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Development and Characterization of Food Packaging Bioplastic Film from Cocoa Pod Husk Cellulose Incorporated with Sugarcane Bagasse Fibre

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A B S T R A C T

Agricultural wastes, including cocoa pod husk (waste from the chocolate industry) and sugarcane bagasse (waste from the sugar industry), are increasing day by day. The development of food packaging biofilms from these two wastes could be beneficial to the environment and human. Therefore, this study was conducted to develop biodegradable plastic films by using cocoa pod husk and sugarcane bagasse. Cellulose and fibre were extracted from cocoa pod husk and sugarcane bagasse, respectively. The developed bioplastic films were divided into several concentration ratios of cellulose and fibre which are 100:0 (100% cellulose), 75:25 (cellulose:fibre), 50:50 (cellulose:fibre), 25:75 (cellulose:fibre), and 0:100 (100% fibre). The physicochemical properties for all bioplastic concentration ratios were determined in terms of sensory evaluation, drying time, moisture content, water absorption and water vapor permeability. From the observation and analysis of the physicochemical properties of bioplastic, we found that the most suitable bioplastic film for food packaging goes to the combination of 75% cellulose and 25% fibre bioplastic, as it demonstrated the lowest water absorption percentage and water vapor permeability.

1 Introduction

Plastic is estimated to make up of 10 percent of household waste, and most of it is dumped to the landfills (Barnes and Milner, 2005). Plastic waste has several effects on human health and the ecosystem. Incorrectly managed landfills lead to the escape of plastic waste containing chemicals. The impact of chemicals on human and ecosystem is due to the chemical present in plastic or plastic waste during transportation. Plastic waste burning will increase heart disease risk, cause rashes, nausea or headaches, as well as damaging the nervous system. According to North and Halden (2013), plastics are disposed of in landfills or through incineration (Verma et al., 2016; Canopoli et al., 2018), but the chemical constituents and energy content of the plastics will be lost in the process (Devasahayam et al., 2019). The polymers diversity and their property versatilities facilitate vast production of plastic products which bring technological advantages, energy savings and a lot of societal benefits (Andrady and Neal, 2009).

In recent years, the enthusiasm for the development of bioplastics has become more substantial and more significant under the idea of “waste to wealth”. The utilisation of biomass like fibre, cellulose and starch to replace petrochemical materials for the production of plastics, is a widely accepted strategy to establish a sustainable society (Fiorentino et al., 2017; Ilyas et al., 2019; Karan et al., 2019; Scott and Buchard, 2019). Bioplastics or biodegradable polymers can be defined as plastics which are made from biomass (Saharan and Sharma, 2012). The bioplastic production saves fossil fuels, reduces carbon dioxide emission and plastic pollution in the environment (Abdul-Latif et al., 2020). The biodegradability of bioplastic is widely publicised. The packaging demand is increasing among retailers and in the food industry. It is because conventional plastic not only takes many years to degrade, but it also produces toxins during degradation. However, the high production cost and the availability of the low-cost petrochemical-based plastic lead to the negligence of bioplastic. Cost of bioplastic production is higher than that of the conventional plastics (Petersen et al., 1999). However, the use of bioplastic can reduce the environmental impact of plastics

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primarily in relating to the toxic pollutant from non-degradable plastics, and the amount of carbon dioxide emitted. Consequently, the greenhouse that caused global warming is reduced because of increasing bioplastic feedstock balance of carbon dioxide in the atmosphere. Some bioplastics have properties similar to those of traditional counterparts (Dietrich et al., 2017). However, these properties must be tested and examined.

There are several types of bioplastic where cellulose, starch, fibre and protein bioplastic are the most common types. Starch-based bioplastic is familiar in the industry as it is in high abundance, low cost, and the renewability. However, starch alone is not thermos-plastic. It has to be combined with a filler to modify and enhance the mechanical properties of the bioplastic (Agustin et al., 2014). Cellulose, starch, fibre and protein can be obtained from agricultural waste produced from food processing of essential products. As an example, cocoa fruit (*Theobroma cacao* L.) offers cocoa powder as the main product, and cocoa pod husk (CPH) as waste whereas sugarcane (*Saccharum officinarum*) yields juice and sugar as the main products; and sugarcane bagasse as waste. The CPH is the unused residue after the removal of the cocoa bean from the fruit, the non-consumable part of the cocoa pod and composed of 52% –76% of the overall weight of cocoa pod (Koay et al., 2013). Meanwhile, sugarcane bagasse is the crushed cane after the juice from sugarcane is being extracted. Both wastes were commonly thrown away without knowing the benefits of transforming it into bioplastic or anything else. Therefore, the CPH can be used as a source of cellulose while sugarcane bagasse can be utilised as a fibre source in the bioplastic production.

