.

Both sets of CAB/Novolac blends showed a distinct increase in the relative ratios of highly substituted phenolic fragments with increasing Hexa content. This was direct chemical evidence for the effective reaction between the Novolac and Hexa. However, this analysis can not distinguish intramolecular reactions from intermolecular reactions.
Figure 4-3 Effect of Hexa on the formation of methylene linkages on 90/10 series blends

Figure 4-4 Effect of Hexa on the formation of methylene linkages on 80/20 series blends
Figure 4-5 Effect of crosslinker on the formation of methylene linkages in both the 90/10 and 80/20 series blends.
Figure 4-5 showed the MBMS results of two series of blends, 90/10 and 80/20. The unfilled symbols showed the data for 90/10 series and the filled ones represented the 80/20 series. This Figure showed that the filled symbols at all concentrations exhibited higher values than the unfilled ones. It indicated that 80/20 blends formed more methylene linkages than 90/10 blends. Since 80/20 blends contained more Novolac, they offer more opportunity for effective reactions between Novolac and Hexa.

4.1.2 Swelling measurement

After a one-day soak in methyl ethyl ketone, all polymer blends (blends with and without crosslinker) dissolved completely. Dissolution of all samples indicated that none of the blends were crosslinked. Solutions of blends prepared with higher Hexa/Novolac ratios were more viscous than those that did not contain Hexa. This result suggested the formation of more branching systems with the addition of more Hexa.

The combined results from the p-MBMS analysis and the swelling tests provided some insight into the molecular architecture of the reacted CAB/Novolac system. The p-MBMS analysis demonstrated a significant increase in the number of methylene linkages, while the complete solubility of the CAB/Novolac blends showed that the “cured” product was not crosslinked. These results were consistent with the formation of more intramolecular, rather than intermolecular linkages. A limited number of intermolecular reactions could be expected since the reactive Novolac chains were “diluted” by a large excess of CAB chains, reducing the physical proximity of the chains.

4.2 Thermal Analysis – Differential Scanning Calorimetry (DSC)

Figures 4-6 to 4-15 showed the DSC curves of neat CAB, Novolac and both 90/10 and 80/20 series blends. All results shown were from the second scan. A single glass
Figure 4-6 DSC diagram of neat CAB

Figure 4-7 DSC diagram of neat Novolac
Figure 4-8 DSC diagram of 90/10/0 blend

Figure 4-9 DSC diagram of the 90/10/0.05 blend
Figure 4-10 DSC diagram of the 90/10/0.1 blend

Figure 4-11 DSC diagram of the 90/10/0.2 blend
Figure 4-12 DSC diagram of the 80/20/0 blend

Figure 4-13 DSC diagram of the 80/20/0.05 blend
Figure 4-14 DSC diagram of the 80/20/0.1 blend

Figure 4-15 DSC diagram of the 80/20/0.2 blend
transition temperature (Tg) was observed in each CAB/Novolac blend, which strongly suggested that all CAB/Novolac blends were completely miscible.

Glass transition temperatures of all components were given in Table 4-1 and the relationship between Tg and the concentration of the crosslinker was displayed in Figure 4-16. The glass transition temperatures of CAB and Novolac were 136 °C and 72 °C, respectively. The glass transition temperatures of both 90/10/0 and 80/20/0 were 111 °C and 106 °C, respectively. The Tg of blends containing no Hexa indicated that the Novolac acted as a plasticizer. Addition of Hexa caused an increase in the Tg of the blends.

The Tg's of the blends without crosslinkers were 111 °C for 90/10 series and 106 °C for 80/20 series. These values did not correlate very well with the glass transition temperature predicted by the rule of mixtures as shown in Table 4.2. These differences had been seen with several cellulose ester/phenolic polymer systems and might be due to disruption of intermolecular interactions between the relatively stiff cellulose chains.
Table 4-1
Tg values of CAB, Novolac and the 90/10 and 80/20 blends

