



Synthesis and characterization of superabsorbent natural polymers from agro-waste fibres

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This work focuses on synthesizing superabsorbent material from polysaccharides, which are derived from environment-friendly raw materials like ligno-cellulosic agro-waste. Three ligno-cellulosic agro-waste fibres, namely banana, sisal and bagasse, have been taken for the present study, to synthesize natural superabsorbent polymer which are eco-friendly and biodegradable. Cellulose has been extracted from sisal, bagasse and banana, of which cellulose yield has been high for banana fibres. Carboxy methyl cellulose (CMC) synthesized from the extracted cellulose is cross-linked with aluminium sulphate octadecahydrate to subsequently synthesize superabsorbent polymer from these agro-waste fibres. The synthesized CMC from all the three agro-waste fibres shows great reduction in immersion time; CMC from sisal shows the least immersion time of 2.67 s. Banana fibres have recorded the highest degree of substitution. The superabsorbent polymer synthesized from banana fibres has shown an increase in absorbency and swelling rate but reduction in immersion time. This owes to the higher yield of cellulose and the higher degree of substitution attained in the case of banana fibres. This has been proved by chemical composition, SEM and FTIR analyses. This study helps in developing eco-friendly superabsorbent sanitary napkins.

Keywords: Agro-waste fibres, Bagasse fibre, Banana fibre, Natural polymer, Sisal fibre, Superabsorbent polymer

1 Introduction

The global sustainability goals and the increasing awareness about saving the environment and hygienic lifestyles among the public have led to stringent eco-friendly processes and practices in textile industry while product development. Ecological concerns have resulted in a resumed interest in the re-use of agro-waste fibres as environmentally safe alternative¹. Natural fibres are used separately or in blend for various applications in order to reuse the agro-waste fibres.

Composites from jute and hollow conjugated polyester fibre-reinforced nonwoven have been used for developing thermal insulation and sound absorption materials, required for indoor and outdoor building². Sanitary napkins, which are major components of the landfills posing a big threat to a healthy environment, have been a big concern for the researchers. Many researchers have contributed to improve the sanitary napkins development by giving new natural, safe and bio-degradable alternatives for its components.

Organic cotton has been recommended as the top sheet for sanitary napkins due to its non-irritant and superior liquid retention properties. Many agro-waste fibres have been researched for their suitability to be used as the absorbent core of the napkins. Jute fibre has been suitable due to 65-70% cellulose content, high water affinity, shorter lengths suitable for pulp preparation, economic cost etc. Banana fibres are found suitable due to their high absorbency owing to natural porosity. Also, banana fibres are being extracted from the stem which is being discarded after collecting the fruits³. Rekha *et al.*¹ studied another agro-waste, namely *Areca catechu* nut fibres. After extracting from the nut, the fibres have been delignified with laccase enzyme and blended with polypropylene fibres to prepare a nonwoven sheet through fusing method. They have proved its suitability for the use as a coverstock for the sanitary napkins. They claimed that the reduction of synthetic fibre proportion in the products can be considered for sustainability.

Sanitary napkin also has superabsorbent polymer in its absorbent core to enhance the absorption and retention capacity. Most of the superabsorbent materials used are acrylic compounds which are

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synthetic by nature and are used due to their balance between their superior prices to performance. Ma and Wen⁴ opine that superabsorbent polymer is classified under types of hydrogels which can absorb water as high as 500-1500 g/g contrary to the absorption capacity of common hydrogels which does not exceed 1000 g/g. Superabsorbent polymers may be categorized based on their mechanism for water absorption, such as chemical and physical absorption. Generally, superabsorbent polymer (SAP) varieties are white sugar-like hygroscopic materials, which are mainly used in disposable diapers and other applications including agricultural use. SAP as hydrogels, relative to their own mass can absorb and retain extraordinary large amounts of water or aqueous solution⁵. Ai *et al.*⁶ present the fact that as compared to the normal hydrophilic substances, water absorption and water retention capacities of SAP materials are very high and they absorb water even under load.

