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BIODEGRADABLE PLASTIC FROM THE SHELLS OF *ARACHIS HYPOGAEA* (GROUNDNUT)

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ABSTRACT

In recent years the enormous study was focused on to overcome the pollution caused by plastics and also an akin alternative for plastic which should not risk the biomass. Mostly starch and cellulose-based plastics are chemically modified using plasticizer where it has a high impact on biodiversity as well as expensive. But our study indicates the extraction of cellulose from natural waste (Shells of *Arachis hypogaea*) and reinforced with moringa resin as a natural binder compressed to form plates and cups which were coated with beeswax. The mould was characterized by Fourier transform infrared spectroscopy (FTIR), X-Ray Diffraction (XRD), Thermal gravimetric analysis (TGA), Scanning Electron Microscope (SEM) analysis and American Society for Testing and Materials (ASTM) standard testing for plastic. The mould is shown to be readily degraded within 7-10 days under controlled soil condition. The mould framed can be used for food packaging industries which would reduce half a chance of pollution caused by dumping the petroleum-based synthetic polymers in agricultural lands and oceans. These biodegradable polymers serve as a promising alternative due to its unique quality of economical and high biodegradability rate.

KEYWORD: Cellulose; Resin; Mould; SEM; TGA; Biodegradable Plastic; Biopolymer.

1. INTRODUCTION

Plastic plays a vital role in day to day life everywhere in the world for their various properties like durable, readily available, inexpensive and versatile (Lubis *et al.*, 2018). Plastics are made up of petroleum-based components which take more than 1000 years to completely decompose a single plastic waste. Because of its unique characteristics of high molecular weight with tightly bonded molecules, they are non-degradable and difficult to be discarded (Nazirah and Zulkafli, 2014). Every ecosystem including marine, freshwater, terrestrial and deserts poses numerous environmental problems by dumping plastic (Thompson *et al.*, 2009). Due to the prodigious utilization of petroleum and oil resources have also been threatened to become depleted (Rasheed, 2011).

On this account, it is consequential to replace the petroleum-based products with products derived from renewable resources. Many scientists have prompted to replace part of a petrochemical-based polymer with other biodegradable compounds called bioplastics due to the environmental, economic and safety challenges (Reddy, Reddy and Gupta, 2008). Bioplastics are made by renewable resources from plant sources such as starch, cellulose, lignin, and animal sources such as casein, protein and lipids (Avérous, 2004). But there are some limitations in the development of bioplastics due to its high production cost. Agriculture waste as a low cost and the renewable substrate has been proposed as the best alternative (Jain and Tiwari, 2015). Starch as a biodegradable polymer becomes renewable material for the production of bioplastics because of its low cost along with their performance and abundant sources (Maulida, Siagian and Tarigan, 2016). Unfortunately, there are some strong limitations for developing starch-based bioplastics, since they are extremely brittle. Films with satisfactory mechanical properties like high percentage elongation, tensile and flexural strength cannot be done with starch without being plasticized, blended with other materials, chemically modified, or modified with a combination of these treatments (Mostafa *et al.*, 2018).

Peanut botanically known as *Arachis hypogaea* belongs to the Leguminosae family. China (2-2.5 million tons) and India (1.5-2 million tons) are the leading producers of groundnut (Bharthare *et al.*, 2014). The peanut harvest and oil extraction process generate sustainable amounts of by-products such as peanut meals, skins, shells, and vines whereas only a few are utilized as animal feed, fertilizers but a large portion is regarded as agricultural waste which are potential pollutants to the environment. At present, many researchers focus on the investigation to facilitate quality production of edible oil from the kernel. Thus, scant attention was given to the by-

products from peanut industries. Especially, very few reports have been done on examining the possibility of peanut shell utilization. Peanut shells are an abundant and inexpensive by-product of the peanut processing industries. Most of the peanut shells are set on fire or arbitrarily discarded. The improved value utilization of peanut shells provides great economic significance. If the nutritional compositions of by-products from peanut processing industries are recovered and recycled, it can be regarded for a crucial economic and social benefit (Zhao, Chen and Du, 2012).

