

Physical, Chemical, and Rheological Properties of Rice Husks Treated by Composting Process

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A composting treatment was employed in an effort to improve the processability of rice husks. Changes in the chemical composition, physical structure, and rheological properties of modified rice husks were analyzed. The results indicated that the average diameter of compost-treated samples was significantly higher than that of the untreated samples because they were able to adhere to each other by the bacterial protein. Scanning electron microscopy images showed that the epidermis became rugged and lumpy because the composition of rice husks (cellulose, hemicellulose, lignin, and pectin) was partially decomposed, an effect confirmed by the chemical composition and FTIR analysis. Thermogravimetric analysis showed that the composted samples had better thermal stability than the untreated ones. Stress-strain curves showed that the treated samples displayed a moderately significant change of slope at about 0 to 10% strain, and they had better mechanical properties than untreated samples. Juxtaposing the rheological properties of both untreated and treated samples determined that the latter had higher apparent viscosity as a result of degradation and bacterial protein effects. All results indicated that the composting treatment changed the physical, chemical, and rheological properties of the rice husks, which are beneficial for its utilization and processability.

Keywords: Rice husks; Compost; Degradation; Processability; Structure

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INTRODUCTION

In recent years, agricultural lignocellulosic fibers have shown the potential to play an important role in the global energy future because they can be grown in a sustainable manner and converted into a variety of products (Wu *et al.* 2013). There has been extensive research on the use of agricultural lignocellulosic fibers, such as straw and rice husks, in the manufacturing of structural and semi-structural fiber-reinforced thermoplastics (Sain and Panthapulakkal 2006). Industrial use of these natural, fiber-reinforced composites is increasing due to their relatively low cost, abundance, and renewability compared to conventional materials. Also, natural fiber-reinforced plastics are of interest as a replacement for synthetic fiber-reinforced plastics in an increasing number of industrial sectors including the automotive, packaging, and furniture production industries (Alemdar and Sain 2008).

However, it is known that processed lignocellulosic fibers can be difficult to use in certain energy-related applications because of their molecular weight and hydrogen bonding. Cellulose, the main component of lignocellulose fibers, has a crystalline structure either with a triclinic or a monoclinic unit cell and has a structure of considerable tensile

strength (Liu *et al.* 2005). However, the crystalline character acts as a barrier to easily using lignocellulose in some applications, such as saccharification, injection molding, compression molding, and so on. And changing the structure of lignocellulose can improve the processability of lignocellulosic fibers.

Various methods such as mechanical (Alang *et al.* 2011), physical (Imanah and Okieimen 2004; Iyasele and Okieimen 2004), chemical (Nigam *et al.* 2009; Cheng *et al.* 2010), and steam explosion (Kristensen *et al.* 2008) have been discussed to improve the processability of lignocellulosic fibers. Physical treatments include soaking/wetting, chopping, grinding, pelleting, steaming under pressure, and gamma radiation. Alkali, acid, and oxidative reagents are used in chemical treatments. Most pretreatments, except biological, require expensive instruments or equipment that can have high energy requirements.

Currently, biological treatment is receiving increased attention as a way to improve the processability of lignocellulose fibers. Biological treatments include composting and fermentation, as well as fungal and enzyme degradation (Koike *et al.* 2014). Fibers prepared after the retting process exhibited better mechanical properties than the un-retted fibers because they had a more lignin-like fiber surface (Sain and Panthapulakkal 2006). Also, the composting treatment is one of the widely used techniques to improve cellulosic fibers from agricultural plants such as hemp, flax, and jute.