In their dry state, the long polymer chains are coiled. When they absorb liquid they uncoil and the network expands. The liquid is then stored within the spaces in the molecular network and the material forms a gel which locks the liquid⁷. Superabsorbent polymers can be described as having a network structure and a moderate degree of cross linking⁸. The polymer backbone in SAP is hydrophilic, i.e. 'water loving' because it contains water loving carboxylic acid groups (-COOH). When water is added to SAP there is a polymer/solvent interaction; hydration and the formation of hydrogen bonds are two of these interactions.

Suo *et al.*⁹ opine that these polymers have been extensively used as absorbents in personal care products, such as infant diapers, feminine hygiene products, and incontinence products. Higher performance of the sanitary napkins is directly related to the higher content of superabsorbent polymer. However, the challenges of using superabsorbent synthetic materials are high production cost, high capital and complex machinery, severe health disorders to users and environmental impact while disposing in the landfills due to their non-biodegradability. Consequently, the uses of highly absorbent cellulose based natural fibres in contrast to synthetic SAP are safe and advisable. This is due to the presence of hydroxyl and oxygen containing groups in the fibre cell walls which have high affinity to moisture. The moisture swells the cell wall, and the

fibre expands until the cell wall is saturated with water. Beyond this saturation point, moisture exists as free water in the void structure and does not contribute to further expansion. When a natural superabsorbent polymer is synthesized from such absorbent fibres it is a safer alternative to synthetic SAP. So, the need for developing eco-friendly superabsorbent polymers from natural and sustainable raw materials is imperative. This approach helps to recycle agro-waste fibres which are cheap and most abundantly available renewable organic materials¹⁰.

Zohuriaan-MehrandKabiri¹¹ classify superabsorbent polymers into synthetic (petrochemical-based) and natural. Source of natural materials are the polysaccharide and polypeptide. The use of superabsorbent material in large quantities as a disposable material will cause environmental problems. Therefore, the environment-friendly materials need to be developed. Kiatkamjornwong *et al.*¹² recommended polysaccharide as the cheapest, most abundantly available and renewable organic material available for SAP preparation. Among the various types of polysaccharides, cellulose is the most potential one, since it is the most abundant natural polymer with excellent biodegradability and biocompatibility. All sources of ligno cellulosic materials are good to extract polysaccharides.

However, a considerable amount of research work is being focused on the extraction of cellulose. Extracted cellulose is converted into simple cellulose derivatives carboxymethyl cellulose (CMC). CMC has been synthesized from diverse plant biomasses, which contain 40 – 50% cellulose, 25-40% hemicellulose and 15-35% lignin on a dry basis. The synthesis of CMC from various agricultural waste cellulose sources, such as sugar beet pulp, cavendish banana pseudo stem¹³, cashew tree gum, sago waste, orange peel, papaya peel and Mimosa pigra peel, have been described by many researchers^{14,15}.

The polar carboxyl groups are cellulose soluble, chemically reactive and strongly hydrophilic and so the application of CMC in superabsorbent fields becomes attractive and promising. Although the majority of superabsorbents is nowadays manufactured from synthetic polymers (essentially acrylics) due to their superior price-to efficiency balance¹⁵⁻¹⁷, the world's firm decision for environmental protection potentially support the ideas of partially/ totally replacing the synthetics by "greener" alternatives¹⁸. This study evaluates the feasibility of

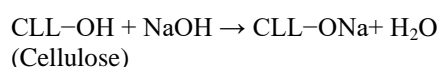
developing biodegradable superabsorbent polymer for the absorbent core, resulting in affordable sanitary pads. The efforts made to develop a eco-friendly super-absorbent material by the synthesis of carboxymethylcellulose from the natural polysaccharide sources of lingo cellulosic fibres, namely banana, sisal and bagasse, and cross-linking with aluminium sulphate octadecahydrate finally culminated in a product, which can be used advantageously in sanitary napkin, are considered to be a valuable contribution. Main advantage of cellulose-based hydrogel over current SAP is that they are environment friendly, biodegradable and has excellent biocompatibility³.