Many functional components present in peanut shells are considered to be safe to humans. They also contain cellulose (40%), lignin (26.4%) and hemicelluloses (14.7%) (Kiran *et al.*, 2013)**.** Cellulose found in all plant material and an ample inexpensive natural polymer equivalent to starch which also comprised glucose monomer units. In cellulose, the glucose units are linked together by β-1,4 glycosidic linkages, which facilitate the tight packing of cellulose chains to form strong inter-chain hydrogen bonds (Liu, 2006)**.** By utilizing the cellulose from peanut shells, biodegradable plastic can be produced. The cellulose-based plastics can also be applied to several fields due to its biocompatibility and biodegradability. However, there are some difficulties to barely use cellulose for the production of biodegradable plastic because of its poor tensile strength and hydrophilic nature. The crucial way to enhance these properties is to combine the polymer matrix with a filler or a reinforcing fibre (Ghazanfari, 2005). The resulting materials are called composite which poses enhanced physical and mechanical properties of the cellulose. The most common gum used for strengthening are moringa, neem, aloe vera and so on, which are readily available in all parts of the world (Choudhary and Pawar, 2014). Resin plays a major role in the pharmaceutical world where it is used as binders for tablets, emulsifiers, gelling agents and coating agents in micro-capsules. As compared to the man-made polymers, these natural gums are non-toxic, cheaper, eco-friendly and do not create any side effects (Goswami and Naik, 2014). Moringa resin contains more polyunsaturated fatty acids and it has a good binding capacity (Pal and Singh, 2014; Builders, P.F., Olayemi, O.J. and Mbah, C.C., 2017). Therefore, it can be used as an excellent binder for the preparation of biodegradable plastic from cellulose which helps to improve the tensile strength and toughness of the material.

Beeswax is a natural glazing agent which is used to prevent water loss in food packaging and long-term storage of food materials. Beeswax helps in the prevention of water loss, retard shrinkage and spoilage in fruit and cheese. Due to its high hydrophobicity beeswax is employed as a most effective material to decrease water vapour permeability of edible films and it also does not change its state at moderate temperature (Trevisani *et al.*, 2017). Beeswax coating enhances the moisture barrier properties in the cellulose-based biodegradable plastic which is a natural alternative to plastic envelopes that do not harm the environment.

The goal of this work is to utilize the low cost cellulosic raw material for the preparation of biodegradable plastic from cellulose with moringa resin as a binder and beeswax is used as a natural glazing agent which prevents water permeability. Moreover, this work investigates the natural and chemical structure of the samples, thermal stability, biodegradability and mechanical property of the samples according to ASTM standards.

2. EXPERIMENTAL

2.1 Materials

Peanut shells were obtained from the local market in Kovilpatti. They were thoroughly rinsed to remove extraneous impurities and dried shells were powdered for extraction. This is an extremely low-cost material, which is used as firewood. Moringa Resin was collected from *Moringa oleifera* tree from the neighbouring garden around Kovilpatti region. The solid formed resin was ground and sewed to remove impurities, which is used as a powder for mould casting. Beeswax was extracted from the honeycomb collected from the garden area in Elayirampannai. Other chemicals used in the experiment, Sodium hydroxide (NaOH) and Sodium hypochlorite (NaOCl) were supplied by Spectrum Reagents and Chemicals Pvt.Ltd. and Reachem Laboratory Chemicals Pvt.Ltd. respectively.