Composting is a natural process capable of stabilizing organic wastes. The stabilization process considerably reduces odor emissions and dries up the waste, making it easier to handle and transport (Petric and Selimbašić 2008). It has been reported that microbial inoculation can have a significant, positive effect on composting processes. Many workers have demonstrated that selected lignocellulolytic fungi are capable of degrading wheat straw (Dorado *et al.* 1999; Romero *et al.* 1999). Biological treatment using white rot fungi, a safe and environmentally-friendly method, is increasingly being advocated as a process that does not require high energy for lignin removal from a lignocellulosic biomass in spite of the long degradation time (Okano *et al.* 2005). Lignin degradation by white rot fungi occurs through the action of lignin-degrading enzymes such as peroxidases and laccase (Gupta *et al.* 2012). Pretreatments with white-rot fungi, which have been shown to completely degrade lignocellulose, increase glucose yields without significant capital or energy intensive steps (Hatakka 1983). The same pattern was observed for the degradation of cellulose, though degradation was shown to have begun later in the course of treatment. Degradation of hemicellulose was nearly 100%, whereas the cellulose degradation was 64 and 70% in the box and reactor, respectively. Lignin was not degraded during the experiments (Eiland *et al.* 2001).

In this research, we explored the use of inoculants in the composting of rice husks to improve its processability, which changed its physical, chemical, and rheological properties. Untreated samples, composted samples with inoculants, and composted samples without inoculants were analyzed in this study.

EXPERIMENTAL

Materials

The main materials employed for composting were fresh rice husks (< 2 mm long), which were obtained from local farmers. Characteristics of the rice husks were analyzed in

the laboratory, and the results were as follows: 9.5% moisture content, 48.1% total carbon, and 0.78% total nitrogen.

The inoculant was mature rice husks after composting, which was purchased from Nanjing Ningliang Biotechnology Co. Ltd. (China). It contained 20% moisture content, 38.3% total carbon, and 11.5% total nitrogen.

Methods

Composting process

Two kinds of composting treatments were applied in this study as shown in Table 1. One kind was rice husks composted without inoculant, and the other rice husks composted with inoculant. The rice husks were composted in a pilot plant for each treatment. The system maintains a temperature ceiling in the pile through the on-demand removal of heat by stirring. This encourages a high decomposition rate by avoiding high temperatures, which would inhibit and slow down decomposition by reducing microbial activity. The rice husks were stirred every five days in order to maintain enough air in the rice husk heap. The initial carbon nitrogen ratio (C/N) was adjusted to 30:1 by the addition of urea (Jiang *et al.* 2011). The duration of composting was 60 days. The temperature variation of rice husks with different treatment is shown in Fig. 1.

Table 1. Components of the Two Kinds of Composting Treatments

Treatment	Composting with inoculants	Composting without inoculants
Weight of rice husks (kg)	1000	1000
Initial carbon nitrogen ratio	30:1	30:1
Initial moisture content (%)	65	65
Ratio of inoculants (%)	5	0
Finale moisture content (%)	35.7	42.9
Finale pH values	7.9	7.5
Finale total -nitrogen (%)	1.5	1.3
Finale total Organic Carbon (%)	36.0	39.0

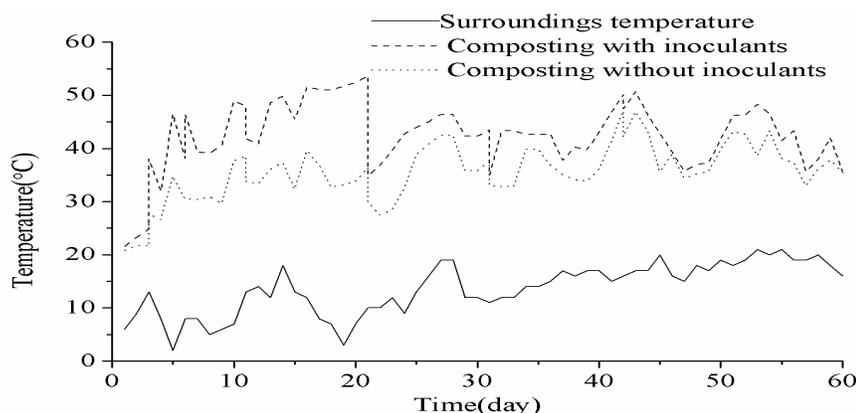


Fig. 1. Changes of temperature during composting treatment for the different treatment

Characterization

The diameter distribution of the rice husks was measured using a fractionating classifier (AS 400, Retsch; Germany). Both treated and untreated rice husks (1000 g) were dried in a drying oven at 105 °C and then measured by the fractionating classifier. The rice

husks were weighted with an analytical balance with an accuracy of ± 0.01 g. The fractional fiber weight was calculated with the following equation,

$$F_i = \frac{X_i}{X_T} \times 100\% \quad (1)$$

where F_i is the proportion of rice husks with diameter range of i ; X_i is the rice husks weight with diameter range of i ; and X_T is the total weight of all rice husks.