2 Materials and Methods

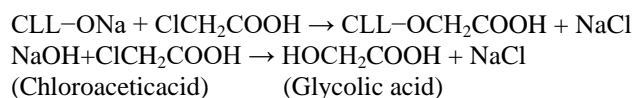
2.1 Synthesis of SAP

Three steps were followed for the synthesis of SAP from agro-waste fibres. Agro-waste lignocelluloses fibres were processed for isolation of cellulose. The cellulose was further processed to convert in carboxy methyl cellulose (CMC). The two main procedures to convert cellulose to CMC are alkalization followed by carboxymethylation¹⁹. The CMC is a soluble biopolymer, odourless cream colored powder; whose degree of solubility depends on substitution of a carboxymethyl group instead of the hydroxyl groups per glucose in cellulose structure^{14,20}. The synthesized CMC was further converted into superabsorbent polymer.

Cellulose is a natural polymer, consisting of glucose units with a uniform chain structure²¹. The cellulose obtained from agro-waste lignocellulosic fibres, was converted to carboxy methyl cellulose by etherification, which involves two steps^{22,23}. In the first step, cellulose was treated with aqueous NaOH followed by reaction with chloroacetic acid, which acts as an initiator, as shown below:



The side reactions that occur are shown in following equations:



2.1 Cellulose Extraction from Agro-waste Fibres

The fibres were cleaned and sun-dried for two days. Dried fibres were cut into small pieces and then grounded into powder. A sample of 25 g of powder

was cooked at 95 °C with 750 mL of 0.5 M NaOH for 2 h with continuous stirring. The dark slurry was obtained, filtered and washed with 1 L of distilled water. The dried cellulose was added with mixture containing 20% (v/v) of nitric acid in ethanol. The mixture was then filtered and washed with cold distilled water until the filtrate did not turn to pink when phenolphthalein and a drop of 0.5 M NaOH were added to it. The residue was dried in an oven at 60 °C overnight to constant weight. Lastly, the dried cellulose was ground and kept in the petri plate for the cellulose modification in the next process. The cellulose was used as precursor for the synthesis of CMC by etherification reaction²⁴. The percent yield of cellulose was determined using the following equation:

$$\% \text{ Yield of cellulose} = \frac{\text{Weight of cellulose}}{\text{Weight of fiber}} \times 100$$

The above procedure was followed for preparation of cellulose from bagasse, sisal and banana fibres.

2.2 Synthesis of Carboxymethyl Cellulose (CMC) from Cellulose

A quantity of 0.5 g of cellulose powder was weighed and added to 150 mL of isopropanol with continuous stirring. Then, 15 mL of 30% w/v NaOH was added drop by drop into the mixture and further stirred for an hour at 27°C (room temperature). The carboxymethylation was initiated when 6 g of mono chloroacetic acid was added with continuous stirring for another 1.5 h. The mixture was covered with aluminum foil and placed into the hot air oven at 60 °C for 3.5 h. The slurry was subsequently soaked in 100 mL of methanol overnight. Next day, the slurry was neutralized with 90% of acetic acid until a pH of 7 was obtained and then filtered using sintered funnel. The final product was washed for three times by soaking in 50 mL of ethanol for 10 min to remove undesirable by-products, and then it was washed again with 100 mL of absolute methanol for the last time. The obtained CMC was filtered and dried at 60 °C to constant weight and kept in a dry place. The percent yield of cellulose was determined using following equation:

$$\% \text{ Yield of CMC} = \frac{\text{Weight of CMC}}{\text{Weight of cellulose}} \times 100$$

The above procedure was followed for the preparation of CMC from bagasse, sisal and banana fibres²⁴.