From the literature, the combination of cellulose and fibre in bioplastic production helps in improving bioplastics physicochemical properties. Thus, the objective of this study is to determine the physicochemical properties of bioplastic made from the CPH incorporated with sugarcane bagasse. The films are targeted as food packaging material, which requires the physicochemical properties test to proof the product shelf life. In this study, fibre and cellulose were extracted from sugarcane bagasse and CPH, respectively, before they were used in the bioplastic production. Sensory evaluation, drying time, water absorption, moisture content and water vapor permeability of each developed bioplastic were measured and analyzed.

2 Materials and Methods

2.1 Sample collection and pre-treatment

The CPH sample was collected from the local chocolate company in Pahang, Malaysia. In contrast, sugarcane bagasse sample was collected from a sugarcane juice stall in Kuala Kubu Bharu, Selangor Malaysia. The collected samples were kept for pre-treatment that involved two steps, which were drying and sizing. The CPH sample was placed in an oven for the drying process at 60 °C for four days after the samples were cut into small pieces approximately 1 cm thick. Meanwhile, sugarcane bagasse sample was cut into small pieces about 3.0 cm × 0.5 cm in size and put in a tray subjected to the sun-drying process for five days. Finally, both dried samples were ground and sieved to obtain the fine powders for extraction.

2.2 Cellulose extraction from cocoa pod husk

Cellulose was extracted from the CPH by referring to the procedure reported by Lisin et al. (2015) with some modifications. Initially, 40 g of fine CPH powder was undergone an alkaline treatment for three hours at 100 °C by soaking the sample in 700 mL of NaOH solution with a concentration of 1 mol/L. After the procedure, the sample was cleaned up by filtering with distilled water to remove excess NaOH residue until neutral pH was achieved. Then, the sample was dried in an oven for 24 h at 60 °C before continuing with the bleaching process for 45 min at 70 °C on the hot plate using hydrogen peroxide (H_2O_2) as a bleacher. Then, the bleached sample was appropriately washed using distilled water to remove the bleach residue until neutral pH was attained. After bleaching, the sample was dried once more in an oven at 50 °C for 24 h. The dried and bleached sample was ground and sieved to obtain cellulose powder.

2.3 Fibre extraction from sugarcane bagasse

The procedure conducted for the fibre extraction from sugarcane bagasse was pointed out by Beninia et al. (2011) with a few adjustments. Firstly, 100 g of fine bagasse powder was treated with 1000 mL of NaOH solution for an hour with constant stirring at room temperature. After that, the treated bagasse solution was washed and filtered using distilled water until a neutral pH was obtained. The extracted fibre was dried in an oven at 50 °C for 24 h. Then, 25 g of the dried fibre was bleached by stirring it with a 200 mL solution containing 1 mL of vinegar and 3 g of NaCl on the hot plate for two hours at 70 °C. The procedure continued with the filtration and washing of the resultant using distilled water to neutralise the pH. The bleached fibre was oven-dried at 50 °C for 24 h. The bleaching treatment was done to remove lignin from the composite. This bleaching process allowed the fibres to be less susceptible to UV and moisture, which was considered as a factor for the degradation process (Beninia et al., 2011). The dried and bleached fibres were kept for further use after grinding and sieving procedures.

2.4 Bioplastic film development

The 1.5 g of the CPH cellulose powder, 1 mL of glycerol, 40 mL of distilled water, and 0.5 g sorbitol were prepared. All of the ingredients were mixed in a 250 mL beaker and stirred on the hot plate for 15 min until the water evaporated, and the solution started to become sticky. Then, the mixture was poured and spread into a glass petri dish to produce a cellulose-based plastic. This bioplastic film was dried in the oven for two hours at 50 °C and then remained at room temperature for three days approximately to reach the constant weight. This bioplastic film is labelled as a control sample for cellulose as it was not added with fibre.

