Pretreatment of Wheat Bran for Suitable Reinforcement in Biocomposites

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ABSTRACT: Wheat bran, abundant but underutilized, was investigated for its potential as a reinforcement in biocomposites through different pretreatment methods. Pretreatment methods included were dilute sodium hydroxide (NaOH), dilute sulfuric acid (H2SO4), liquid hot water (LHW), calcium hydroxide (CaOH), organosolv such as aqueous ethanol (EtOH), and methyl isobutyl ketone (MIBK). Changes in chemical composition and fiber characteristics of the treated bran were studied using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). Cellulose content increased to 35.1% and 29.6% in brans treated with H2SO4 and NaOH, respectively. The SEM micrographs showed surface cleaning of treated bran while maintaining sufficient surface roughness for the H2SO4, NaOH, and MIBK treated brans. Crystallinity index increased slightly for all treatments except H2SO4, NaOH and H2SO4 pretreated brans achieved important fiber characteristics, which could be useful for making thermoplastic biocomposites. Innovative use of bran in thermoplastic will create more opportunities for growers while enhancing biodegradability.

KEYWORDS: Biocomposites, fiber composition, pretreatment, surface treatment, wheat bran

1 INTRODUCTION

The use of petroleum-based plastics is thought to be the cause of widespread environmental issues because they do not decompose readily after their disposal. The use of biobased natural fibers as fillers or reinforcement agents in manufacturing biocomposites has received much attention recently. Several factors, such as their ability to degrade quickly, cheaper cost, light weight, high specific strength, and renewability, are favorable to applications for biocomposites [1–3]. The high relative advantages and diversified applications are reflected by the growth rate of biocomposite development. From 2003 to 2007, the average annual global growth rate of biocomposites was 38%. Globally, the volume of biobased plastics is likely to increase from 0.36 million metric tons in 2007 to 2.33 million metric tons by 2013 and to 3.45 million metric tons in 2020 [4]. However, biobased composites are still in their developmental stage, and, in combination with commodity synthetic polymers, they are an option for obtaining overall cost and more environmentally favorable processing [1].

A large number of fiber sources were investigated, such as wood, hemp, feather, kraft pulp, and pineapple [5–11]. In addition, there is a continued search for new fiber sources for biocomposites. There are large amounts of grain by-products, such as straw, wheat bran, rice husk, and corn stalk, which can be used for producing biodegradable composites [12]. Biocomposites prepared from agricultural waste and macromolecular materials are more beneficial compared to other fiber materials due to their water absorption characteristics, workability, and superior mechanical properties [13]. For example, wheat kernel contains about 14.5% of bran, which is produced in huge amounts as a by-product every year from the milling of wheat [14]. Only 10% of this by-product is used in bakeries and in breakfast cereals as a dietary fiber supplement. The 90% of the remaining bran could be sold as animal feed, but due to high transportation costs, millers often dispose of the bran as waste, which causes environmental hazards.

Wheat bran contains phenolic compounds [15], starches [16], soluble and insoluble dietary fibers [17],...
2 EXPERIMENTAL

2.1 Milling and Bran Extraction

The bran used in this study was collected from hard red spring wheat. The wheat sample was milled using a Buhler MLU-202 laboratory mill. A sample of wheat was prepared for milling with a Carter-Day dockage tester (Minneapolis, MN, USA) with a number 8 sieve. The sample was then tempered in three stages: 1) pre-tempered to 12.5% moisture content (MC) for 72 h before milling if MC was below 11%; 2) tempered to 16% MC for 24 h before milling; and 3) finally tempered to 16.5% MC for 20 to 30 min before milling. The Buhler MLU-202 produced six flour products, one bran product, and one shorts product. The bran fraction was collected and used for experiment in this study.