2.3 Synthesis of super absorbent polymer

2.3.1 Cooking of Corn Starch

Finally, superabsorbent polymer (SAP) was synthesized from CMC. A quantity of 2.4 g corn starch was cooked for 45 min at 75°C in 53 mL distilled water.

2.3.2 Preparation of CMC Solution

Carboxymethyl cellulose (CMC) was mixed with 200 mL of distilled water and kept on a magnetic stirrer for 1 h at 60°C to prepare the CMC solution.

2.3.3 Preparation of Cross-linked Polymer Solution

Later the above prepared CMC solution was added to 697 mL of distilled water in a Hobart mixer followed by addition of cooked starch. The aqueous polymer mixture was mixed for 60 min. Then 0.2 g of aluminium sulphate octadecahydrate was mixed with in 50 mL distilled water and then added to the aqueous polymer mixture. The aqueous polymer mixture was then mixed for 30 minutes to provide a cross-linked polymer gel²⁵. The cross-linked polymer gels from banana, bagasse and sisal CMC were separately poured into the casting tray. The above prepared cross-linked polymer gels, poured in casting tray, were dried at 65°C in a safety oven to prepare a thin SAP film.

2.4 Testing Methods for Super-absorbent Polymer(SAP)

Various tests were conducted for superabsorbent polymers. Testing standards and instruments details are given in Table 1.

2.4.1 Yield Measurement

Product yield was measured, based on dry weight basis. The net dry weight of carboxymethyl cellulose was divided by 5g of cellulose to get the yield value^{26,27}. Following equation was used to calculate product yield:

$$\text{Product yield} = \frac{\text{Weight of dried CMC}}{\text{Dry weight of cellulose}} \times 100$$

2.4.2 Swelling Rate by Vortex Method

Known quantity of distilled water (M) was poured in a beaker and its temperature was adjusted at 30°C.

Table 1 — Tests conducted for super-absorbent polymers

Testing parameters	Testing standards/Instruments
Absorbency and immersion time	MA001-1-diapers-worldwide.com and US patent (5,419,955)
Chemical composition test	AATCC Test Method 20A-2011
Swelling rate	Vortex method
Yield measurement	Calculated values
Degree of substitution	Calculated values
SEM analysis	ZEISS EVO, Carl Zeiss Microscopy

It was stirred at 600 rpm using a magnetic stirrer. Superabsorbent sample was added and the time taken for the SAP to disappear into the fluid was noted with the help of a stopwatch and was recorded as T . The swelling rate (SR, g/g.s) was calculated using the following equation¹¹:

$$SR = (M/a)/T$$

where M is the mass of test solution(g); a , the mass of sample (g); and t , the time to end point (s)

2.4.3 Degree of Substitution

The degree of substitution of synthesized CMC was determined by a sequence of procedures. A quantity of 0.5 g (weight in dry basis) was ignited in a crucible. After cooling, the crucible was then placed into a 500 mL beaker and 250 mL of water was added. Then 35 mL of N/10 sulphuric acid was added by a pipette. It was then boiled for 30 min. The solution was titrated with N/10 potassium hydroxide, and then phenolphthalein was added as an indicator by shaking it gently. The degree of substitution (DS) was calculated using the following relationship:

$$DS = \frac{(162)(A)}{(1000) - (80)(A)}$$

$$A = \frac{af - bf_1}{\text{Wt. of the dry sample (g)}}$$

where A is the alkalinity or acidity; a , the volume (mL) of 10N H₂SO₄ used; f , the factor of 10N H₂SO₄; b , the volume (mL) of N/10 KOH required; and f_1 , the factor of N/10 KOH. When the absolute value is (+), it is alkalinity and when the absolute value is (-), it is acidity¹⁹.

