Evaluation of a Modified pH-Shift Process to Reduce 2-Methylisoborneol and Geosmin in Spiked Catfish and Produce a Consumer Acceptable Fried Catfish Nugget-Like Product


Abstract: Muddy and/or musty off-flavors in farmed-raised catfish occur as a result of the absorption of geosmin (GEO) and 2-methylisoborneol (MIB), compounds produced by algae. Previous research suggests the acid pH-shift method may be able to reduce off-flavors to produce a consumer acceptable product. The objective of this research was to evaluate application of the acid pH-shift method using a shaker sieve for protein recovery and to evaluate consumer acceptability of a resultant batter-coated fried nugget-like catfish product. Farm-raised catfish were either allowed to depurate (control) or treated with 1 ppb GEO or MIB. Fillets from each replicate were collected and ground and treated by the acid pH-shift process. Samples from all treatments and replicates were evaluated for residual GEO and MIB. In addition, batter-coated fried catfish samples were prepared for a consumer sensory evaluation. Results demonstrated that the pH-shift process decreased moisture, ash, and collagen content of catfish fillet tissue ($P < 0.05$). Flavor of control samples was preferred ($P < 0.05$). Texture of catfish samples treated by the pH-shift process was preferred ($P < 0.05$). Results demonstrate the pH-shift process can be utilized to reduce off-flavors and increase the acceptability of a processed catfish product.

Keywords: catfish, geosmin, methylisoborneol, off-flavor, pH-shift

Practical Application: Use of a sieve as an economic alternative for the pH-shift process was evaluated for removing off-flavors from catfish. Difficulties were encountered with regard to protein recovery using the sieve and suggestions are made to, perhaps, make the process more applicable for a sieve-based recovery step. The process as described reduced off-flavors, but only 2-fold suggesting the process would work best on catfish near or just over off-flavor thresholds. Results also indicated the pH-shift process could be used to improve texture of a fried catfish product designed to be similar to chicken nuggets.

Introduction

U.S. farm-raised catfish growers processed 471 million pounds and reported sales of $423 million during 2011 (USDA-NASS 2012a, 2012b). The most widely used production technology system for farm-raised catfish is pond production (Lazar and Britt 1997) which depends on photosynthetic algae and beneficial bacteria to improve water oxygen levels. However, one problem resulting from these systems includes the production of odorous metabolite compounds, geosmin (GEO) and 2-methylisoborneol (MIB). As a result, off-flavor in pond cultured catfish has been associated with the absorption of GEO and MIB (Klausen and others 2005) which result in the development of muddy (MIB) or musty (GEO) flavors (Grimm and others 2004). Cultured ponds and purging facilities are used to remove these off-flavors (Tucker and van der Ploeg 1999) which can delay harvest up to several days or even months (Schrader and others 2003). King and Dew (2003) reported that approximately 80% of harvestable fish each year can and will be considered off-flavored which can result in economic losses of approximately $60 million annually (Schrader and others 2003).

Preventative preharvest methods to reduce or remove off-flavors/odors in catfish have been attempted. Unfortunately, the majority fail to provide safety, time effectiveness, and/or economic efficiency in a typical production type scenario (Tucker and van der Ploeg 1999). Thus, postharvest methods for removal of off-flavor may be more successful than preharvest methods.

Postharvest off-flavor removal includes methods to mask or physically/chemically remove off-flavor compounds. Mireles DeWitt and others (2007b) determined the acid pH-shift method (Hultin and Kelleher 1997) can reduce GEO and MIB in catfish. Kristinsson and others (2005) demonstrated that application of
acid pH-shift process using centrifugal conditions of 10,000 × g to recover proteins resulted in higher protein recovery, lower lipids, and higher whiteness values when compared to surimi processing. Mireles DeWitt and others (2007b) also compared the acid pH-shift process using centrifugal conditions of 3000 × g and found the resultant raw and cooked product contained lower lipids than untreated catfish. Improved whiteness was only observed in raw pH-shift treated product. In addition, it was observed that although water holding ability was reduced in the pH-shift product, there was no difference in gel cook yield and texture profile parameters were significantly higher. Davenport and Kristinnson (2011) also demonstrated improved gel strength of acid pH-shift treated catfish when compared to ground muscle. In addition, they observed that cook loss was not impacted by the pH-shift process and noted that pH-shift process catfish muscle may provide an isolate that does not need cryoprotectants and phosphates.