<table>
<thead>
<tr>
<th>Series Name</th>
<th>CAB Wt%</th>
<th>Novolac Wt%</th>
<th>Hexa/Novolac Wt%</th>
<th>Tg (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neat CAB</td>
<td>100</td>
<td>0</td>
<td>---</td>
<td>136</td>
</tr>
<tr>
<td>Neat Novolac</td>
<td>0</td>
<td>100</td>
<td>---</td>
<td>72</td>
</tr>
<tr>
<td>90/10</td>
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<td>10</td>
<td>0</td>
<td>111</td>
</tr>
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<td></td>
<td>90</td>
<td>10</td>
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<td>129</td>
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<td>0.1</td>
<td>131</td>
</tr>
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<td></td>
<td>90</td>
<td>10</td>
<td>0.2</td>
<td>142</td>
</tr>
<tr>
<td>80/20</td>
<td>80</td>
<td>20</td>
<td>0</td>
<td>106</td>
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<td>133</td>
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<td>80</td>
<td>20</td>
<td>0.2</td>
<td>140</td>
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</table>
Figure 4-16 Effect of Hexa on Tg
Table 4-2
Comparison of actual Tg and predicted Tg of blends containing no Hexa

<table>
<thead>
<tr>
<th>Blend</th>
<th>90/10</th>
<th>80/20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actual Tg</td>
<td>111 °C</td>
<td>106 °C</td>
</tr>
<tr>
<td>Predicted Tg*</td>
<td>125 °C</td>
<td>115 °C</td>
</tr>
</tbody>
</table>

* \( \frac{1}{Tg} = \frac{w_1}{Tg_1} + \frac{w_2}{Tg_2} \) where w was the weight fraction of each component

The general increase in the Tg with the addition of Hexa could be explained in terms of an increase in the molecular weight of the Novolac. An increase in the molecular weight of the Novolac increased the Tg of the Novolac and provided for a corresponding increase in the Tg of the CAB/Novolac blend. At higher Hexa/Novolac ratios above 0.15 the effectiveness of additional Hexa was low. This effect might be due to a limited number of reactive sites on the Novolac and the “dilute” nature of the reactive system which meant that the potential reactive sites did not come close enough contact to allow for an effective reaction, and a further increase in molecular weight.

4.3 Rheological Test – Dynamic Oscillatory Shear

Figures 4-17 and 4-18, respectively, showed the results of dynamic oscillatory shear measurements on the 90/10 and 80/20 series of blends. Very good superposition in G’ and G” curves were seen in both series. In Figures 4-17 and 4-18, the G’ and G” curves moved upward as the concentration of the crosslinker increased while the crossover frequency shifted to a lower region. Differences between G’ and G” at the plateau region increased with the additional Hexa though the shapes of the G’ and G” curves were similar at all three Hexa concentration levels.
Figure 4-17 $G'$ and $G''$ curves of the 90/10 series
Figure 4-18 $G'$ and $G''$ curves of the 80/20 series
In Figure 4-18 (80/20 series), the $G'$ and $G''$ curves of the blends with no crosslinker (80/20/0) showed a much higher crossover frequency with lower $G'$ and $G''$ values than the blends with Hexa (80/20/0.1 and 80/20/0.2). A dramatic jump of the $G'$ and $G''$ curves and a big decrease in the crossover frequency were observed after adding Hexa, the crosslinker. Similar trends were seen in the 90/10 series (Figure 4-17), although the addition of the crosslinker showed a larger effect on 80/20 blends than on 90/10 blends. This might be due to the formation of more intermolecular linkages in the 80/20 blends.

Figures 4-19 and 4-20 compared the effect of crosslinker on 90/10 and 80/20 blends. When no crosslinker was present (Hexa/Novolac = 0), the 90/10/0 blend exhibited higher values of $G'$ and $G''$ and a lower crossover frequency. After Hexa was added, the $G'$ and $G''$ curves of the 80/20 sample increased and the crossover occurred at a much lower frequency. After the Hexa addition, larger changes in the shapes of two curves were observed in 80/20 series compared to the 90/10 blends. Once again, the addition of crosslinker showed a bigger influence on the 80/20 blends than the 90/10 blends.