The alkalinity or acidity of the sample may also be measured as follows. One gram of dry CMC sample was measured accurately and dissolved in 200 mL water. Afterwards, 5 mL of N/10 sulphuric acid was added by a pipette and the solution was boiled for 10 min. After cooling, it was titrated with N/10 potassium hydroxide (5mL) using phenolphthalein as the indicator. A blank test (without CMC) was pursued with the same time and procedure (B mL) and the following equation was used for calculation:

$$(B-S)(f_1)$$

$$\text{Alkalinity or acidity} = \frac{\text{Wt. (g) of sample}}{\text{Wt. (g) of sample}}$$

Here also, when the absolute value is (+), it is alkalinity and when the absolute value is (-), it is acidity.

3 Results and Discussion

3.1 Analysis of Chemical Composition of Raw Fibres, Extracted Cellulose and Synthesized CMC

Table 2 presents the estimated chemical composition of the three ligno-cellulosic fibres selected, viz banana, sisal and bagasse in raw stage, extracted cellulose stage and during the synthesized CMC stage.

The results show the removal of lignin during cellulose stage itself and the gradual increase in the cellulose content. Not much difference is observed in the cellulosic content, due to the fact that CMC is just a conversion of cellulose. The cellulose content is highest in the case of banana fibres, followed by sisal and finally bagasse.

3.2 Measurement of Cellulose and CMC Yield

Figure 1 shows the yield of cellulose from raw fibres and the yield of CMC from cellulose. While analysing the cellulose extracted from raw fibres, it is found that the banana has given the best cellulose yield as compared to sisal and bagasse. But, while analysing the yield of CMC from cellulose, we observe that bagasse has given the maximum yield followed by banana and the least in the case of sisal.

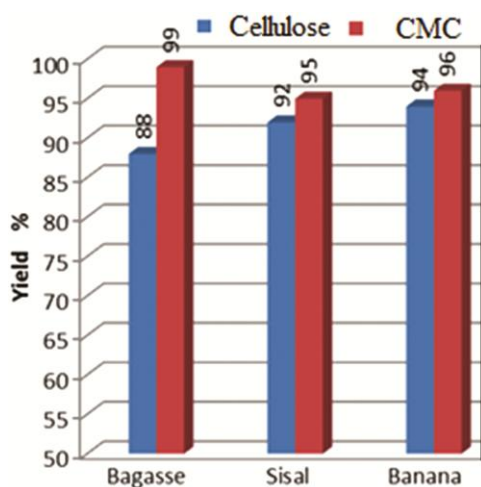


Fig. 1 — Yield of cellulose and CMC

3.3 Absorbency and Immersion Time for Banana, Sisal and Bagasse

Figures 2 and 3 show the immersion time and absorbency of all three fibres in raw stage, extracted cellulose stage and during the synthesized CMC stage. While comparing the immersion time of the raw fibres, it is found that the sisal has the highest followed by bagasse and banana fibres; banana's immersion being the best preferred. Various stage from raw, to cellulose and then to CMC stage, it is found that the immersion time has reduced and the same order is maintained in all the three stages. While comparing the CMC stages, the immersion time has been recorded as 2.67, 3.33 and 5.33 for sisal, banana and bagasse respectively.

Due to the conversion of cellulose to CMC, the fibres have opened up and resulted in better absorbency. This proves the slow transition of raw fibres towards the synthesis of superabsorbent polymer. In the raw stage, bagasse has the highest absorbency due to the presence of pith. Next highest absorbency is recorded by banana, which is a finer fibre than sisal. In the CMC stage, the banana CMC has recorded the highest absorbency followed by bagasse and finally sisal. The trend of improvement in immersion time (Fig. 2) and absorbency% of the three fibres (Fig. 3) proves the slow transition of raw fibres towards the synthesis of super absorbent polymer.