2.2 Extraction of Cellulose

Groundnut shell powder was treated with 0.5N Sodium hydroxide in 1:1 ratio at 80◦C for 4 hours for the removal of lignin and hemicellulose. Then the solution was centrifuged, and the supernatant was discarded. The precipitate obtained was washed thoroughly with distilled water. The precipitate was again treated with 1:1 aqueous solution of Sodium hypochlorite solution at 80◦C for 15 mins. Then the suspension was filtered, and the residue obtained was rinsed with distilled water several times (Annika Durve Gupta, Tejas Suryawanshi, Vineetha Nair, 2017). The wet cellulose was sun-dried, and 60% moisture was maintained. The image of the cellulose extracted from peanut shells was shown in Fig. 1.

Fig. 1: Cellulose extracted from Peanut Shells.

2.3 Extraction of Beeswax

Beeswax was extracted from the honeycomb. The honeycomb was packed in cheesecloth and heated in 1000ml of water for 1 hr. The filtrate was drained and cooled. The wax that formed on the top of the container was extracted.

2.4 Mould Formation

An equal volume of cellulose and moringa resin were blended well for casting. 2g of the mixture was cast with the mould pressure of 100 psi at 150^0C for 120 secs in a compression moulding machine (Pintiaux *et al.*, 2013), due to the presence of 60% moisture in the cellulose effective binding of the cast was achieved. Then the mould was coated with beeswax. The image of the mould as shown in Fig. 2.

Fig. 2: Biodegradable mould.

3. Characterization

3.1 Test for moisture content

By measuring the weight of the treated sample before and after drying, the moisture content was estimated. The samples were weighed accurately. The moisture content of the cellulose is calculated by

$$\text{Moisture content \%} = \frac{W_1 - W_2}{W_1} \times 100 \qquad (1)$$

Where, W_1 = Weight of the sample before drying and W_2 = Weight of the sample after drying (Abdel-Halim, 2014).

3.2 Fourier Transform Infrared Spectroscopy

FT-IR analysis of raw as well as chemically treated groundnut shells was done to obtain the composition of the sample before and after treatment. Moringa resin and cellulose-resin mixture were also analysed for the composition. The test samples were mixed with KBr to form pellets and spectra were recorded within the spectral range of 4000-400 cm^{-1}.

3.3 X-ray diffraction

The crystalline structure of the cellulose and resin samples was examined by X-ray diffraction. X-ray diffraction (XRD) measurements were performed, using θ-2θ reflection geometry, on a D8 ADVANCE Eco diffractometer using CuKα radiation (λ= 1.54060Å), operated at a generator voltage of 40 kV, a current of 25 mA. Scan range was 5,000 to 10,000 degrees. The Crystallinity index (CI) of samples were determined using the following equation (Trilokesh and Uppuluri, 2019).

$$\text{CI (\%)} = 100 * \frac{A_{crystalline}}{A_{amorphous} + A_{crystalline}} \qquad (2)$$

Where, $A_{morphous}$ is the area under the amorphous curve, and $A_{crystalline}$ is the area under the sample curve.

The cellulose size was determined using Scherrer's equation (Trilokesh and Uppuluri, 2019).

$$\text{Crystal size } L = k\lambda/\beta \cos\theta \qquad (3)$$

Where, λ = 0.1540 nm, k is the correction factor of 0.9, θ = diffraction angle in radians and β = full width at half maximum.

The d spacing of the samples can be determined by using Bragg's law (Trilokesh and Uppuluri, 2019).

$$n\lambda = 2d\sin\theta \qquad (4)$$

where d is the interplanar distance between lattice planes, θ is the scattering angle in degrees, n is a positive integer and λ is the wavelength of the x-ray.

3.4 Hardness testing

The hardness testing for the mould was carried out using the Rockwell hardness test method as defined in ASTM D785, is the most commonly used hardness test method.

3.5 Thickness Measurement

The thickness of the mould is measured using a manual micrometre. Before measuring the thickness, the micrometre was calibrated using standards. The measurement was taken at different random locations and the average values were calculated and reported.