Table 4-3 listed the crossover frequencies and the differences of $G'$ and $G''$ at the plateau region for all the blends with different concentrations of the crosslinker. As the crosslinker concentration increased, the crossover frequency decreased and the difference of $G'$ and $G''$ at the plateau region grew larger. More significant changes were observed in 80/20 blends where the content of Novolac was higher.
Figure 4-19 Comparison of $G'$ and $G''$ curves of the 90/10 and 80/20 series before the addition of Hexa (Hexa/Novolac=0)
Figure 4-20 Comparison of G' and G'' curves of the 90/10 and 80/20 series before the addition of Hexa (Hexa/Novolac=0.2)
Table 4-3
Numerical values of the crossover frequency and the modulus difference at the plateau region at different crosslinker concentrations

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<th>Ratio of Hexa % to Novolac %</th>
<th>Crossover Frequency [Hz]</th>
<th>G'/G'' at the Plateau Region</th>
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The rheological data showed that in both the 90/10 and 80/20 series, G' and G'' increased with the amount of Hexa in the system while the crossover was lower. The crossover frequency was the reciprocal of the maximum relaxation time, an important indication of molecular weight. Therefore these results signified that the molecular weight of the blend was increasing with increasing concentration of Hexa.

The difference between G' and G'' at the plateau region was another indication of molecular weight, with greater differences corresponding to higher molecular weight. Difference between G' and G'' became larger when more Hexa was added into the blends as seen in both Figures 4-17 and 4-18. This supported previous evidence that the blend with more Hexa had higher molecular weight. The shapes of the G' and G'' curves could also offer insight into the polymer blends. The curves in Figure 4-17 and 4-18 did not show significant differences in the shape, which supported the results of the swelling measurements that showed that none of the blends were crosslinked.
The G' and G'' curves of neat CAB was shown in Figure 4-21. The crossover frequency of neat CAB was $3.00 \times 10^{-4}$ Hz. The rheological behavior of neat CAB and two series (90/10 and 80/20) were shown in Figures 4-22 and 4-23, respectively. The neat CAB exhibited rheological behavior similar to 90/10/0.2 blend as shown in Figure 4-22. The G' and G'' curves of the neat CAB lied between those of 80/20/0 and 80/20/0.1 blends.

A comparison between neat CAB and 90/10 blends indicated that the blend with 90% CAB and 10% Novolac could reach an equivalent strength at the maximum addition of the crosslinker. When 80/20 series blends were compared to neat CAB, the blends with 80% CAB and 20% Novolac could easily demonstrated better rheological behavior with the addition of a small amount of Hexa. These comparisons also signified that growing the molecular weight of CAB/Novolac blend could provide better rheological properties.

When no crosslinker was added, lower G' and G'' curves and higher crossover frequency were observed in both 90/10 and 80/20 blends. In Figure 4-18, the G' and G'' curves of 80/20/0 blend were much lower and crossed at a much higher crossover frequency than the other two concentrations while in Figure 4-19, 80/20/0 blend again showed lower trends G' and G'' curves than 90/10/0 sample. It was because that Novolac, a low molecular weight polymer, acted as a plasticizer in the CAB/Novolac blend. The plasticizer diluted the system and would reduce the rheological properties. 80/20 blend had a higher content of Novolac and therefore had a larger reduction in G', G'' and the maximum relaxation time.
Figure 4-21 $G'$ and $G''$ curves of neat CAB
Figure 4-22 Comparison of $G'$ and $G''$ curves between neat CAB and the 90/10 series.
Figure 4-23 Comparison of $G'$ and $G''$ curves between neat CAB and blends with 80% CAB/20% Novolac (80/20 series)
Since Novolac was the only chemical providing phenolic groups and reacting with Hexa, there were more reaction taking place in 80/20 blends than in 90/10 series. It was anticipated that with a higher content of Novolac, the system would become more branched, resulting higher molecular weight blend. The results shown in Figures 4-19 and 4-20 fully supported this expectation. When the crosslinker was added, it was apparent that the $G'$, $G''$ and the maximum relation time of 80/20 blend were much bigger than those of 90/10 blend. Though the polymers without the crosslinker exhibited lower $G'$ and $G''$ than neat CAB in both series, the blends with Hexa showed a satisfying effect. From Figure 4-22 and 4-23, the increases in $G'$, $G''$ and the maximum relaxation time were more dramatic in 80/20 blends than in 90/10. These observations supported the formation of a higher molecular weight polymer by introducing Hexamethylenetetramine to the blend of CAB and Novolac.

4.4 Mechanical Analysis – Three Point Bending

The results of the three-point bending test on CAB/Novolac blends were displayed in Figures 4-24 to 4-31. An arbitrary displacement unit was used in plotting the load-displacement diagrams since the actual displacement exceeded the instrument limitation. All the curves of 90/10 blends reached the maximum load and then dropped before failing. While the 80/20 blends broke into half or three pieces at the maximum load, as shown in the Figures 4-28 to 4-31.