The morphology of the rice husks was determined using a JEOL JSM-6380LV scanning electron microscope (SEM; Japan) operating at an accelerating voltage of 30 kV. The treated and untreated specimens were mounted on metal stubs with double-faced tape, and the surface was coated by a gold alloy.

The chemical composition (*i.e.*, cellulose, holocellulose, lignin contents, alcohol-benzene extract, and mycoprotein) of the treated and untreated rice husks were analyzed according to the ethanol nitrate method (Ververis *et al.* 2007), GB/T 2677.10 (1995), GB/T 2677.8 (1994), GB/T 2677.6 (1994), and the Kjeldah method (Tang *et al.* 2011), respectively.

Attenuated total reflectance Fourier transform infrared spectroscopy (ART-FTIR) was used to examine any changes in the structure of the rice husks during their composting treatment. Spectra of dried powdered samples were obtained with a Nicolet iS10 ART-FTIR (Thermo Scientific, USA) in the range of 4,000 to 500 cm^{-1} ; a total of 32 scans were accumulated at a resolution of 4 cm^{-1} .

Thermogravimetric analysis (TGA) was performed to study the degradation characteristics of the rice husks before and after composting. The thermal stability of each sample was determined using an S II-7200 series thermogravimetric analyzer (TA Instruments, USA) with a heating rate of 20 $^{\circ}\text{C}/\text{min}$ in a nitrogen environment (20 mL/min). The temperature range was from room temperature to 600 $^{\circ}\text{C}$. The sample pan was placed on the Pt basket in the furnace, and approximately 5 mg of each sample was used for measurement.

Compression tests were carried out according to ASTM D638 (2007) with a universal mechanical testing machine (MWW-50, Shanghai Hengyi Precision Co., Ltd.; China). The cross-head speed was 10 mm/min, and the tests were performed at a temperature of 25 $^{\circ}\text{C}$.

All rheological experiments were performed using a ZJL-200 torque rheometer (Changchun Zhineng Instrument Co., Ltd.; China) operated in controlled-stress or strain rate modes. Both the treated and untreated rice husks were dried. The rotational speed and experiment temperature were 80 rpm and 120 $^{\circ}\text{C}$, respectively.

RESULTS AND DISCUSSION

Diameter Distribution

The diameter distribution of the rice husks before and after the composting processes is shown in Fig. 2. About 31.0% of untreated rice husks had a diameter in the range of 250 to 380 μm ; 8.2% of samples composted with inoculants and 16.2% of samples composted without inoculants had diameters in the same range. Another interesting result is that at diameters greater than 1700 μm , the fiber fraction percentage increased from 7.0% to 37.0% for composting with inoculants and to 29.7% for composting without inoculants.

About 50.8% and 45.6% of the rice husks processed with and without inoculants, respectively, had diameters in the range of 830 to 1700 μm , and 42.4% of the untreated husks had diameters in the same range.

The average diameter of the composted rice husks with inoculants was larger than that without inoculant. The average diameter of the composted rice husks was also larger than that of the untreated husks. This may be because the rice husk particles adhered to each other by bacterial protein produced in the composting process. Another phenomenon is that the partial removal of hemicellulose and surface impurities during the composting process provided the husks with a more uniform and homogenous structure. Similar results were reported earlier by Hornsby *et al.* (1997) and Sain and Panthapulakkal (2006).