A major hurdle to the commercial application of the pH-shift process is the recovery of the flocculated protein by centrifugation. The objective of this study was to evaluate the use of a shaker screen in place of the centrifugation step as a more economical method of protein recovery and to determine its effectiveness in reducing off-flavor compounds GEO and MIB. In addition, evaluations were conducted to determine the ability of the pH-shift method to produce a consumer-acceptable fried catfish product designed to be similar to a chicken nugget.

Materials and Methods

Live catfish treatment and harvest

Due to restrictions in catfish holding tank sizes, interconnection of the recirculating systems and labor required for processing catfish each treatment was applied on a different day. For example, nonspiked catfish (control) were processed in week 1, MIB-spiked catfish were spiked in week 2, GEO-spiked in week 3. Catfish were obtained from ponds at Langston Univ. All catfish obtained in this study were fasted for 1 wk to enhance excretion of food and bile. Approximately 68 kg (about 60 catfish) of live pond raised channel catfish (Ictalurus punctatus) were collected and randomly distributed among 3 1050 L high-density polyethylene plastic tanks (Polystank Inc., Litchfield, Minn., U.S.A.) each containing 1000 L of aerated municipal water at 20 °C. More catfish than needed for the study were placed in tanks at this stage to ensure a 1:1 male:female ratio. Tanks were interconnected and filtered in a recirculating system using a floating bead filter. For the next 24 h, catfish were allowed to acclimate and depurate in the filtered tank water. Following purging, tanks were drained and refilled with 945 L of fresh municipal water. Water was sampled from each tank at this time to evaluate initial MIB and GEO levels. Fish were held in tanks for another 24 h. Additional water samples were obtained at 30 min and 24 h. All water samples were collected in clear glass vials without headspace. They were stored at 4 °C until shipping. Once the final “tank water” samples were collected, the vials were all packed on ice, and shipped via overnight delivery to the Thad Cochran Research Center for MIB and GEO analysis. Catfish were harvested by placing them into coolers containing a crushed ice and rock salt slurry.

A similar process was repeated to obtain MIB or GEO off-flavor catfish. The only change in protocol was after sampling for initial levels of MIB or GEO, tank water was either spiked with MIB (98% purity; 10 mg/mL solution in methanol; Sigma-Aldrich Inc., St. Louis, Miss., U.S.A.) or GEO (98% purity; 2 mg/mL solution in methanol; Sigma-Aldrich Inc.) at a level of 1 ppb.

Catfish filleting

After harvesting, catfish were immediately transported to the Robert M. Kerr Food & Agricultural Products Center (FAPC) for further processing the same day in a 4 °C refrigerated room. Before filleting, individual catfish length, and weights were recorded (Table 1). During fillet fabrication, catfish were eviscerated and gender was determined. Catfish fillet weights were recorded (Table 1) and randomly distributed into 3 replicates. Each replicate contained fillets from 18 fish with a female: male ratio of 1:1.

Table 1: Measurement of length (cm), body weight (kg), and final fillet weight (kg) ± standard deviation of fish before processing stratified by week.

<table>
<thead>
<tr>
<th>Week</th>
<th>Tank treatment</th>
<th>Length (cm)</th>
<th>Body weight (kg)</th>
<th>Final fillet weight (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CON</td>
<td>43.8 ± 2.50</td>
<td>0.84 ± 0.05</td>
<td>0.24 ± 0.05</td>
</tr>
<tr>
<td>2</td>
<td>GSC</td>
<td>46.0 ± 2.50</td>
<td>0.92 ± 0.14</td>
<td>0.28 ± 0.05</td>
</tr>
<tr>
<td>3</td>
<td>MSC</td>
<td>47.0 ± 4.70</td>
<td>0.98 ± 0.15</td>
<td>0.28 ± 0.05</td>
</tr>
</tbody>
</table>

CON, not spiked control catfish; GEO, geosmin-spiked catfish; MIB, 2-methylisoborneol spiked catfish.
**Remediating off-flavor catfish**