3.6 Thermogravimetric analysis

Thermal stability and decomposition pattern of the Cellulose-Resin mixture sample was characterized using a thermogravimetric analyser (NETZSCH STA 2500). For measurement, approximately 4.2mg of the sample was used. Patterns were recorded under a nitrogen atmosphere by heating the material from 27°C to 350°C at a heating rate of 10 K/min.

3.7 Scanning Electron Microscopy

Morphological investigations were performed on mould cast from Cellulose-Resin mixture by using SEM. The acceleration voltage was kept as 20 kV, and the working distance was fixed to 10 mm. Before the SEM analysis, Samples were layered with gold.

3.8 Biodegradability test

The biodegradability of the mould cast from Cellulose-Resin mixture was observed by burying the mould in the

controlled soil condition. The commercial cellulose-based plastic is used as a control. After a few days, the degradation was identified.

4. RESULTS AND DISCUSSION

4.1 Test for moisture content

The moisture content of the delignified samples was measured using the equation (1) 60% of the moisture was calculated from the weight of the sample before drying 120g and weight of the sample after drying 48g. At 60% moisture, the sample has shown effective bonding during casting so drying was carried out only for 4 hours. The moisture of the sample plays a significant role in the self-bonding mechanism of cellulose which is based on the intermolecular hydrogen bonds (Fahmy and Mobarak, 2013). When the cellulose fibre gets over dried it may lead to the reduction in bonding ability is known as Hornification (Fernandes Diniz, Gil and Castro, 2004). From this, we confirmed the moisture content in the sample helped in effective binding, tensile strength and shelf life of the cast.

4.2 Fourier Transform Infrared Spectroscopy

The FTIR spectra of the Peanut shell, Alkaline treated peanut shell, Moringa resin and Cellulose-Resin mixture were displayed in Figure 3. The difference in intensities of FTIR bands showed that changes in the characteristic absorption patterns to the specific functional groups. The hydrophilic tendency of these samples is reflected in the broad band in between the region 3700-3100 cm^{-1} corresponds to O-H stretching vibrations of the hydrogen-bonded hydroxyl group (Oh *et al.*, 2005; Sun *et al.*, 2005; Morán *et al.*, 2008; Kaushik and Singh, 2011). The peaks at 2929 cm^{-1} are due to the aliphatic saturated C–H stretching vibration of lignocellulosic polysaccharides (Sun *et al.*, 2005; Punnadiyil, Sreejith and Purushothaman, 2016; Trilokesh and Uppuluri, 2019). The peak at 1635 cm^{-1} may be due to the bending mode of the absorbed water (Punnadiyil, Sreejith and Purushothaman, 2016; Trilokesh and Uppuluri, 2019). The peaks at 1513 cm^{-1} and 1433 cm^{-1} in the peanut shell attribute to lignin aromatic rings gradual decrease of these bands in alkaline treated peanut shell spectra reveals the removal of lignin compared to polysaccharides (Sun *et al.*, 2005; Liu and Kim, 2017). The peak at 1735 cm^{-1} in the peanut shell attributed to C=O stretching vibration of acetyl and uronic ester groups of the hemicelluloses or from the ester linkage of the carboxylic group of the ferulic and p-coumaric acids of lignin and/or hemicelluloses but the absence of this signal in the alkaline treated peanut shell spectra indicates the cleavage of the ester bond from lignin and hemicellulose (Sun *et al.*, 2005; Kaushik and Singh, 2011; Punnadiyil, Sreejith and Purushothaman, 2016). The small sharp broad band at 905 cm^{-1} indicates the typical structure of cellulose (due to β-glycosidic linkages of glucose ring of cellulose) in the alkaline treated peanut shell powder (Sun *et al.*, 2005; Kaushik and Singh, 2011; Punnadiyil, Sreejith and Purushothaman, 2016). The absorbances at 1440, 1380, 1320, 1266 and 1052 cm^{-1} in the alkaline treated peanut shell are associated with the typical values of cellulose (Sun *et al.*, 2005). The O-H functional group such as amide alcohols and phenols formed a peak at 3308 cm^{-1} with higher transmittance intensity in the Moringa Resin indicates the presence of epoxy resin. The peak at the 3343 cm^{-1} regions in the cellulose and resin mixture indicates the bonding between the cellulose and resin which forms an intermolecular hydrogen bond.