2.2 Pretreatment Procedures

2.2.1 NaOH Pretreatment

Milled wheat bran was loaded into a conical flask at 10 wt% solids in deionized water. The sodium hydroxide loading was 100 mg/g of dry bran. The flask was heated in a water bath at 80 °C for 3.5 h with occasional low speed shaking. After heating, the resulting slurry was removed from the flask and separated into solid and black liquors. To separate the black liquor, the slurry was first centrifuged for 10 min at 4000 rpm and the resulting caustic black liquor supernatant was decanted from the tube and discarded. The solids were washed three times through resuspension in 1 L of deionized water. The wash water was decanted from the solid fraction. Finally, the solid was vacuum
filtered on a 2 μm pore size PTFE filter to remove the small remaining amount of wash water. The washed solid was dried at 60 °C and weighed periodically until a constant weight between two consecutive measurements was achieved. The dried bran sample was used for composition analysis, SEM imaging, and IR experiments.

### 2.2.2 Organosolv Pretreatment (EtOH)
Ethanol (90%) was mixed with bran at a ratio of 6:1 in a centrifuge tube. The centrifuge tube was placed in a beaker and heated for 4 h at 95 °C with periodic agitation. The samples were cooled to room temperature, and the pulp and liquor were separated by centrifuging for 10 min at 4000 rpm. The resulting black liquor was decanted. The pulp was resuspended and washed three times in 300 mL of aqueous ethanol with the same concentration as cooking liquor. The wash water was discarded, and the remaining solid fraction was vacuum filtered to remove remaining liquid. The washed solid was dried at 60 °C and weighed periodically until a constant weight between two consecutive measurements was achieved.

### 2.2.3 Liquid Hot Water (LHW) Pretreatment
Bran biomass was immersed in liquid water at 9 wt% solid loading. The LHW was carried out at 140 °C and 33 psi with a 1-h contact time in an autoclave. The treated sample was then centrifuged for 10 min at 4000 rpm. The resulting liquor from centrifugation was decanted from the top of the tube. The remaining solid was resuspended in 250 mL of deionized water for washing. Wash water was discarded, and solids with remaining water were vacuum filtered to remove excess water. After filtration, the solids were dried at 60 °C to a constant weight.

### 2.2.4 Lime (CaOH) Pretreatment
Lime (calcium hydroxide) was used as a pretreatment agent to dissolve lignin from wheat bran. Wheat bran was treated with lime at the ratio of 1 g of lime to 1 g of bran, and with water at a ratio of 7 mL of water to 1 g of bran. The bran was thoroughly mixed with the water and lime, and the mixture was heated for 2.5 h at 100 °C. After heating, the samples were centrifuged for 10 min at 4000 rpm. The liquor from the top of the centrifuge tube was decanted, and the solids were resuspended three times in 250 mL of deionized water for washing. Wash water was discarded, and the solids with remaining water were vacuum filtered to remove the excess water. After filtration, solids were dried at 60 °C to a constant weight.

### 2.2.5 H₂SO₄ Pretreatment
Wheat bran was treated using diluted sulfuric acid at a concentration of 4% at 100 °C. The experiment was performed at a liquor/solid ratio of 10 g liquor to 1 g wheat bran (dry basis). The mixture of bran and acid was heated for 2.5 h. After heating, the mixture was taken from the reaction media and centrifuged for 10 min at 4000 rpm. The liquor from the top of the centrifuge tube was decanted, and the remaining solids were washed three times in 500 mL of deionized water and then vacuum filtered to separate the solids. After filtration, solids were dried at 60 °C to a constant weight.

### 2.2.6 Methyle Isobutyle Ketone (MIBK) Pretreatment
Organic solvents dissolve lignin, which may facilitate separation of lignocellulosic materials into their components. A modified method of Black et al. [30] was used in this study to treat bran. A single phase pulping liquor composed of 24% water, 44% methyl isobutyl ketone (MIBK), and 32% ethanol was prepared in a glass container. Bran was mixed with liquor at the ratio of 10 mL of liquor to 1 g of bran, and 2 mL of 0.05 M H₂SO₄ catalyst was added. The sample mixture was heated at 100 °C for 2 h. The resulting pulp was washed with fresh neutral liquor, vacuum filtered, and dried at 60 °C to a constant weight.