(MSC-NP), GEO spiked catfish pH-shift processed (GSC-P), and GEO spiked catfish not pH-shift processed (GSC-NP). The moisture content of samples was adjusted to 80% by adding ice. Next, 1% NaCl and 0.05% NaHCO₃ were added based on the moisture adjusted weight of the protein mixture. The sample was mixed in a food processor (Cuisinart® Pro Classic™, Model 86279, East Windsor, N.J., U.S.A.) at 1000 rpm (medium speed) for 2 min. Samples were formed into 6 g balls and coated with Uncle Bucks Light N Krispy™ fish batter (Bass Pro Shops, Cat. No. 990232, Richardson, Tex., U.S.A.). Batter-coated samples were placed on a cookie sheet and blast frozen at −28 °C for 15 min, placed in a vacuum-sealed bag, labeled, and stored in a freezer at −15 °C, until the day of the sensory panel evaluation.

Batter-coated samples were cooked so an internal temperature of 63 °C (145 °F) was reached before serving to panelists. Briefly, batter-coated samples were placed in deep fried in canola oil for 2 min at 177 °C, until internal temperature reached 49 °C using dual basket Pro Fry™ (Presto, Model 0546607, Eau Claire, Wis., U.S.A.) fryers. Each fryer was assigned to 1 treatment. After frying, oil was allowed to drain from samples for at least 1 min. Then, 2 fried samples from each treatment were placed into labeled serving cups (Dart Container Corporation, Mason, Mich., U.S.A.) and lid-covered. Serving cups were labeled with a randomly generated 3-digit number. To keep samples warm, serving cups containing fried batter-coated samples were placed in a FWE® food warmer (Food Warming Equipment Co. Inc. Model PS-1220–15, Crystal Lake, Ill., U.S.A.) at 76 °C for approximately 30 min or until serving began.

**Sensory evaluation**

Batter-coated samples were subjected to a sensory evaluation by untrained panelists (n = 120) using an incomplete-block design (Cochran and Cox 1957). Each panelist tested and evaluated only 1 replicate sample (1, 2, or 3) from each treatment (n = 6). Samples from each treatment were randomly given to panelists monadically. Thus, each replicate from each treatment was evaluated by 40 panelists. Panelists scored samples for texture, flavor, and overall acceptability using a 9-point hedonic scale ranging from 9 = like extremely to 1 = dislike extremely. Before sensory evaluation, panelists were asked to fill a general questionnaire. The objective of the general questionnaire was to collect panelist demographic information (age, gender, origin, frequency of catfish consumption, and favorite cooking method) and liking preferences in terms of overall impression, flavor, and texture of batter-coated fried catfish samples.

**Compositional analysis**

Triplicate samples from each treatment (n = 6) and each replicate (n = 3) were powdered using liquid nitrogen and a frozen waring blender in a cold room at 4 °C. Then powdered samples were analyzed for moisture (Association of Official Analytical Chemists [AOAC 1997], method number 950.46), fat (AOAC 1997, method number 960.39), protein (AOAC 1997, method number 928.08), and ash (AOAC 1997, method number 920.153).

**GEO and MIB analysis**

Samples from tank water, RET, MSC-NP, MSC-P, GSC-NP, GSC-P, and WW were analyzed for residual GEO and MIB by the USDA Thad Cochran Research Center using solid-phase microextraction and gas chromatography as noted by Schrader and others (2005) and Grimm and others (2004).

Collagen determination

Collagen content was assessed by determining the amount of hydroxyproline in the tissue. Collagen connective tissue on average contains 12.5% (w/w) hydroxyproline when protein content is calculated using a-6.25 protein factor (Kolar 1990). Hydroxyproline was determined as described by AOAC (1997; method number 990.26).

**Statistical analysis**

Data were analyzed using the Mixed procedure of SAS (SAS 2003). Both sensory evaluation and compositional analysis were analyzed as 3 × 2 factorial in a randomized block design where block (panelist number and sample number, respectively) were considered the random variable, factorial A was treatment and factorial B was the type of process. When needed, means were separated using Tukey’s Studentized Range (Tukey’s HSD test) for pairwise comparison among means. Tests were conducted at significant level of α = 0.05.