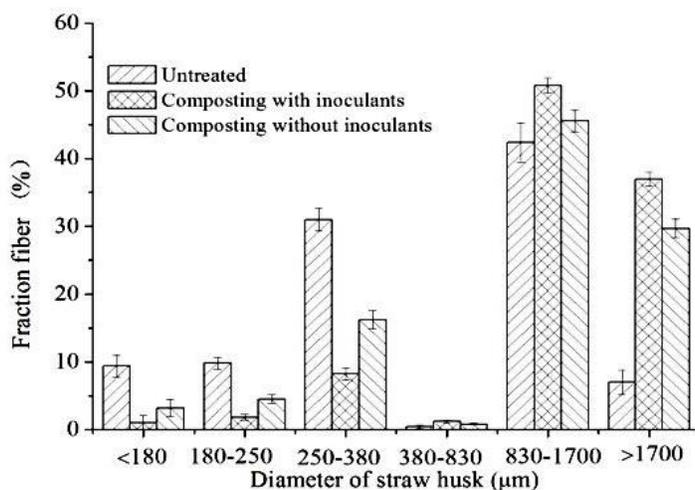


Fig. 2. Diameter distribution of untreated and treated rice husks. Data presented as the mean \pm standard deviation (N=5)

SEM Characterization

The composting treatments resulted in physical structural changes, as well as chemical changes. The SEM images of the rice husks were taken to investigate the structure of these husks, and the corresponding images are shown in Fig. 3. As is well known, the epidermis of the husks in both composting treatments is rich in cellulose, hemicellulose, lignin, and pectin, and has a concentrated layer of silica on the surface (Hornsby *et al.* 1997). Usually, the hemicelluloses and cellulose are easily decomposed. Figure 3a shows an image of the untreated rice husks. It can be seen that the structure of its epidermis is tight and smooth. After the composting treatment, the epidermis appears loose, and part of the epidermis is degraded (Figs. 3b and 3c). The images also suggest that the composition of epidermis (cellulose, hemicellulose, lignin, and pectin) was partially decomposed. It was also proved the result of diameter distribution. Also, the epidermis of the rice husks with inoculants was much rougher than that without inoculants. This showed that the rice husks with inoculants were degraded severely.

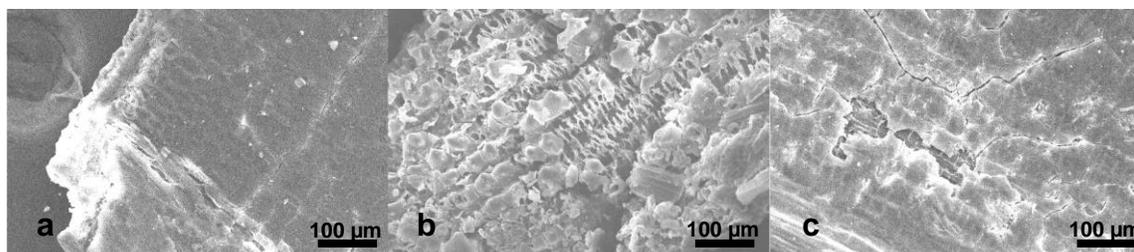


Fig. 3. Surface morphological structure of rice husks that are (a) untreated, (b) composted with inoculants, and (c) composted without inoculants

Chemical Composition

Table 2 shows the main chemical composition of rice husks with or without composting treatment. The chemical composition was related to the degree of degradation. For example, it was found that after composting the rice husks with inoculants, cellulose content decreased from 35.23% to 22.06%, and hemicelluloses content decreased from 24.39% to 19.30%. Also, lignin content increased from 12.92% to 18.31% and alcohol-benzene extract content increased from 1.28% to 1.97%. Epidermis that was in direct contact with microorganisms degraded first, with the degradation of cellulose and hemicelluloses. The degradation of cellulose and hemicellulose can also be confirmed from the morphological surface structure of rice husks, as shown in Fig. 3. The degradation occurred because the microorganisms in inoculants produce enzymes, such as cellulase and xylanase, which can decompose cellulose and hemicellulose. Meanwhile, the content of mycoprotein increased as result of microorganism metabolism. It was also demonstrated that the diameter of samples after composting increased because the microorganisms secrete a protein that allows adhesion between the rice husk particles.