### 2.3 Composition Analysis
The recovered solids were dried for several days in a vacuum oven at 60 °C until a constant value of the mass was obtained from two consecutive weighings. Analysis of the solids for composition was subsequently conducted. A total of seven components, including lignocellulosic fractions, were analyzed in triplicate. The analyses were performed at the Animal Sciences Department of North Dakota State University. The parameters analyzed included crude protein, neutral detergent fibers, acid detergent fibers, acid detergent lignin, fat, starch, and dry matter content. Dry matter was determined according to AOAC Method 967.03 [31], with few modifications. The samples were weighed at room temperature and then heated at 100 °C for 24 h. After heating, samples were conditioned in desiccators and weighed again. The percentages of neutral detergent fiber (NDF), acid detergent fiber (ADF), and acid detergent lignin (ADL) were determined using an ANKOM-200/220 fiber analyzer (ANKOM Technology, Macedon, NY, USA), according to methods specified in the USDA Agricultural Handbook [32]. The percentage of starch was determined using an acid and enzymatic isolation technique.
assay and microtiter reading with a SPECTRAMax®
340 microplate reader (Molecular Devices, Sunnyvale,
USA). The cellulose and hemicellulose percentages
were calculated using Equations 1 and 2.

\[
\text{% Cellulose} = \text{% Acid Detergent Fiber} - \text{% Acid Detergent Lignin} \quad (1)
\]

\[
\text{% Hemicellulose} = \text{% Neutral Detergent Fiber} - \text{% Acid Detergent Fiber} \quad (2)
\]

### 2.4 Scanning Electron Microscopy (SEM)

The morphology of treated and untreated brans was
analyzed by SEM at the Electron Microscopy Center at
North Dakota State University. Samples were attached
to cylindrical aluminum mounts using double-stick
carbon adhesive tabs (Ted Pella, Redding, CA, USA)
and then sputter coated (Cressington 108 Auto, Ted
Pella) with a conductive layer of gold. Images were
obtained with a JEOL JSM-6490LV scanning electron
microscope (JEOL USA, Inc., Peabody, MA, USA) at an
accelerating voltage of 15 kV.

### 2.5 Fourier-Transform Infrared
Spectroscopy (FTIR)

FTIR was performed at the Materials Characterization
and Analysis Laboratory at North Dakota State
University and was used to observe the compositional
change in the bran before and after treatment with dif-
ferent thermophysical and chemical methods. Bran
specimens were prepared by mixing a small amount of
bran with potassium bromide (KBr), followed by cold
pressing to form discs. Infrared absorbance spectra of
the bran specimens were recorded at ambient tempera-
ture and atmospheric pressure with a Nicolet 8700 FT-IR
spectrometer (Thermo Electron Scientific Instruments
LLC, Madison, WI, USA). The spectra were obtained
by recording 32 scans, which were performed with a
resolution of 4 cm\(^{-1}\) between 400 and 4000 cm\(^{-1}\). The
peak signals were recognized using software (OMNIC,
Thermo Electron Scientific Instruments LLC, Madison,
WI, USA). The crystallinity indices of the untreated and
treated brans were calculated by infrared ratio, \(I_{1427}/I_{2920}\ cm^{-1}\), as suggested by [33], which applies to
both cellulose I and II and mixed lattices.

### 2.6 Data Analysis

Analysis of variance (ANOVA) of the different treat-
ments was performed using GLM procedure of the
Statistical Analysis System (version 8.0, SAS Institute
Inc., Cary, NC, USA).

### 3 RESULTS AND DISCUSSION

The wheat bran was treated by several different meth-
ods. A number of treatment levels were investigated
for each method. The best results from each method
have been reported and discussed regarding their
effectiveness for producing suitable fillers for thero-
plastic biocomposites. Based on the treatment results,
pretreatment methods have been suggested for bran
because they might render superior biocomposite
characteristics for large-scale uses.

#### 3.1 Change in Compositions

The cellulose fraction in bran increased after treatment
with different methods, as shown in Figure 1. A sig-
nificant increase in the cellulose content was observed
for the NaOH and H\(_2\)SO\(_4\) treated brans. The highest
increase of 35.0% was observed in the H\(_2\)SO\(_4\) treat-
tment, followed by 29.6% in the NaOH treatment, com-
pared with untreated bran, which contained 9.75%
cellulose. Although CaOH, MIBK, LHW, and EtOH
treatments increased the cellulose fraction, their cellu-
lose contents were not significantly different than that
of the untreated bran. Chauvelon \textit{et al}.
[25] observed
a similar amount of cellulose enrichment in bran with
H\(_2\)SO\(_4\) and KOH treatments, which were 38.3% and
31.7%, respectively. In another study, NaOH treated
wheat straw under moderate temperature and pres-
sure increased the cellulose fraction up to 63.1% [34].