On the other hand, the same procedure was performed for bioplastic film production from sugarcane bagasse. All of the ingredients consisting of 1.5 g of bagasse fibres, 1 mL of glycerine, 40 mL of distilled water, and 0.5 g sorbitol were prepared and blended in a 250 mL beaker. After that, the solution was stirred on the hot plate for 30 min until the evaporation ultimately occurred, and the solution became viscous. Then, the mixture was poured and spread into a glass petri dish to produce a fibre-based plastic. The bioplastic film was dried for an hour at 50 °C in the oven and then dried at room temperature for two days approximately.

The sequential steps in developing the bioplastic film were repeated for the bioplastic derived from cellulose (CPH) incorporated with fibre (sugarcane baggage) at different concentration ratios. However, the total weight for each ratio is still the same, which is 1.5 g. The concentrations used were 100:0, 75:25, 50:50, 25:75, and 0:100 of cellulose to fibre ratio and vice versa. Table 1 shows the stirring time for each different concentration ratios together with the drying time required for bioplastic in the oven. Different stirring and drying times occurred because of the modified rate of the evaporation process that depends on the viscosity of the solution.

Table 1 Stirring and drying time for each concentration ratio

Ratio of cellulose to fibre	Stirring time (min)	Drying time (h)
100:0	15	2.0
75:25	15	2.0
50:50	20	1.5
25:75	25	1.0
0:100	30	1.0

2.5 Bioplastic film characterisation and measurement

Three parameters were chosen for the bioplastic characterisation and measurement, which were drying time, water absorption and moisture content. Each bioplastic film weight was acquired before the bioplastic drying process started. Then, the bioplastic film was weighed every day until a constant weight was reached. Once the constant weight was obtained, the drying time measurement was completed and recorded.

Meanwhile, water absorption analysis was conducted in triplicate to get the average result by submerging each of the bioplastics into 20 mL distilled water at room temperature. The time for the bioplastic to start degrading or breaking down was recorded by observing the changes strictly on its appearance. At this critical time, the sample was taken out from the beaker, and the sample weight was measured immediately after wiping out the excess water on its surface. Before the water absorption analysis, the trial-and-error method was done to determine the right duration needed for the bioplastic to start degrading when submerged into water. The percentage of water absorption was calculated according to Equation (1):

$$W_A\% = \frac{[W_S - W_0]}{W_s} \times 100\% \quad (1)$$

where W_A (%) is the percentage of water absorption, W_S is the sample weight after submersion (wet weight), and W_0 is the sample weight before immersion (dry weight).

The measurement of bioplastic moisture content started with the weighing of initial bioplastic weight. After that, the sample was dried at 105 °C in the oven for 24 h before the sample was reweighed. These steps were repeated until the sample reached a constant weight. The moisture content in bioplastic was calculated according to Equation (2).

$$Moisture\,content\ (\%) = \frac{(w-d)}{w} \times 100\% \tag{2}$$

where w is the initial weight of the bioplastic, and d is the final weight or the constant dry weight of bioplastic.

The measurement of water vapor permeability was evaluated by using the gravimetric method at 25 °C, referring to ASTM E-96-00. All samples were sealed using elastic-plastic in Erlenmeyer flasks containing a desiccating agent which was silica. The flasks were placed in a desiccator at 75% relative humidity (RH) at 25 °C. The procedures followed the methodology explained by Luchese et al. (2015). Water vapor permeability (WVP) was measured by determining the weight gained of the samples after 48 h according to Equation (3).

$$WVP = \frac{w}{t} . \frac{1}{A} . \frac{e}{P_s\left(RH_1 - RH_2\right)} \tag{3}$$

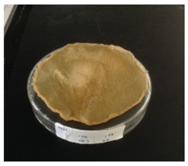
where $\frac{w}{t}$ is mass gain (g/d), A is biofilm area (m^2), e is the average thickness of biofilm (μm), P_S is saturation pressure of water vapor at tested temperature (mmHg), RH_1 is relative humidity inside the Erlenmeyer flask, and RH_2 is relative humidity inside the desiccator. This method uses Fick's first law and Henry's law to calculate film WVP and assumes that film solubility and diffusivity are constant (Bertuzzi et al., 2007).