To calculate the modulus of elasticity (MOE), a diagram of load versus actual displacement was plotted as shown in Figure 4-32. Take the 90/10/0.2 blend as an example. The slope of the curve, $(\Delta F/\Delta d)$, obtained from Figure 4-32 was 0.7977. The value of the modulus of elasticity could be calculated with the following equation (Eq. 4-1).

$$
\text{MOE} = \frac{\Delta F L^3}{4 \ b \ d^3 \Delta d}
$$

Eq. 4-1

where $L$ is 50 mm, $b$ is 12 mm and $d$ is 2 mm
Figure 4-24 Load-arbitrary displacement diagram of the 90/10/0 blend

Figure 4-25 Load-arbitrary displacement diagram of the 90/10/0.05 blend
Figure 4-26 Load-arbitrary displacement diagram of 90/10/0.1 blend

Figure 4-27 Load-arbitrary displacement diagram of the 90/10/0.2 blend
Figure 4-28 Load-arbitrary displacement diagram of the 80/20/0 blend

Figure 4-29 Load-arbitrary displacement diagram of the 80/20/0.05 blend
Figure 4-30 Load-arbitrary displacement diagram of the 80/20/0.1 blend

Figure 4-31 Load-arbitrary displacement diagram of the 80/20/0.2 blend
Figure 4-32 Load-actual displacement diagram of the 90/10/0.2 blend
The modulus of rupture (MOR) was derived by inserting the maximum load and sample dimensions into Eq. 4-2. The maximum load ($F_{\text{Max}}$) of this sample was 4.528 kg as shown in Figure 4-27. 

$$\text{MOR} = \frac{3 F_{\text{Max}} L}{2 b d^2}$$ \hspace{1cm} \text{Eq. 4-2}

The results of MOE and MOR of both series were shown in Figure 4-33. The right Y-axis represented MOE and the left Y-axis represented MOR. When no Hexa was added, both MOE and MOR of 80/20 blend were higher than those of 90/10 blend were. When Hexa was added, both moduli began to fall off for all blends.

Without any crosslinker in the system, 80/20 blend showed higher MOE and MOR than 90/10 blend. These results suggested that the stiff Novolac acted as a filler to reinforce the blend, causing the blend to become stiffer and thus enhancing MOE and MOR. It was further anticipated that when the chains in the stiff Novolac grew due to the addition of Hexa, the values of MOE and MOR should also increase. It was expected that the MOE and MOR of blends with higher Hexa contents should be larger than those of the blends without the crosslinker, however, the results shown in Figure 4-33 were not consistent with this expectation.

4.5 References

Figure 4-33 Effect of the addition of Hexa on modulus of elasticity and modulus of rupture of the 90/10 and 80/20 blends
CHAPTER 5
CONCLUSION AND FUTURE WORK

5.1 Conclusion

The initial goal of this work was to prepare a series of semi-interpenetrating polymer networks by crosslinking the Novolac component in the CAB/Novolac blends with hexamethylenetetramine. The results of the swelling measurement showed that none of the cellulose acetate butyrate/Novolac blends were crosslinked and thus interpenetrating polymer networks were not formed. However, the p-MBMS analysis provided direct chemical evidence that with the addition of hexamethylenetetramine, more methylene bridges were formed on Novolac chains and Novolac became more branched. The DSC and dynamic oscillatory shear measurements further supported the conclusion that the molecular weight of the polymer blend increased with the addition of crosslinker. Specifically the glass transition temperature, maximum relaxation time and dynamic modulus all increased, reflecting the effect of chain extension. It can be concluded that the blends possessed higher molecular weight which resulted from chain extension but were not crosslinked. The failure in crosslinking Novolac might be due to the small size of Novolac and its low content in the mixture. The mechanical properties (modulus of elasticity and modulus of rupture), unexpectedly, decreased with the addition of hexamethylenetetramine. These results strongly suggested that cellulose acetate butyrate has undergone some chemical degradation.