The H\(_2\)SO\(_4\) treatment was effective in solubilizing
hemicellulose and thereby reduced the fraction from
bran, as shown in Figure 2. In H\(_2\)SO\(_4\) treated bran, a
five-fold decrease in hemicellulose was observed.

The hemicellulose content decreased from 32.0%
in the untreated bran to 6.83% in the H\(_2\)SO\(_4\) treated
bran. Compared with untreated bran, hemicellulose

![Figure 1 Cellulose content (%) in untreated and treated wheat brans. The error bar (N = 3) indicates standard deviation. Columns with different letters in parentheses are significantly different at the 95% confidence level.](image-url)
percentage increased with all other treatments except the CaOH treated bran, which had 24.9% hemicellulose and was higher than that of the H2SO4 treated bran. However, among others which increased the hemicellulose content, NaOH treated bran had the highest content at 46.8% and was not significantly different from the LHW and EtOH treated brans. Lamsal et al. [35] found similar hemicellulose removal (~5%) from destarched bran with the same acid concentration but with a higher temperature and lower retention time.

Acid detergent lignin or lignin content is usually lower in wheat bran [12], but none of the pretreatment methods used in the present study reduced the lignin content (Figure 3). Several studies have suggested that lignin removal from lignocellulosic materials depends on the part of plant materials targeted for lignin removal. Chemical pulping of wheat straw with NaOH removed 70% lignin [34]. However, a slight increase in lignin content was observed in wheat bran treated with diluted H2SO4 [35], which supports the results of the present study.

The effectiveness of starch removal by various treatments used in the present study is presented in Figure 4. In all treatments, a significant removal of starch was observed. Almost all starch (0.68% in treated bran) was removed by H2SO4 treatment, followed by NaOH (1.8% in treated bran) and LHW (8.09% in treated bran) compared with untreated bran (15.5%). Generally, biomass is treated with α-amylase to remove starch. Lamsal et al. [35] observed starch removal in wheat bran from 20% to 9% by α-amylase treatment. However, the present study effectively removed starch from bran using NaOH and H2SO4 treatments.

The crude protein fraction was significantly reduced by the NaOH, CaOH, and EtOH treatments, as shown in Figure 5. The highest removal was observed by

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**Figure 2** Hemicellulose content (%) in untreated and treated wheat brans. The error bar (N = 3) indicates standard deviation. Columns with different letters in parentheses are significantly different at the 95% confidence level.

**Figure 3** Acid detergent lignin content (%) in untreated and treated wheat brans. The error bar (N = 3) indicates standard deviation. Columns with different letters in parentheses are significantly different at the 95% confidence level.

**Figure 4** Starch content (%) in untreated and treated wheat brans. The error bar (N = 3) indicates standard deviation. Columns with different letters in parentheses are significantly different at the 95% confidence level.

**Figure 5** Crude protein content (%) in untreated and treated wheat brans. Error bar (N = 3) indicates standard deviation. Columns with different letters in parentheses of abscissa are significantly different at 95% confidence level.
NaOH (4.67% in treated bran), followed by CaOH (11.8%) and EtOH (18.5%) compared with untreated bran (22.3%). However, LHW treatment increased the crude protein fraction, but the increase was not significantly different than that in untreated bran. Chauvelon et al. [25] treated wheat bran with alkali (KOH) and observed a decrease in protein fraction from 10.8% to 0.4% and 0.2% using hydrogen peroxide and sodium hypochlorite, respectively, which supports protein removal by alkali in the present study. A 27.9% crude protein removal by lime treatment was also observed in switchgrass [36].