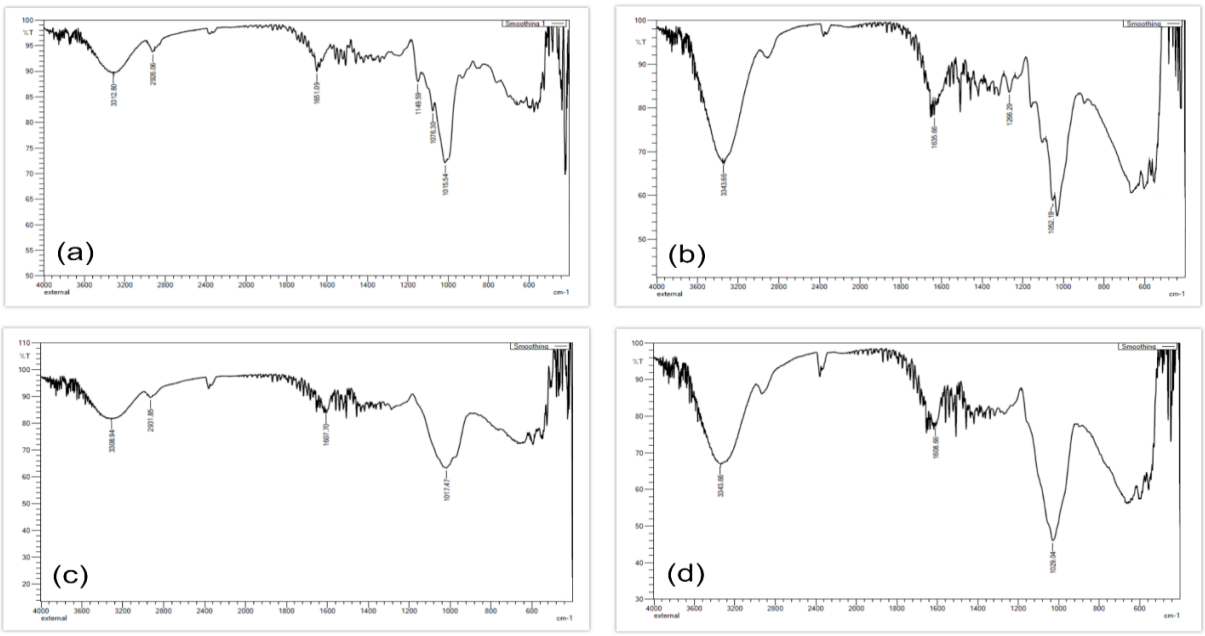

Fig. 3: FTIR spectra of samples (a) Peanut shell (b) Alkaline treated peanut shells (c) Moringa Resin (d) Cellulose-Resin Mixture.

Table 1: Absorption bands for functional groups of cellulose.

S. No.	Functional group	Compounds	Wavenumber cm^{-1}		Reference
			Isolated cellulose	Reported Cellulose	
1	OH	Aliphatic, Aromatic	3343	3375–3340	(Oh *et al.*, 2005)
2	H–C–H	Alkyl, Aliphatic	2929	2925	(Sun *et al.*, 2005)
3	Fibre OH	Adsorbed water	1635	1632	(Sun *et al.*, 2005)
4	C–O–C	Pyranose ring skeletal	1052	1170-1082	(Morán *et al.*, 2008)
5	C-H	Glycosidic linkages between sugar units	905	903-896	(Sun *et al.*, 2005)