3 Results and Discussion

3.1 Sensory evaluation of bioplastic film

Table 2 shows the sensory evaluation including, colour, texture, smell and physical appearance for each bioplastic formed in a different ratio of the CPH and sugarcane basse. These properties are essential since they are significant factors affecting the

Table 2 Sensory evaluation of bioplastic film

Bioplastic ratio of cellulose to fibre	Sensory evaluation			Physical appearance
	Colour	Texture	Smell	
100:0	Brownish-yellow	Moist	Sweet smell	
75:25	Slightly brownish	Slightly moist	Sweet smell	
50:50	Yellowish	Sticky	Sweet smell	
25:75	Slightly greenish-grey	Dry, brittle	Slightly sweet	
0:100	Greenish grey	Dry, hard	Odourless	

quality perception of the produced product. The results for the colour analysis present the influence of cellulose and fibre concentrations in forming the bioplastic. It is found that the bioplastic colour of the 100% cellulose is brownish yellow; meanwhile, the bioplastic colour of the 100% fibre is greenish-grey. The bioplastic colours indicate the percentage of cellulose to fibre, where the highest cellulose content results in the more brownish bioplastic colour, as shown in Table 2. This brownish colour comes from the CPH, which is the source of cellulose in the bioplastic. It is also noticed that the brownish-yellow of the bioplastic colour faded to greenish-grey as the concentration of cellulose decreases. It is because the fibre colour is white, where the increase of fibre concentration makes the brownish colour of bioplastic become faded.

In the case of texture analysis, it is found that the texture of bioplastic depends on the concentration of cellulose. The bioplastic becomes dryer and harder with the lower cellulose concentration as compared with other ratios. Besides, the presence of cellulose increases the moisture content of bioplastic, where the bioplastic with 100% cellulose is moister than the others. Cellulosic materials slowly absorb or desorb moisture from or to the surrounding air until they reach an equilibrium moisture content (Wang et al., 2018). On the other hand, bioplastics with high cellulose concentration (75% and 50%) have a sweet smell, but, those with a high level of fibre have a slightly sweet aroma. Bioplastic made from 100% of fibre showed greenish grey, hard, dry and odourless while bioplastic made from 100% of cellulose displayed brownish-yellow, moist and sweet smell. This phenomenon occurs because the cellulose source comes from cocoa which initially has the sweet smell.

3.2 Drying time of bioplastic film

Figure 1 presents the drying time of bioplastics at different cellulose (CPH) to fibre (sugarcane bagasse) ratios. From the observation, the longest drying time for the bioplastic to dry (ratio of cellulose to fibre) is seven days as given by the 100:0, followed by the 75:25 with five days, 50:50 with three days, 25:75 with 2.5 days, and two days as the shortest drying time for the 0:100. As can be spotted, the drying time of the bioplastic is directly proportional to the cellulose concentration since it is declined along with the decrease in cellulose concentration. This situation indicates that the level of cellulose gives impact to the bioplastic drying time as it takes a longer time to dry when the percentage of cellulose concentration is high. The incorporation of lower cellulose and higher fibre contents in producing bioplastic would increase the drying time. It is because the fibre is a fast degrader as it takes a shorter time to degrade, thus shorter time to completely dry. In addition, fibre composites exhibit much more weight losses rather than cellulose composites (Song and Zheng, 2009). Thus, the addition of cellulose to fibre in the bioplastic development improves the water absorption properties and drying time as well. In a different case, Herrera et al. (2014) studied the effect of Nano-fibrillated cellulose from Opuntia ficus-indica parenchyma cell reinforced on starch matric. It had revealed that the reinforcement of Nano-fibrillated cellulose enhanced both mechanical and water barrier properties.