Figure 6 shows the effect of pretreatment by different methods on the change in fat content in wheat bran. The fat was significantly removed by all treatments except H2SO4. The decreased fat contents ranged from 0.22% by MIBK to 1.47% by EtOH in treated bran compared with untreated bran (3.8%). However, compared with untreated bran, fat content increased more than two-fold (8.05%) with H2SO4 treatment. In a different study, about a three-fold reduction of fat, from 0.60% to 0.20%, was observed in spelt treated with enzymes [37].

Pretreatment of wheat bran by different methods significantly increased the dry matter fraction, as shown in Figure 7. The average dry matter content in the untreated bran was 89.2%, while the dry matter content in the treated bran ranged from 91.6% (CaOH) to 95.2% (H2SO4). Increase of dry matter fraction by MIBK, LHW, and H2SO4 was significantly different. Moisture uptake in biomass occurs mainly in hemicellulose, non-crystalline cellulose, accessible cellulose, starch, lignin, and the surface of cellulose [38]. Although moisture uptake by bran was not investigated, it is likely that pretreatment changed the chemical composition, texture, and structure, which influenced the water retention properties of the treated bran under ambient storage conditions.

3.2 Scanning Electron Microscopy (SEM)

Figure 8 shows the SEM micrographs of untreated and treated brans with different thermophysical and chemical methods. The surface morphology of untreated bran in Figure 8a shows the presence of protein, starch, fat, and globular particles. The presence of a smooth waxy surface over fibers called cuticle, which was identified as aliphatic wax [39], is more visible in the magnified view shown in Figure 8b. The fiber surface containing cellular materials, as shown in Figure 8b, was modified and became relatively cleaner through the treatments.

Sulfuric acid (H2SO4) treatment removed fat, starch, some hemicellulose, and waxy cuticles and exposed the fiber surfaces, as shown in Figure 8c. Although there was evidence of defibrillation, node-like cell materials held together adjacent fibers. Despite the cleaning of surfaces due to treatment, the microstructure of the surface shows roughness, which is advantageous for making biocomposites. Calcium hydroxide (CaOH) treatment did not solubilize cuticle layers, which were clearly visible in treated bran (Figure 8d). The observed terraces and pits in the treated bran were likely due to the removal of fat and globular materials. Similar to CaOH, LHW treatment also caused pits, but they were larger in size and number than those of CaOH treatment (Figure 8f). At a higher magnification (inset micrograph), the deposition of pseudo-lignin and/or protein on the surface of the holocellulose was observed [40]. Methyl isobutyl ketone (MIBK) treatment of bran removed most of the fat particles and resulted in a smoother surface (Figure 8e). At a higher magnification, the micrograph showed
solid-like fibers, which were superimposed, one over another, and appeared as a sandwich-like structure, as evidenced from the inset micrograph. Treatment with EtOH did not remarkably change the surface morphology from that of untreated bran, which is evidenced from the presence of cellular materials, including starch and protein (Figure 8g). The smooth waxy surface that resulted from EtOH treated bran might

Figure 8 SEM micrographs of wheat bran. Untreated (a) and (b), and treated with (c) H$_2$SO$_4$, (d) CaOH, (e) Methyl isobutyl ketone (MIBK), (f) Liquid hot water (LHW), (g) Ethyl alcohol (EtOH), and (h) NaOH.
not help with increasing fiber-matrix adhesion, which is essential for improving the mechanical strength of biocomposites. Alkali (NaOH) treatment removed all fat, starch, and protein particles from the bran, and the layer of cuticles was dissolved, resulting in a smoother and cleaner surface (Figure 8h). However, a closer look at the magnified micrograph revealed rough microstructures and some pits (inset micrograph). A rough surface could be advantageous for manufacturing biocomposites. Similar to H$_2$SO$_4$, NaOH treatment also shows node-like structures (inset micrograph), which bonds adjacent fibers together.