Table 2. Chemical Composition of Untreated and Treated Rice Husks

Rice husks	Cellulose ^a (%)	Hemicellulose ^a (%)	Lignin ^a (%)	Alcohol-benzene extract ^a (%)	Protein ^a (%)
Untreated	35.23±1.26	24.39±1.98	12.92±1.4 2	1.28±0.15	3.75±0.26
Composting with inoculants	22.06±1.95	19.30±1.02	18.31±1.6 9	1.97±0.18	5.56±0.56
Composting without inoculants	29.48±1.96	21.79±1.89	15.29±1.7 9	1.46±0.14	7.18±0.28

^a All values are reported as mean± standard deviation (N≥5)

ATR-FTIR Analysis

The FTIR spectra of the untreated and treated rice husks, which are shown in Fig. 4, can reveal the chemical structure changes that occur during the composting process. The peaks at 3422 and 1056 cm^{-1} indicate the stretching vibrations of O-H and C-O, respectively (Xiao *et al.* 2001). The prominent peak at 1737 cm^{-1} in the treated rice husks can be attributed to either the acetyl and uronic ester groups of the hemicellulose or the ester linkage of the carboxylic group of the ferulic and *p*-coumeric acids in lignin and/or hemicellulose (Sain and Panthapulakkal 2006). This peak height decreased completely after the composting process because of the degradation of most hemicellulose in the rice husks. The decrease in the intensity at 1456, 1366, 1312, and 1242 cm^{-1} suggest the partial removal of the hemicellulose. The small band at 1705 cm^{-1} originated from the

unconjugated ketone or carbonyl stretching, while a shoulder at 1638 cm^{-1} is attributed to carbonyl stretching conjugated with aromatic rings. Aromatic skeleton vibrations are assigned at 1606 , 1513 , and 1426 cm^{-1} . Absorption at 1466 cm^{-1} is indicative of C–H deformations and aromatic ring vibrations. The peaks at 1507 cm^{-1} and 1436 cm^{-1} in the untreated rice husks, representing the aromatic C=C stretch of aromatic rings of lignin, were observed (Xiao *et al.* 2001). The intensity of these peaks decreased in the compost-treated fibers because of the partial removal of the lignin. The sharp peak observed at 1385 cm^{-1} reflects C–H asymmetric deformations. The peaks in the region 1200 to 950 cm^{-1} are due to C–O stretching (Sun *et al.* 2005). The absorption bands at 750 and 710 cm^{-1} in the FTIR spectra of cellulose were assigned to the I_{α} and I_{β} phases, respectively (Sugiyama *et al.* 1991). In the FTIR spectra of compost-treated fibers, the absorption band at 750 cm^{-1} was not detectable, which indicates that there was no cellulose I_{α} crystalline polymorphism in the rice husks. There was only the absorption band at 710 cm^{-1} , which indicates that only cellulose I_{β} crystalline polymorphism existed in the rice husks. The composition changes discussed above were beneficial to the processability of rice husks.