5.2 Future Work

Further testing on mechanical properties, including creep resistance and impact strength, should be conducted to complete the investigation on the effect of the crosslinker on the strengths of the cellulose acetate butyrate/Novolac blends. The decrease in mechanical
properties with the addition of the crosslinker could be explained by gel permeation chromatography analysis that could show the actual molecular weight of each sample. If there was degradation or bond breakage taking place in cellulose acetate butyrate, new methods of blending and molding should be sought. Additionally, future studies should focus on the formation of an interpenetrating polymer network. To obtain a fully crosslinked system, replacing Novolac with a higher molecular weight phenolic polymer such as polyvinylphenol and increasing the content of the phenolic polymer in the cellulose ester/phenolic blend are alternatives.
Bibliography


APPENDICES
# TABLE OF CONTENTS IN APPENDICES

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<td>B. Shifting Factors of the 90/10 and 80/20 Series</td>
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<td>C. Original Rheological Data</td>
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Table B-1 Shifting factors of 90/10 series blends at a reference temperature of 150 °C

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Table B-2 Shifting factors of 80/20 series blends at a reference temperature of 150 °C

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Table C-2 Rheological data of the blend containing 90% CAB, 10% Novolac and 0% Hexa [90/10/0] at 150 °C

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Table C-5 Rheological data of the blend containing 90% CAB, 10% Novolac and 1% Hexa (Hexa/Novolac=0.1) [90/10/0.1] at 140 °C

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Table C-7 Rheological data of the blend containing 90% CAB, 10% Novolac and 1% Hexa (Hexa/Novolac=0.1) [90/10/0.1] at 160 °C

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Table C-8 Rheological data of the blend containing 90% CAB, 10% Novolac and 1% Hexa (Hexa/Novolac=0.1) [90/10/0.1] at 170 °C

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Table C-12: Rheological data of the blend containing 90% CAB, 10% Novolac and 2% Hexa (Hexa/Novolac=0.2) [90/10/0.2] at 150 °C

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Table C-13 Rheological data of the blend containing 90% CAB, 10% Novolac and 2% Hexa (Hexa/Novolac=0.2) [90/10/0.2] at 160 °C

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Table C-14 Rheological data of the blend containing 90% CAB, 10% Novolac and 2% Hexa (Hexa/Novolac=0.2) [90/10/0.2] at 170 °C

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Table C-15 Rheological data of the blend containing 90% CAB, 10% Novolac and 2% Hexa (Hexa/Novolac=0.2) [90/10/0.2] at 180 °C

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Table C-16 Rheological data of the blend containing 90% CAB, 10% Novolac and 2% Hexa (Hexa/Novolac=0.2) [90/10/0.2] at 190 °C

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Table C-17 Rheological data of the blend containing 80% CAB, 20% Novolac and 0% Hexa (Hexa/Novolac=0) [80/20/0] at 140 °C

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Table C-18 Rheological data of the blend containing 80% CAB, 20% Novolac and 0% Hexa (Hexa/Novolac=0) [80/20/0] at 150 °C

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Table C-19 Rheological data of the blend containing 80% CAB, 20% Novolac and 0% Hexa (Hexa/Novolac=0) [80/20/0] at 160 °C

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Table C-20 Rheological data of the blend containing 80% CAB, 20% Novolac and 0% Hexa (Hexa/Novolac=0) [80/20/0] at 170 °C

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Table C-22 Rheological data of the blend containing 80% CAB, 20% Novolac and 0% Hexa (Hexa/Novolac=0) [80/20/0] at 190 °C

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Table C-23 Rheological data of the blend containing 80% CAB, 20% Novolac and 2% Hexa (Hexa/Novolac=0.1) [80/20/0.1] at 140 °C

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Table C-24 Rheological data of the blend containing 80% CAB, 20% Novolac and 2% Hexa (Hexa/Novolac=0.1) [80/20/0.1] at 150 °C

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Table C-25 Rheological data of the blend containing 80% CAB, 20% Novolac and 2% Hexa (Hexa/Novolac=0.1) [80/20/0.1] at 160 °C

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Table C-26 Rheological data of the blend containing 80% CAB, 20% Novolac and 2% Hexa (Hexa/Novolac=0.1) [80/20/0.1] at 170 °C

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Table C-27 Rheological data of the blend containing 80% CAB, 20% Novolac and 2% Hexa (Hexa/Novolac=0.1) [80/20/0.1] at 180 °C

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Table C-28 Rheological data of the blend containing 80% CAB, 20% Novolac and 2% Hexa (Hexa/Novolac=0.1) [80/20/0.1] at 190 °C