### 3.3 Structural Characteristics of Untreated and Treated Brans by FTIR

Figure 9 shows the spectra of untreated and treated brans with different thermophysical and chemical methods, in which the majority of peaks are labeled with the wave number in it. The functional groups of the characteristic peaks from the spectra are identified and presented in Table 1 [41–43]. The absorption peaks near 3402 to 3423 cm$^{-1}$ bands observed in different treatments were attributed to the stretching vibration of hydroxyl groups. The OH group may include absorbed water, aliphatic primary and secondary alcohols found in cellulose, hemicellulose, carboxylic acids, and phenolic compounds [44]. Intense bands of spectra observed in 2923 to 2926 and 2855 cm$^{-1}$ were attributed to the C-H stretching vibration of methyl, methylene, and methane groups, which are the moieties in polysaccharides (cellulose and survived hemicelluloses) [44–46]. The band near 1736 cm$^{-1}$ appeared only in NaOH, H$_2$SO$_4$, and MIBK treated brans, suggesting the presence of carbonyl and unconjugated ketone and carboxyl group stretching. The absorption bands ranging from 1660 to 1630 cm$^{-1}$ are attributed to conjugated carbonyl stretching [45]. The absorption band near 1539 cm$^{-1}$ in untreated bran shifted to 1518, 1530, and 1519 cm$^{-1}$ in LHW, H$_2$SO$_4$, and MIBK treated brans, respectively, but disappeared in NaOH and CaOH treated brans. The N-H vibration of amine indicated the presence of an amine group in protein, and the disappearance of this band in NaOH and CaOH treated brans could be indicative of the removal of protein. The range of absorption peaks from 1027 to 1053 cm$^{-1}$ is attributed to the lignin component, guaiacyl unit, and an aromatic C-H plane deformation [44, 45]. The appearance of absorption peaks from 1162 to 1170 cm$^{-1}$ and near 898 cm$^{-1}$ for NaOH, H$_2$SO$_4$, and MIBK treated brans are seen in the spectra, which are typical of pure cellulose [46].

### 3.4 Crystallinity Index (CI)

Table 2 shows the crystallinity index of untreated and treated brans with different thermophysical and chemical methods. A CI value of 0.94 was obtained for the untreated bran sample. An increase in CI was observed for CaOH, EtOH, MIBK, and NaOH treated brans, and a decrease was observed for H$_2$SO$_4$ and LHW treatments. The highest increased CI was 0.98 for the NaOH and CaOH treated samples, and
Table 1: Characteristic functional groups of untreated and treated wheat bran with different methods in FTIR spectra.

<table>
<thead>
<tr>
<th></th>
<th>Untreated</th>
<th>EtOH</th>
<th>NaOH</th>
<th>LHW</th>
<th>CaOH</th>
<th>H₂SO₄</th>
<th>MIBK</th>
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<tbody>
<tr>
<td>Wave number (cm⁻¹)</td>
<td>Characteristic group</td>
<td>Wave number (cm⁻¹)</td>
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<tr>
<td>2925</td>
<td>C–H</td>
<td>2923</td>
<td>C–H</td>
<td>3416</td>
<td>O–H</td>
<td>2924</td>
<td>C–H</td>
</tr>
<tr>
<td>1655</td>
<td>C=O</td>
<td>1654</td>
<td>C=O</td>
<td>2924</td>
<td>C–H</td>
<td>1655</td>
<td>C=O</td>
</tr>
<tr>
<td>1540</td>
<td>N–H</td>
<td>1539</td>
<td>N–H</td>
<td>1736</td>
<td>C=O</td>
<td>1518</td>
<td>N–H</td>
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<tr>
<td>1411</td>
<td>CH₃</td>
<td>1414</td>
<td>CH₃</td>
<td>1641</td>
<td>C=O</td>
<td>1453</td>
<td>CH₃</td>
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<tr>
<td>1047</td>
<td>C–H</td>
<td>1045</td>
<td>C–H</td>
<td>1425</td>
<td>CH₃</td>
<td>1036</td>
<td>C–H</td>
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<tr>
<td></td>
<td></td>
<td>1162</td>
<td>C–O</td>
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<td>CH₃</td>
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<td></td>
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<td>898</td>
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<td>898</td>
<td>C–H</td>
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the lowest decreased CI was 0.79 in the H$_2$SO$_4$ treated sample. The change in CI observed was relatively low (~5%) in this study, but could be advantageous for biocomposite manufacturing. Mwaikambo and Ansell [47] observed superior mechanical strength with low crystalline fibers compared to fibers with high crystallinity. The higher crystallinity may be obtained by the destruction of the primary cell wall, which may result in decreasing the mechanical properties.