**Results and Discussion**

**GEO and MIB concentration in tank water**

Tank water samples were obtained before and after treatments were applied (at 0 min, 30 min, and 24 h). Results indicated that levels of GEO were minimal in the tank water of MSC and levels of MIB were minimal in the tank water of GSC (Figure 1). For GSC tanks, results indicated that tank water before spiking (0 min) had insignificant concentrations of GEO. GSC tank water samples collected 30 min after spiking contained 0.5 ppb GEO. After 24 h, the tank water concentration was less than 0.1 ppb demonstrating significant absorption of GEO by catfish in the tanks. For MSC, initial levels of MIB in the tank water were about 0.1 ppb. Tank water samples collected 30 min after spiking contained 0.4 ppb MIB. After 24 h, levels fell to less than 0.2 ppb again suggesting absorption of MIB by the catfish in the tank.

**GEO and MIB concentration in samples**

The concentration of GEO and MIB in samples collected from each replicated treatment (n = 18) was measured. As expected, there was a significant main-effect as a result of off-flavor treatment and process. Figure 2 reports the amount of GEO measured in CON, GSC, and MSC samples. Results indicated there was an insignificant amount of GEO in both CON and MSC. Data are reported on a dry weight basis to normalize against the effect of moisture.

For the measurement of GEO in both WW and RET, there was a main effect of off-flavor treatment. Although it may appear there was a significant amount of GEO in the WW-CON (Figure 2), this is not really the case as the data are reported on a dry weight basis and WW-CON was 1.2% solids (Table 2). For catfish treated with GEO, 16.1 ppb GEO on a dry weight basis was measured in the nonprocessed samples (Figure 2). The insoluble material (RET) removed precipitation and recovery of the myofibrillar protein contained 10.6 ppb GEO on a dry weight basis. The RET represents those diluted proteins that were not solubilized by the low-acid conditions. Theoretically, they should be collagen. However, the variability in hydroxyproline content (Table 3) of RET suggest that myofibrillar proteins likely made-up a significant portion of the RET. The GSC-P sample contained about 10 ppb GEO on a dry weight basis.

The pH-shift process did remove GEO as previously reported (Mireles DeWitt and others 2007a), but not as much as expected.
In the previous study MIB and GEO were reduced >10-fold. However, reductions seen with this study were only approximately 2-fold. A significant difference between the pH-shift process reported in previous studies and this current study is that centrifugation was not utilized to aid in protein separation and recovery. MIB and GEO are thought to be collected in the lipids of catfish (Johnsen and Lloyd 1992). Previous studies (Mireles DeWitt and others 2007a) using centrifugation were able to separate fat much more effectively (75% compared with <28% reduction) than the current study (Table 4). Although there is a trend of lower fat in processed samples, the differences are not significant and suggest that the use of a shaker screen and cheesecloth does little to remove fat which in turn impacted GEO reduction. Figure 2 also reports the amount of MIB measured in CON, GSC, and MSC samples. Observations for MIB are similar to those reported for GEO. Almost 50% of MIB was removed as a result of the acid pH-shift process (from about 22 to 12 ppb, dry weight basis). Although there was a significant main effect of off-flavor treatment on measured MIB in WW, an effect was not seen in RET. The amount of MIB from RET-MSC was slightly lower (7 ppb dry weight basis) than what was reported for RET-GSC. Although the amount of MIB found in WW seems high at 4.2 ppb, as it is reported on a dry weight basis and WW-MSC was about 1.4% solids (Table 2) the actual amount on a wet basis would be about 0.06 ppb.
The absorption targets for GSC and MSC were both 1 ppb. However, results demonstrate that uptake of GEO and MIB by catfish tissue calculated on a wet basis, were higher than targeted (4.04 ± 0.67 ppb and 5.38 ± 0.83 ppb, for GSC and MSC, respectively). These higher uptakes of GEO and MIB could be attributed to a higher water vapor absorption and variations in temperatures (pond, cold and tank, warm). According to Wellborn (1988), appetite and metabolism are increased with increasing water temperatures. This effect can lead to higher absorption of GEO and MIB by catfish. In addition, catfish fat and content fat also can influence absorption of GEO and MIB by catfish. Johnsen and Lloyd (1992) concluded that larger catfish containing greater fat contents absorb and store greater amounts of MIB than leaner catfish. However, our results for fat content were lower than USDA reported values, suggesting that catfish used in this study was lean. Thus, it can be stated that, the higher absorption of GEO and MIB observed in this study was likely a result of change in metabolism of catfish due to environmental temperature changes.