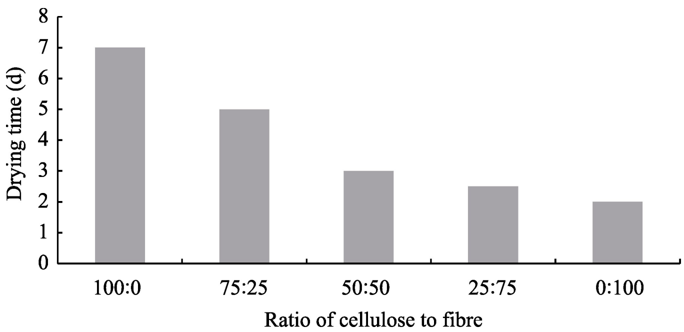


Fig. 1 Drying time for different bioplastic ratios

3.3 Moisture content of bioplastic film

Figure 2 exhibits the moisture content of the bioplastic from cellulose incorporated with fibre in various ratios. The ranking of the moisture content percentages from the highest to the lowest can be arranged based on this cellulose to fibre ratio: 100:0 (20.89%) > 75:25 (17.43%) > 50:50 (12.66%) > 0:100 (11.84%) > 25:75 (8.01%). According to the ranking, it can be seen that the highest percentage of moisture content for the produced bioplastic is contributed by the 100:0 of cellulose to fibre ratio. In contrast, the lowest percentage is given by 25:75 of cellulose to fibre ratio. This case shows that the moisture content decreases with the decrease of cellulose concentration in the bioplastic. However, the trend changed for 0:100 of cellulose to fibre ratio that denoted slightly increase since there is no cellulose presence in the bioplastic.

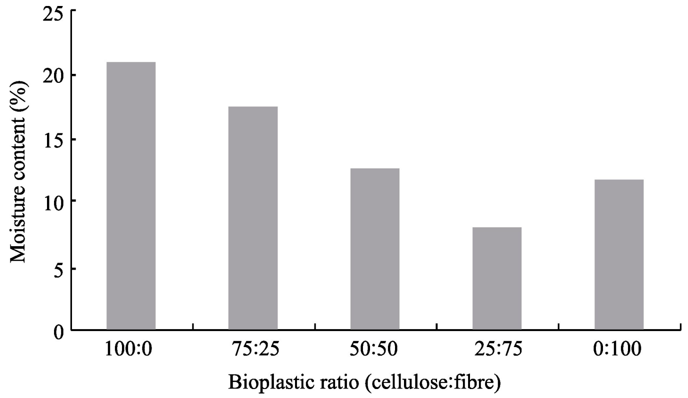


Fig. 2 Moisture content for different bioplastic ratios

The presence of cellulose can reduce the moisture uptake in bioplastic. In this case, hydrogen-bonding within the composite prevents the formation of voids where water molecules can pass through (Agustin et al., 2014). Based on the result, 25% cellulose in the bioplastic is the best bioplastic for moisture content property, as it displays the lowest moisture content, which is 8.01%. Low moisture content helps the bioplastic to reduce the possibility of mould growth which could affect the bioplastic appearance and mechanical property. High moisture content promotes the faster metabolic activity of microorganisms (Borah et al., 2019). In this study, 25% of cellulose bioplastic is the most suitable to be applied as a food packaging material. The high fibre content of bioplastic is preferable to be used as a plastic container because it shows a robust mechanical property. Fibre content increases the mechanical property of the resulting bioplastic (Simão et al., 2016).

3.4 Water absorption analysis of bioplastic film

Table 3 shows water uptake of bioplastic at different cellulose to fibre ratios after 10 min. The percentage of water absorption for each bioplastic was calculated by using Equation (1), where the amount of water uptake after submersion into the water for a specific time is demonstrated in Fig. 3. The percentage of water absorption for each bioplastic is also presented in this figure. The highest water absorption is given by 50:50 (ratio of cellulose to fibre) bioplastic with 46.76%, while the lowest water absorption is 17.62% provided by 75:25 (ratio of cellulose to fibre) bioplastic. The trend of water absorption decreased from 25.63% (100% cellulose-based plastic) to 17.62% (75:25 bioplastic) but, increased for 50:50 (ratio of cellulose to fibre) bioplastic with 46.76%.