4.3 X-ray diffraction

X-ray diffraction patterns of cellulose extracted from alkaline treated peanut shells, Moringa resin and Cellulose-Resin mixture were recorded at different stages shown in the figure. The broad peaks at 2Θ=26.64º, 25º and 24.31º, which indicates the crystalline behaviour of the sample. The samples show the mechanical and thermal properties depending on the crystalline characteristics. Fig. 4a and 4b spectra attributed the presence of crystalline and amorphous material respectively. The crystalline nature of the cellulose extracted from alkaline treated peanut shells is confirmed by the major sharp peak at 26.64º which shows the removal of amorphous hemicellulose and matrix of lignin (Kaushik and Singh, 2011). Fig 4c spectra indicate both crystalline and amorphous structure due to the Cellulose-Resin mixture. The crystallinity was formed due to disintegration in the amorphous region.

Table 2 gives the crystallinity index (CI), Crystal size and interplanar distance for cellulose extracted from alkaline treated peanut shells, Moringa resin and Cellulose-Resin mixture. The crystallinity index (CI) of Cellulose-Resin mixture was found to be 69.43%, it shows the uniform binding of the biopolymers. The smaller crystal size (0.032nm) improves the compatibility of the material which aids in the increment of mechanical and thermal properties of the mould. The interplanar distance values give the well-defined crystalline structure of the samples.

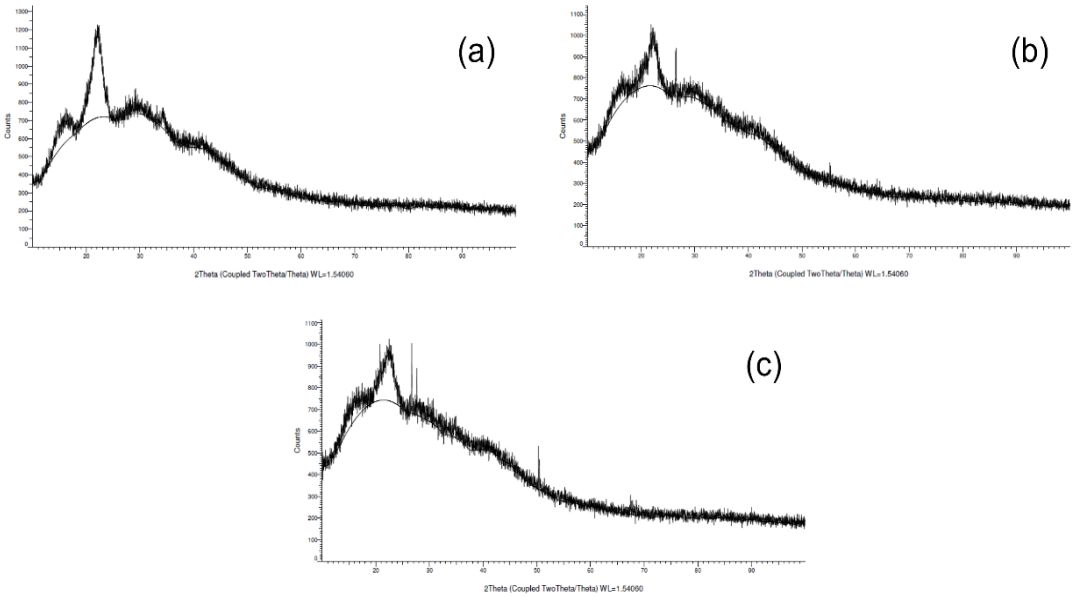

Fig. 4: XRD spectra of samples (a) Alkaline treated peanut shells (b) Moringa Resin (c) Cellulose-Resin Mixture.

Table 2: Crystalline characteristics of samples.