The content of GEO and MIB in catfish was reduced as result of acid pH-shift processing. As stated earlier, the amount removed was less than expected and is likely linked to ineffective lipid removal. Calculated on a wet basis, GSC tissue contained 4.04 ± 0.67 ppb GEO whereas MSC tissue contained 5.38 ± 0.83 ppb MIB, before the pH-shift process. After solubilization of myofibrillar proteins, levels were reduced to 2.72 ± ppb and 2.97 ± 0.86 for GSC and MSC, respectively. This is equivalent to a reduction on a dry basis of 42% and 48%, respectively.

This reduction is similar to those reported by Yamprayoon and Noonhorm (2000) and Forrester and others (2002). Yamprayoon and Noonhorm (2000) reported 35% reduction of GEO for smoked tilapia placed in 7% acetic acid brine solution for 2 wk.

### Table 2-Compositional analysis of retentate and waste water samples obtained from the pH-shift process.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Moisture %</th>
<th>Protein %</th>
</tr>
</thead>
<tbody>
<tr>
<td>RET-CON</td>
<td>93.5 ± 0.66&lt;sup&gt;a&lt;/sup&gt;</td>
<td>3.78 ± 0.40&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>RET-GSC</td>
<td>90.6 ± 0.62&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.00 ± 0.35&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>RET-MSC</td>
<td>89.5 ± 1.19&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.90 ± 0.82&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>WW-CON</td>
<td>97.8 ± 0.13&lt;sup&gt;b&lt;/sup&gt;</td>
<td>51.0 ± 0.11&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>WW-GSC</td>
<td>99.0 ± 0.21&lt;sup&gt;b&lt;/sup&gt;</td>
<td>53.0 ± 0.22&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>WW-MSC</td>
<td>96.0 ± 0.07&lt;sup&gt;b&lt;/sup&gt;</td>
<td>51.0 ± 0.08&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a</sup>Means ± standard deviation appearing in the same column with different superscript are significantly different (P < 0.05).

**Table 3-Hydroxyproline (HP) content of CON, GSC, MSC, RET, and WW.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>% HP, wet weight basis</th>
<th>% HP, dry weight basis</th>
</tr>
</thead>
<tbody>
<tr>
<td>CON-NP</td>
<td>1.40 ± 0.04&lt;sup&gt;a&lt;/sup&gt;</td>
<td>6.86 ± 0.04&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>CON-P</td>
<td>1.36 ± 0.20&lt;sup&gt;b&lt;/sup&gt;</td>
<td>6.57 ± 0.20&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>GSC-NP</td>
<td>1.72 ± 0.23&lt;sup&gt;b&lt;/sup&gt;</td>
<td>6.83 ± 0.23&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>GSC-P</td>
<td>0.87 ± 0.09&lt;sup&gt;c&lt;/sup&gt;</td>
<td>3.25 ± 0.09&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>MSC-NP</td>
<td>2.27 ± 0.10&lt;sup&gt;b&lt;/sup&gt;</td>
<td>9.14 ± 0.10&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>MSC-P</td>
<td>1.35 ± 0.14&lt;sup&gt;b&lt;/sup&gt;</td>
<td>5.12 ± 0.14&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>RET-CON</td>
<td>4.15 ± 0.17&lt;sup&gt;b&lt;/sup&gt;</td>
<td>63.82 ± 0.17&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>RET-GSC</td>
<td>3.22 ± 0.50&lt;sup&gt;a&lt;/sup&gt;</td>
<td>37.40 ± 0.50&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>RET-MSC</td>
<td>3.63 ± 0.49&lt;sup&gt;b&lt;/sup&gt;</td>
<td>34.53 ± 0.49&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>WW-CON</td>
<td>0.58 ± 0.01&lt;sup&gt;b&lt;/sup&gt;</td>
<td>48.29 ± 0.01&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>WW-GSC</td>
<td>0.59 ± 0.07&lt;sup&gt;b&lt;/sup&gt;</td>
<td>58.91 ± 0.07&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>WW-MSC</td>
<td>0.64 ± 0.03&lt;sup&gt;b&lt;/sup&gt;</td>
<td>45.83 ± 0.03&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a,b</sup>Means ± standard deviation appearing in the same column with different superscript are significantly different (P < 0.05).