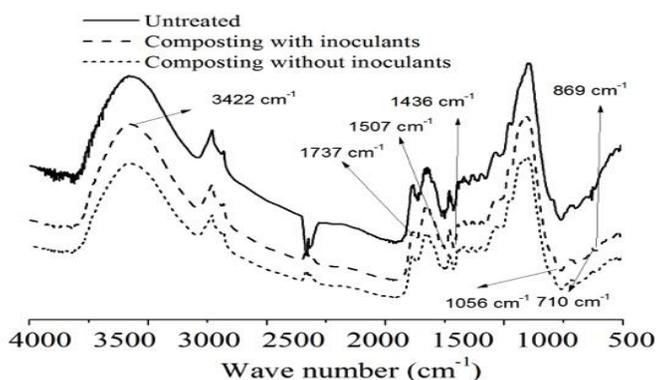


Fig. 4. FTIR spectra of rice husks with and without composting

Thermogravimetric Analysis

Investigation of the thermal properties of the rice husks is important in order to gauge their applicability for processing. The TGA thermograms of the rice husks prepared by treated and untreated samples are presented in Fig. 5. The degradation characteristics of the rice husks treated with and without composting are shown in Table 3. In general, there are three stages of degradation in the TGA curves of all the samples. The first stage in the TGA curves, which were observed at room temperature to $130\text{ }^{\circ}\text{C}$, was related to the loss of adsorbed and bounded water. Both of the treated samples adsorbed less water, with their loss depending on the initial moisture content of their fibers. The second stage was related to severe weight loss and was observed at 200 to $400\text{ }^{\circ}\text{C}$. Both the second and third stages were due to the decomposition of the major components in the husk fibers. These results regarding the second and third stages are consistent with the data reported in the literature (Shafizadeh *et al.* 1982; Ghetti *et al.* 1996). The higher temperature onset for the degradation of the composted rice husks, with and without inoculants, and untreated samples occurred at $231.2\text{ }^{\circ}\text{C}$, $228.7\text{ }^{\circ}\text{C}$, and $217.5\text{ }^{\circ}\text{C}$, respectively. This demonstrates the suitability of these fibers for processing with thermoplastics, which indicates improved thermal stability of the rice husks after composting treatments.

The higher thermal stability of the treated fibers relative to untreated fibers is attributed to the higher lignin content in the raw material, since lignin has a lower softening

temperature. Composted rice husks also exhibit lower peak degradation temperatures (349.5 °C when using inoculants, while 350.6 °C without inoculants) than the untreated samples (360.7 °C). The decrease of maximum temperature indicates a lower diffusion of the degradation products formed. However, the rate of degradation in the untreated fibers (2.42 wt.%/min) was higher than that for the compost-treated fibers (2.15 wt.%/min and 2.28 wt.%/min). There is also a distinction between the amounts of residues remaining in treated and untreated fibers after being heated to 600 °C. Results showed that treated husk fibers have more residue remaining at 600 °C, which may be attributed to the partial removal of silica from the rice husks that occurs during composting.

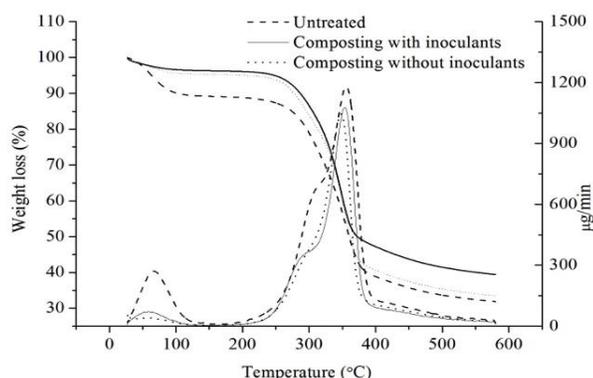


Fig. 5. TGA thermograms of rice husks with and without composting

Table 3. Degradation Characteristics of Rice Husks With and Without Composting

Rice husks	Moisture content (%)	Onset temperature of degradation (°C)	Temperature of maximum reaction velocity(°C)	Rate of degradation (wt. %/min)	Residue after 600 °C (%)
Untreated	9.8	217.5	360.7	2.42	31.8
Composting with inoculants	3.5	231.2	349.5	2.15	39.3
Composting without inoculants	3.9	228.7	350.6	2.28	33.5

Compressive Properties

Figure 6 shows typical stress-strain curves of rice husks with and without composting treatment. The initial, relatively flat part of the curve represents elastic behavior, and the slope of the curve defines the elastic modulus. It can be observed that the compressive modulus of elasticity was proportional to the maximum load. The stress-strain curves for composted husk fibers showed a moderate change of slope at about 0 to 10% strain. The composting treated curve showed a typical yielding process and brittle nature followed by strain hardening at the low strain rate used in the compressive test (10 mm/min). The untreated rice husks exhibited a quasi-ductile behavior under compressive deformation. Both of the rice husks tended to be brittle when the modulus of elasticity was high and tended to be ductile or flexible when the value was low. The composted samples showed a higher modulus of elasticity than untreated samples. This means that rice husks

with the composting treatment have better mechanical properties than untreated samples (Premalal *et al.* 2002). The mechanical properties of composted rice husks with inoculants were better than those without inoculants because they were degraded more extensively.