Samples	Crystallinity index (%)	Crystal size (nm)	D- spacing (nm)
Alkaline treated peanut shells	80.05	0.026	0.334
Moringa Resin	65.09	0.023	0.356
Cellulose-Resin Mixture	69.43	0.032	0.365

4.4 Hardness testing

Hardness property of the mould was tested at the L-Scale unit according to ASTM D785. The Rockwell scale determines the depth of penetration of an indenter under a large load compared to the indentation made by a pre-load. In general, the normal range of hardness for plastic is 30-130L. The hardness of the mould which we prepared from the Cellulose-Resin mixture was measured as 65L. This confirmed that the mould has

good hardness strength, ductility and wear resistance where it can be applied in food packaging.

4.5 Thickness Measurement

The thickness of the mould was measured at different places using manual micrometre, and the average was calculated. The average thickness of the mould was formed to be 30mm (3000 microns). The result shows that the prepared mould has a thickness of 3000 microns, hence it can be used for biodegradable plates and cups. Commercially used plastic and biodegradable plates have a thickness around 2500 micron. However, our biodegradable mould will play a vital role in holding capacity and strength for the production of plates and cups.

4.6 Thermogravimetric analysis

Thermal stability of the Cellulose-Resin mixture was determined by using TGA. Various stages of thermal degradation of the sample were observed by the changes in weight. At the initial stage, the sample degraded gradually till 90°C due to evaporation of moisture (Marichelvam, Jawaid and Asim, 2019). In the range of 90°C to 230°C steady-state degradation was monitored due to cellulose polymerization and melting of resin. The final stage of degradation from 250°C to 350°C occurred by the depolymerization of the carbon residues. A small weight loss of 7% was noted at the initial stage below 100°C and there was no decomposition till 230°C which confirms the thermal stability of isolated cellulose and the absence of hemicellulose (Trilokesh and Uppuluri, 2019). The major decomposition rate of 60% was recorded between 250°C to 300°C and a linear range of weight loss was observed after 320°C. From this TGA study, we confirm the sample was stable up to 230° which can be used in various thermal applications.

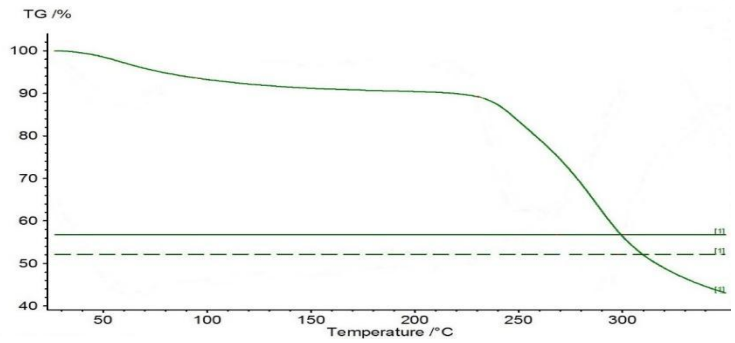

Fig. 5: Thermogravimetric analysis of Cellulose-Resin mixture.

4.7 Scanning Electron Microscopy

An SEM image of the mould cast from the Cellulose-Resin mixture was taken to investigate the morphological structure of these polymers. SEM micrographs of the mould at three different magnifications were shown in Figure 6. The typical structure of the mould in Figure 6(a) shows the bonding between the particles are relatively good and no gaps were visible at the magnification 6000x. The micrographs at magnification 15000x and 60000x show the effective bonding between two different biopolymers where the particles were uniformly dispersed within the matrix (Ghazanfari, 2005).