Protein, fat, and ash are presented on a wet weight basis.

### Table 4-Compositional analysis of catfish fillet tissue stratified by treatments.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Moisture %</th>
<th>Protein %</th>
<th>Fat %</th>
<th>Ash %</th>
</tr>
</thead>
<tbody>
<tr>
<td>CON-NP</td>
<td>79.6 ± 2.30&lt;sup&gt;a&lt;/sup&gt;</td>
<td>18.0 ± 1.14&lt;sup&gt;a&lt;/sup&gt;</td>
<td>3.0 ± 0.21&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1.1 ± 0.09&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>CON-P</td>
<td>79.3 ± 1.98&lt;sup&gt;b&lt;/sup&gt;</td>
<td>18.0 ± 0.97&lt;sup&gt;b&lt;/sup&gt;</td>
<td>2.2 ± 0.43&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.7 ± 0.26&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>GSC-NP</td>
<td>74.7 ± 1.11&lt;sup&gt;b&lt;/sup&gt;</td>
<td>16.7 ± 2.00&lt;sup&gt;b&lt;/sup&gt;</td>
<td>6.6 ± 0.62&lt;sup&gt;b&lt;/sup&gt;</td>
<td>1.0 ± 0.04&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>GSC-P</td>
<td>73.3 ± 1.77&lt;sup&gt;b&lt;/sup&gt;</td>
<td>16.3 ± 3.78&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.9 ± 0.94&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.6 ± 0.03&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>MSC-NP</td>
<td>75.2 ± 0.78&lt;sup&gt;b&lt;/sup&gt;</td>
<td>17.5 ± 0.76&lt;sup&gt;b&lt;/sup&gt;</td>
<td>5.8 ± 0.80&lt;sup&gt;b&lt;/sup&gt;</td>
<td>1.0 ± 0.04&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>MSC-P</td>
<td>73.7 ± 2.99&lt;sup&gt;b&lt;/sup&gt;</td>
<td>19.1 ± 2.62&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.8 ± 0.46&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.6 ± 0.04&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a,b</sup>Means ± standard deviation appearing in the same column with different superscript are significantly different (P < 0.05).

Protein, fat, and ash are presented on a wet weight basis.

Moisture, fat, protein, and ash content were determined for all treatments (Table 4). There was no difference in moisture content. Compositional analysis of catfish fillet samples Forrester and others (2002) reported 36% reduction of MIB in catfish vacuum tumbled in 2% citric acid. The reductions in the current study, however, are much less than those reported by Mireles DeWitt and others (2007b). The former study used a similar process, however, centrifugation was utilized to aid in protein separation and recovery. For centrifugation forces of 3000 × g, about 85% of both GEO and MIB were removed. For centrifugation forces of 10000 × g about 96% of GEO and MIB were removed.

This study used a sieve for separating and collecting proteins. A sieve was chosen because of its low cost and ability to obtain sufficient product for the subsequent sensory evaluation that was to follow. Previous work with a 3-stage pilot scale separator designed for milk separation demonstrated that temperature was difficult control and resulted in too much of the protein being denatured and ejected. Temperature control was not a problem with the shaker sieve as all work was conducted in a 4 °C processing room.

Problems aside from fat separation, however, were encountered with the shaking sieve. First, collection of insolubles (collagen) from solubilized protein proceeded quickly and it was determined the shaking sieve worked quite well for this stage of the process. However, recovery of precipitated protein (the processed sample) was problematic. The protein created a very fragile floc at pH 5.5. The shaking caused a significant amount of floc to pass through the sieve. As a result, cheese cloth was not only used as a final dewatering step, but it was used to recover protein that was lost through the sieve. Three layers of cheesecloth effectively recovered most of this lost protein, however it was a very labor intensive process.

One recommendation that resulted due to the difficulty of recovering flocculated protein is that perhaps proteins should be recovered at pH 5.0, instead of pH 5.5. The isoelectric point of myofibrillar proteins is around 5.0 which means this would be the pH at which a minimum amount of water is held by the protein. There was a stage during precipitation that the proteins appeared to create a very dense floc that looked like islands in pools of water just prior to pH 5.5. It might be that the dense floc should be the point which pH adjustment is stopped. As pH was increased, the floc became lighter and visually less dense. It was difficult to assign a pH when the thick floc was formed because the pH was not very stable. Although difficulties were encountered with the shaker sieve, it is possible that a decanter style centrifuge similar to those used in the surimi industry may be the most effective means in recovering the protein.
between CON and the GSC and MSC samples. There was a main effect from off-flavor treatments ($P < 0.005$), but not pH-shift treatment. There was not an interaction effect. Moisture content was significantly higher for CON than GSC-P and MSC-P ($P < 0.05$). Samples that were P and NP were not significantly different within treatment ($P > 0.05$).