Table 3 Water uptake of bioplastic at different ratios of cellulose to fibre after 10 min

Bioplastic ratio of cellulose to fibre	Dry weight (g)	Wet weight (g)	Water uptake (g)
100:0	1.09	1.46	0.37
75:25	1.09	1.33	0.23
50:50	0.74	1.39	0.65
25:75	0.79	1.22	0.43
0:100	0.84	1.04	0.20

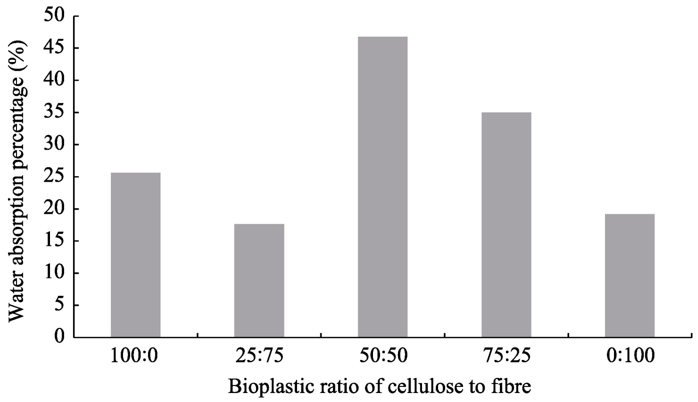


Fig. 3 Percentage of water absorption for different bioplastic ratios

Then, the pattern decreased in sequence for 25:75 (ratio of cellulose to fibre) bioplastic and 100% fibre-based plastic. The phenomenon proved the ability of water absorption for each bioplastic based on its hydrophobic or hydrophilic properties during the submersion. For the 100% cellulose-based plastic, the percentage of water absorption was not too high since cellulose was hydrophilic (Li et al., 2018; Nazarian et al., 2019) towards water cause the water uptake reduced with the concentration of cellulose.

Meanwhile, the water absorptions for 50:50 (ratio of cellulose to fibre) and 25:75 (ratio of cellulose to fibre) bioplastics were relatively higher than others as they were more hydrophilic and the concentration of cellulose decreased for both concentrations. Therefore, both bioplastics absorb more water during the submersion until they broke down at specific durations. In addition, the incorporation between cellulose and fibre also gave an impact on water absorption analysis. The water absorptions for 75:25 (ratio of cellulose to fibre) bioplastic and 100% fibre-based plastic are low as they absorb less water because the bioplastics become less hydrophilic, resulting in less sensitivity towards the water during the submersion (Judawisastra et al., 2017). The best bioplastic is 75:25 (ratio of cellulose to fibre) bioplastic as it exhibits the lowest water absorption percentage. Meanwhile, the least preferred bioplastic is 50:50 (ratio of cellulose to fibre) due to the highest water absorption percentage. This result shows that the bioplastic film is highly sensitive towards the water, where it might decrease the mechanical property of bioplastic as a food packaging.

3.5 Water vapor permeability (WVP)

Table 4 shows the data for water vapor permeability for all developed bioplastic films. It is found that bioplastic with ratio of 75:25 presents the lowest WVP as compared with the others. Low WVP means that the bioplastic can prevent the transfer of moisture between the food and environment (Luchese et al., 2015). Therefore, bioplastic with low WVP is the best to be applied as food packaging. Bertuzzi at al., (2007) reported that cellulose derivatives could be applied as edible films and coatings in food packaging and preservation.

Table 4 Water vapor permeability of different bioplastic ratios of cellulose to fibre

Bioplastic ratio of cellulose to fibre	Water vapor permeability $\left(\frac{\text{g } \mu\text{m}}{\text{day m}^2 \text{ mmHg}}\right)$
100:0	0.32
75:25	0.30
50:50	0.42
25:75	0.43
0:100	0.50

4 Conclusions

In this study, the utilisation of the CPH incorporated with sugarcane bagasse as bioplastic materials were investigated. The results showed that the best bioplastic film with balanced tested physicochemical properties went to 75:25 (ratio of cellulose to fibre) bioplastic, as water absorption and the WVP properties played the crucial roles in selecting the suitable bioplastic for food packaging. It could reduce the possibility of mould growth on the bioplastic surface and could prevent the transfer of moisture between food and environment, which could preserve the bioplastic and food for more longer. In addition, the hydrophilic nature of cellulose-based bioplastic reduced the water vapour barrier that could cause the brittleness and poor mechanical properties of the resulting packaging material. The addition or incorporation of fibres in a small amount in the bioplastic composite reduced the susceptibility towards the water as water molecules could not diffuse into the composite. In the future, more studies on the CPH incorporated with sugarcane bagasse waste could contribute to the economic growth of society and promote a green environment as the agricultural wastes can be converted into new value-added products.

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