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Table C-29 Rheological data of the blend containing 80% CAB, 20% Novolac and 4% Hexa (Hexa/Novolac=0.2) [80/20/0.2] at 140 °C

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Table C-30 Rheological data of the blend containing 80% CAB, 20% Novolac and 4% Hexa (Hexa/Novolac=0.2) [80/20/0.2] at 150 °C

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Table C-31 Rheological data of the blend containing 80% CAB, 20% Novolac and 4% Hexa (Hexa/Novolac=0.2) [80/20/0.2] at 160 °C

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Table C-32 Rheological data of the blend containing 80% CAB, 20% Novolac and 4% Hexa (Hexa/Novolac=0.2) [80/20/0.2] at 170 °C

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Table C-33 Rheological data of the blend containing 80% CAB, 20% Novolac and 4% Hexa (Hexa/Novolac=0.2) [80/20/0.2] at 180 °C

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Table C-34 Rheological data of the blend containing 80% CAB, 20% Novolac and 4% Hexa (Hexa/Novolac=0.2) [80/20/0.2] at 190 °C

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Figure D-1 Rheological behavior of 90/10/0 blend at 140 °C

Figure D-2 Rheological behavior of 90/10/0 blend at 150 °C
Figure D-3 Rheological behavior of 90/10/0 blend at 170 °C

Figure D-4 Rheological behavior of 90/10/0 blend at 190 °C
Figure D-5 G' curves of 90/10/0 blend in the range of 140-190 °C

Figure D-6 G'' curves of 90/10/0 blend in the range of 140-190 °C
Figure D-7 Rheological behavior of 90/10/0.1 blend at 140 °C

Figure D-8 Rheological behavior of 90/10/0.1 blend at 150 °C
Figure D-9 Rheological behavior of 90/10/0.1 blend at 160 °C

Figure D-10 Rheological behavior of 90/10/0.1 blend at 170 °C
Figure D-11 Rheological behavior of 90/10/0.1 blend at 180 °C

Figure D-12 Rheological behavior of 90/10/0.1 blend at 190 °C
Figure D-13 $G'$ curves of 90/10/0.1 blend in the range of 140-190 °C

Figure D-14 $G''$ curves of 90/10/0.1 blend in the range of 140-190 °C
Figure D-15 Rheological behavior of 90/10/0.2 blend at 140 °C

Figure D-16 Rheological behavior of 90/10/0.2 blend at 150 °C
Figure D-17 Rheological behavior of 90/10/0.2 blend at 160 °C

Figure D-18 Rheological behavior of 90/10/0.2 blend at 170 °C
Figure D-19 Rheological behavior of 90/10/0.2 blend at 180 °C

Figure D-20 Rheological behavior of 90/10/0.2 blend at 190 °C
Figure D-21 $G'$ curves of 90/10/0.2 blend in the range of 140-190 °C

Figure D-22 $G''$ curves of 90/10/0.2 blend in the range of 140-190 °C
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Figure D-24 Rheological behavior of 80/20/0 blend at 150 °C
Figure D-25 Rheological behavior of 80/20/0 blend at 160 °C

Figure D-26 Rheological behavior of 80/20/0 blend at 170 °C
Figure D-27 Rheological behavior of 80/20/0 blend at 180 °C

Figure D-28 Rheological behavior of 80/20/0 blend at 190 °C
Figure D-29 $G'$ curves of 80/20/0 blend in the range of 140-190 °C

Figure D-30 $G''$ curves of 80/20/0 blend in the range of 140-190 °C
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Figure D-32 Rheological behavior of 80/20/0.1 blend at 150 °C
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Figure D-34 Rheological behavior of 80/20/0.1 blend at 170 °C
Figure D-35 Rheological behavior of 80/20/0.1 blend at 180 °C

Figure D-36 Rheological behavior of 80/20/0.1 blend at 190 °C
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Figure D-38 G'' curves of 80/20/0.1 blend in the range of 140-190 °C
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Figure D-40 Rheological behavior of 80/20/0.2 blend at 150 °C
Figure D-41 Rheological behavior of 80/20/0.2 blend at 160 °C

Figure D-42 Rheological behavior of 80/20/0.2 blend at 170 °C
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Figure D-44 Rheological behavior of 80/20/0.2 blend at 190 °C
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Figure D-46 $G''$ curves of 80/20/0.2 blend in the range of 140-190 °C