CON catfish moisture content was similar to the value reported by the USDA National Nutrient Database for Standard Reference (2012) for raw farmed channel catfish fillet. Data are in conformity with Seo and others (1995) who reported there is an inverse relationship between moisture content and catfish weight. The higher moisture content CON samples had smaller average body weights (Table 1). The pH-shift process had no effect on moisture content for CON samples ($P > 0.05$). This might be because fat content for CON was significantly lower than GSC and MSC samples ($P < 0.05$). CON catfish were collected 1 and 2 wk before MIB-spiked and GEO-spiked catfish, respectively. There was an effect of pH-shift on fat content, but there was neither an effect of off-flavor treatment nor an interaction effect. Once more, these findings are in agreement with Seo and others (1995) who observed increases in catfish body weight is positively correlated with fat content. Although there was a trend of lower fat in pH-shift processed samples, the difference was not significant from samples that were not pH-shift processed ($P > 0.05$).

For protein content, both main effects and their interaction were nonsignificant ($P > 0.05$). However, high variability in protein content among replicates was observed. These observations are likely result of lack in consistency during the dewatering process. Finally for ash, there was a within treatment difference for samples that were or were not pH-shift processed. There was, however, no off-flavor or pH-shift treatment effect nor interaction effect on ash content ($P > 0.05$). Water soluble ash is likely lost during the pH-shift process.

**Compositional analysis of RET and WW**

The RET and WW from each pH-shift treatment (except non-pH-shift, as that was not a factor for these samples) were analyzed for moisture and protein content. For WW, there was “near significance” ($P = 0.0665$) of off-flavor and for RET there were significant differences ($P = 0.0175$) for moisture. There was no effect of off-flavor on protein content for either WW or RET. For RET, moisture content was significantly higher for CON when compared with GSC and MSC (Table 2). There was no significant treatment effect for protein. On a total solids basis, percent protein was 4.0%, 4.4%, and 5.5% for CON, GSC, and MSC treatments, respectively. For WW samples, no significant difference in moisture and protein content were observed ($P > 0.05$; Table 2). However, variability among replicates was high. On a total solids basis, percent protein was 43%, 53%, and 37% for CON, GSC, and MSC treatments, respectively. Variability in protein content in RET and WW suggests that the dewatering process is a key step in protein recovery.

**Collagen content**

Catfish tissue, RET, and WW from each pH-shift treatment were analyzed for hydroxyproline content (Table 3). Collagen content is calculated by determining the amount of hydroxyproline in the tissue. Analysis compared different sample types (catfish tissue, RET, and WW) and did not evaluate off-flavor or pH-shift as factors. There was a main effect of sample type. On a wet basis, catfish tissue had significantly more hydroxyproline content than RET or WW. On a solid basis, RET samples were significantly higher in hydroxyproline content than other samples analyzed ($P < 0.05$). This can be explained by the solubilization of myofibrillar protein that occurs at pH 2.5 during the pH-shift process. After myofibrillar protein solubilization, the colloidal suspension that results is passed through the sieve. Insoluble proteins such as collagen are retained on the sieve surface. The RET represents those proteins retained on the sieve surface at pH 2.5. Theoretically, the separated insoluble material should be primarily collagen, which is confirmed above. In addition, it was observed that P samples contained lower amounts of hydroxyproline than NP samples. Thus it can be concluded that the pH-shift process using a sieve for separation and recovery effectively reduced collagen.