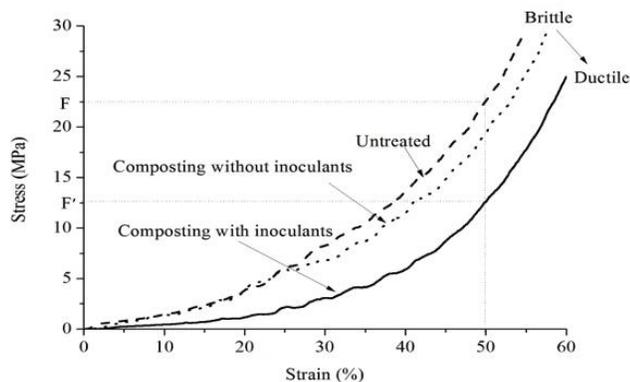


Fig. 6. Stress-strain curves of rice husks with and without composting

Rheological Properties

The torque of rice husks with and without composting is shown in Fig. 7. It can be observed that an initial loading peak was registered immediately after the rotors were started and that reflects the high viscosity of the rice husks. A sudden decrease of torque immediately after the loading peak and subsequent gradual decrease of torque indicates this drop because the rice husks became uniform in the mixing chamber. Then torque rose gradually again because the mobility of the macromolecule chains of husk fibers decreased.

Once the dispersion and incorporation were completed, torque decreased gradually and tended to become stabilized in all of the husks. The loading peaks of the treated samples (8.7 N·m for rice husks with inoculants and 8.4 N·m for rice husks without inoculants) were both higher than that of untreated samples (8.1 N·m). The time untreated samples started to melt under shear and high stock temperature was after 40 s. The melting time was 90 s for treated samples with inoculants, and 70 s for treated samples without inoculants. When the stock temperature stabilizes at the set temperature, the viscosity of the material remains at an approximately constant value (Premalal *et al.* 2002). Further investigation of Fig. 6 shows that the lapse of time to achieve stable torque in untreated samples was shorter than that of treated samples. Also, it is clearly indicated that the treated samples exhibited higher stabilization torque than the untreated samples.

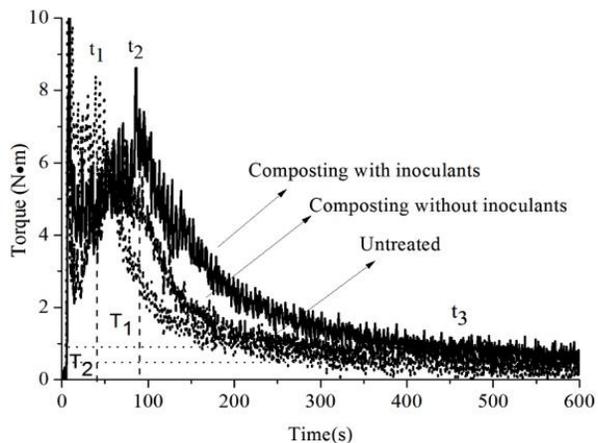


Fig. 7. Rheological properties of rice husks with and without composting

Such increase in stabilization torque could be due to either the adhesion of rice husk particles by the bacterial protein or the formation of stronger hydrogen bonds. After shearing for 400 s, untreated samples showed almost stable torque (0.5 N·m) and indicated the completion of melting and almost constant viscosity at fixed mixing conditions. The stable torque of compost-treated samples with inoculants was 0.9 N·m and occurred at 500 s. For compost-treated samples without inoculants, stable torque was 0.7 N·m, and this value was observed after 500 s.

CONCLUSIONS

1. The physical structure of rice husks was changed during the composting process. The average diameter of rice husks increased as result of bacterial protein. The surface morphological of rice husks became rugged and lumpy because of lignocellulose decomposition.
2. The chemical composition and structure also was changed in the process. The lignin content and alcohol-benzene extract were increased in compost-treated husks. Meanwhile, the proportional amounts of cellulose and hemicellulose were decreased as result of degradation. The ATR-FTIR and TGA also showed that the functional groups and thermal stability were higher, which is beneficial to the usage.
3. The rheological properties were improved by the composting treatment. The stress-strain curves for composting husks showed a moderately change of slope at about 0 to 10% strain, which was useful for improving the strength of the rice husks. The change in rheological properties showed that the loading peak and stable torque of compost-treated samples were higher than that of untreated samples because the apparent viscosity was improved under the composting treatment.
4. All these results indicate that the composting process is an effective method to improve the processability of rice husks.

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