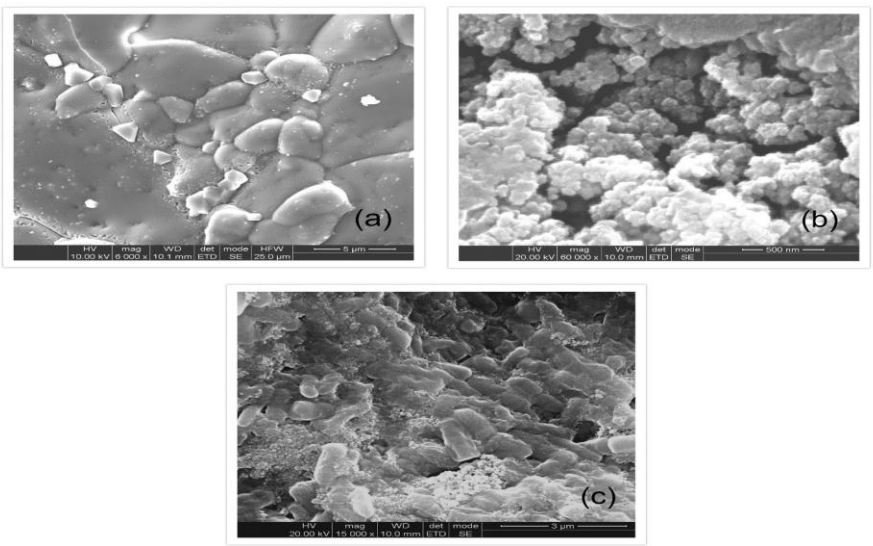

Fig. 6: SEM Micrographs of Cellulose-Resin Mixture at different magnifications.

4.8 Biodegradability test

Biodegradability test has shown positive results after 7 days the mould cast from Cellulose-Resin mixture got reduced in size due to the process of biodegradation by soil microbiome. From figure 7(b) it can be seen samples were broken into pieces. But the control sample showed no changes during the period of degradation. Therefore, we conclude that the mould produced from groundnut shells have a natural biodegradable property which can be used effectively as an alternative for commercial plastics.

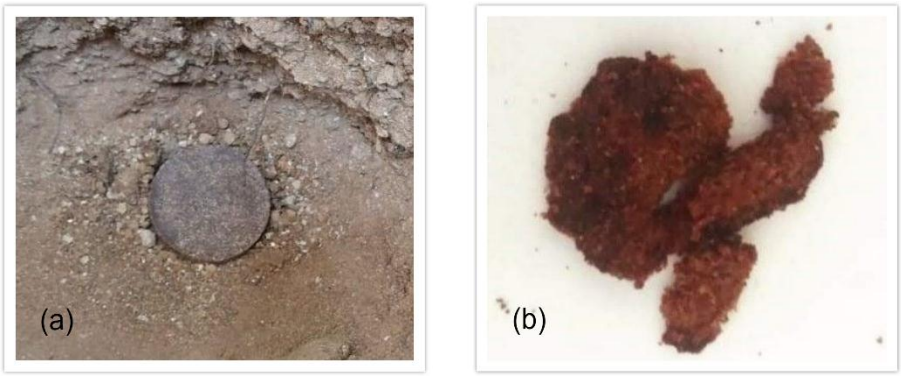

Fig. 7: Biodegradability test of mould (a) Mould before degradation (b) Mould after degradation.

CONCLUSION

In this present study, cellulose was isolated from peanut shells using the alkaline treatment. Moringa resin was added to improve the mechanical property and shelf life of the cast. The constituent of the peanut shells was found to cellulose by characterization study. 60% moisture in the isolated cellulose has shown more effective bonding between the biopolymers. Functional characteristics of the mould were studied by FTIR analysis which shows the intermolecular hydrogen bonding in the matrix. Crystalline characteristics of the samples were studied by XRD. The mould shows good hardness strength with the hardness value of 65L. The thickness of the mould was found to be 3000 microns. TGA of the mould has shown 60% degradation after elevated temperature. Morphological characteristics of the mould was studied from the SEM analysis. The biodegradability test for the mould has shown the best result compared to other commercial plastics since the mould was degradable within a week it strives as an eco friendly product to the society. From these obtained properties of the mould, we suggest that the Cellulose-Resin mixture used in mould casting can be used for the production of biodegradable plates and cups. This can be employed in food packaging applications which acts as an extensive product.

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