**Panelist demographics**

Approximately 41% of the panelists were between 18 and 25 y old, 30% were in the 25 to 35 age range, 18% were in 35 to 50 age range, and 11% were 50 y old and up. House and others (2003) reported in their survey on catfish consumer demographics that 53% of the U.S. population is older than 45. Since 71% of the consumers in the current study were less than 35, age was therefore weighted towards a much younger population demographic. The majority of panelists were female (58% female against 42% male). The majority of panelists (67%) identified themselves as being from the United States. About 22% of panelist identified themselves as being from an Asian country, followed by South America at 5% and Europe at 2.5%. Australia, the Mid-East and Africa were designated by only 1 panelist each. Of the panelists that were from the USA, 85% were from the South/Southeast region of the USA. Of those panelists from the South region of the USA, 63% were Oklahoma and 16% where from Texas.

When asked how often they consumed catfish, 41% of panelists reported consuming catfish at least once in a period of 6 mo. About 25% of panelists reported they consumed catfish once a month, 18% once a year, 6% once a week, 5% once every 2 wk, and 5% never. Consumption, on average was therefore fairly infrequent as 59% reported consuming catfish only once or twice a year. Only 36% reported consuming catfish at least 1 or more times per month. Finally, 6 of the 120 panelist reported never having consumed catfish before this trial. These numbers are not too much different than those reported by the Southern Regional Aquaculture Center (SRAC, 1998). They telephoned interviewed 3600 consumers across the nation and found that 43% were considered catfish consumers. They stated a catfish consumer was someone who “reported that they ate catfish to any extent, either at home or away from home.” Catfish consumers identified by this study are ones that eat catfish at least once per month and they represented 36% of respondents.

Panelists reported consuming catfish mostly in restaurants (58%). Other locations reported were at home (38%), other (3%), and never (1%). Fried was the most preferred method of cooking (73%), followed by grilled (16%), baked (6%), and finally other (5%).

**Sensory evaluation**

Panelist where first instructed to evaluate their overall impression of the fried catfish sample. Significant main effects were found for panelist, off-flavor and process (Table 5). There was no off-flavor/pH-shift interaction. For off-flavor CON was preferred, for process P was preferred ($P < 0.05$).

The next attribute rated by panelists was flavor. There was a main effect of panelist and off-flavor treatment, there was not an effect of process. There was no off-flavor/pH-shift interaction.
Panels preferred the flavor of fried catfish samples made from CON rather than GEO-spiked and MIB-spiked protein ($P < 0.05$). There was observed a very slight within treatment trend for preference of samples that were made from processed protein. The preference, however, was not statistical ($P > 0.05$). Based on GC analysis, it is not surprising that panelists preferred the flavor of CON samples. Initial levels of MSC or GSC in the catfish flesh were much higher than targeted (4 to 5 ppb, wet basis). Processing only reduced their levels to slightly less than 3 ppb. Although GEO and MIB levels were reduced as a result of processing, those levels were still above the odor-detection threshold, which according to Grimm and others (2004), lies between 0.1 and 0.2 μg/kg for MIB and 0.25 to 0.5 μg/kg for GEO.

The final attribute evaluated by panelists was texture. There was a main effect of process, but not off-flavor. There was no off-flavor/pH-shift interaction. Panelist clearly preferred the texture of fried catfish samples made from the pH-shift processed catfish ($P < 0.05$). The overall mean for samples made from pH-shift processed catfish was 6.1. This was significantly higher than the rating of 4.6 from those catfish there were not pH-shift processed. Thus, it can be stated that the texture of fried catfish samples was improved by the pH-shift process. Positive adjectives used to describe the samples served to panelist were crunchy, juicy, perfect, great texture, and flavorful. Although the negative included salty, fishy, soil flavor, dirt taste, oily, soapy, and chewy. We hypothesize the texture of the pH-shift treated catfish was preferred because it was firm and juicy like a chicken nugget, a product most panelists would be familiar with.

**Conclusion**

Application of the pH-shift process on catfish fillet tissue effectively reduced amounts of GEO, MIB, moisture, ash, and collagen. Although concentrations of GEO and MIB in fillet tissue were reduced by almost 50%, the reductions observed in this study were not enough to obtain a consumer acceptable catfish product. However, sensory attributes such as texture and overall acceptability were observed to be improved by the pH-shift process. Data suggested that use of a sieve for the pH-shift process may be effective in improving catfish acceptability if GEO and MIB levels are double threshold values or less, however, it was noted use of the sieve for protein recovery still had its challenges. Future commercial applicability of the pH-shift process is dependent on developing a process that efficiently and economically recovers the protein.

**References**