### 3.5 Discussion

The performance of polymer composites reinforced with natural fibers depends on several factors such as fibers’ chemical compositions, cell dimensions, microfibrillar angle, defects, structures, physical properties, chemical properties, and the interaction between the fiber and polymer matrix [3]. Pretreatment of fibers could modify many of the fiber properties, which may result in improving the performance of the resulting biocomposites from the pretreated fibers. A number of pretreatment methods have been proposed for fibers based on the differences in fiber properties and chemical compositions. The chemical composition varies from species to species as well as within the same species due to the differences in climate and environment. It also varies within different parts of the same plant because of the physiological functions. In this study, a broad range of treatment methods have been used to pretreat wheat bran for making a suitable reinforcing material for biocomposites.

The chemical composition of fibers is the most important characteristic of any lignocellulosic material. While cellulose fraction increases the mechanical strength of biocomposites, amorphous hemicelluloses, lignin, protein, and fat have little or no contribution to the resulting biocomposite strength. Sodium hydroxide (NaOH) and H$_2$SO$_4$ treated brans showed higher fractions of cellulose content and lower fractions of hemicelluloses, fat, protein, and starch compared to all other treatments. Pretreatment of bran by these two methods could be considered as candidates in preparing wheat bran biocomposites. Sufficient surface roughness was observed in the SEM micrographs of the bran treated with MIBK, H$_2$SO$_4$, and NaOH, despite the cleaning of cellular materials from the bran surfaces. Alkali treatment disrupted the hydrogen bonding in the network structure, which increased the surface roughness [3]. Alkali treatment increases the interfacial adhesion, which may increase compressive and tensile strength and lead to better moisture resistance [48,49]. Alkali treatment also repairs the defects in fibers, thus increasing the fracture strain [49]. Crystallinity increased in NaOH treated fiber, but decreased in H$_2$SO$_4$ treated bran. Crystallinity has the effects of increasing mechanical strength and decreasing moisture absorption. Alkali treatment solubilizes some amorphous materials and allows the repacking of celluloses, which increases the crystallinity of bran. It also decreases the spiral angle and increases molecular orientation [50]. For better water resistance in H$_2$SO$_4$ treated bran, surface modification, such as grafting, may be used.

Wheat is one of the most important crop plants in the world. The utilization of wheat bran as biocomposites in plastic would make it more environmentally friendly and would find an alternative use for wheat bran in industrial products. This study would also encourage researchers to find other usages for wheat bran, such as isolation and characterization of nanoparticles, which could be used for drug and micronutrient delivery in animals and crop plants, respectively.

### 4 CONCLUSIONS

1. Cellulose content increased to 35.1% and 29.6% in treated bran with H$_2$SO$_4$ and NaOH, respectively. Hemicelluloses content decreased to 6.83% and 24.9% for H$_2$SO$_4$ and CaOH treated brans, respectively. No improvement in lignin fraction decrease was observed by any treatment.

2. Starch content decreased to 0.68% and 1.86% by H$_2$SO$_4$ and NaOH treated brans, respectively. Fat content was removed effectively by all the pretreatment methods except H$_2$SO$_4$. NaOH treatment removed the crude protein effectively, and the crude protein fraction decreased to 4.67%.

3. SEM micrograph showed surface cleaning of treated bran while maintaining sufficient surface roughness for H$_2$SO$_4$, NaOH, and MIBK treated brans.

4. Presence of a pure cellulosic functional group was observed in the spectra of brans treated with H$_2$SO$_4$, NaOH, and MIBK.

5. Crystallinity index increased, though slightly, for all treatments except H$_2$SO$_4$ treated bran.

6. A considerable amount of hemicellulose was not removed, and future studies should address this limitation to enhance the removal of more hemicelluloses.

In ongoing studies, the preparation of bran treated with NaOH is being pursued because of the reduction...
in crude protein and fat content compared to H$_2$SO$_4$ treated bran even though H$_2$SO$_4$ treated bran has slightly higher cellulose content, which can be useful in plastic production as a reinforcing material. After analyzing the physico-mechanical properties of treated wheat bran biocomposites, the use of wheat bran as an effective filler will be proven.

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