3.4 Degree of Substitution

It is observed that the degree of substitution values of synthesized CMC from bagasse, sisal and banana

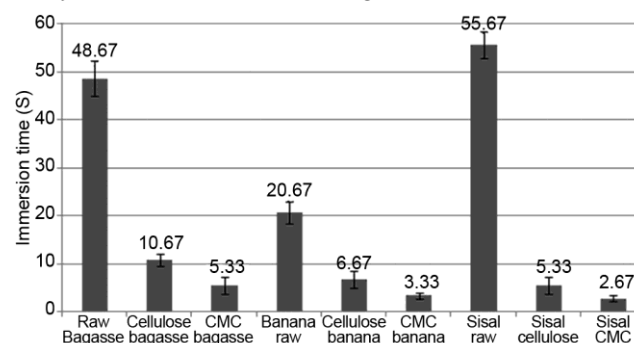


Fig. 2 — Immersion time of banana, sisal and bagasse fibres in raw, cellulose and CMC stages

Table 2 — Chemical composition of banana, sisal and bagasse fibres in raw stage, extracted cellulose stage and during the synthesized CMC stage

Fibre stages	Bagasse			Banana			Sisal		
	Cellulose	Hemicellulose	Lignin	Cellulose	Hemicellulose	Lignin	Cellulose	Hemicellulose	Lignin
Raw fibre stage	42	31	17.67	59.67	26.33	11	50	16	10
Cellulose stage	65.33	31	0.02	71	29.33	0.02	66.33	29.67	0.02
CMC stage	68	30	0.02	73	20	0	70	28	0.02

are 0.56, 0.69 and 0.99 respectively. CMC has a commercial value with higher value of DS to range between 0.4 and 1.4. Also, the higher the DS value, the better will be the performance. Banana has recorded the highest degree of substitution, which confirms the suitability of banana CMC for further synthesis of superabsorbent polymer.

3.5 SEM Analysis

Figure 4 presents the SEM images of cellulose and CMC extracted from bagasse, banana and sisal. Decrease in diameter has been observed among the raw, cellulose and CMC stages of all the three fibres, namely bagasse, banana and sisal. The size reduction

in the cellulose stage is mainly attributed to the separation of the fibre's primary cell wall due to the removal of lignin and hemicelluloses.

An important consequence of diameter reduction is the increase in aspect ratio (L/d , where L = length and d = diameter), which provides higher reinforcing ability of the cellulose for CMC. Most particles displayed a diameter and aspect ratio in the range of (banana) 100 - 150 μm and (bagasse and sisal) 20 - 25 respectively. The size of cellulose is reduced from 20-25 μm to 13-10 μm , which clearly shows the removal of hemicellulose and lignin.

All the SEM images of CMC, the polymer synthesized from all three fibres, has the characteristic porous structure. These pores act as water permeation and show interaction sites for external objects bearing copolymers with hydrophilic groups. Figure 5 presents the SEM images of superabsorbent polymer synthesized from the CMC of banana fibres. Though the size of the fibres in the synthesized SAP still ranges between 10.36 μm and 12.61 μm , the SEM image presents the characteristic cross-linked structure.

3.6 Swelling Rate

The swelling rate of banana CMC is found highest as compared to commercial CMC. Swelling ratios of banana, sisal and bagasse SAP are 0.5, 0.55 and

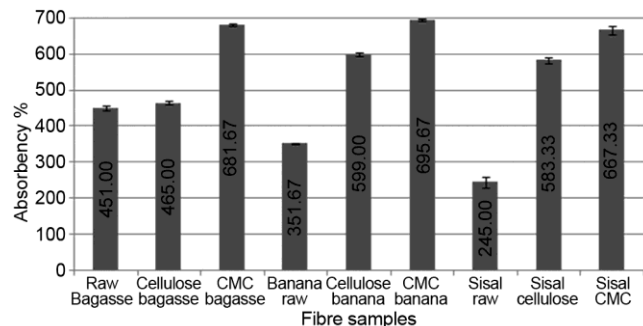


Fig. 3 — Absorbency % of banana, sisal and bagasse fibres in raw, cellulose and CMC stages

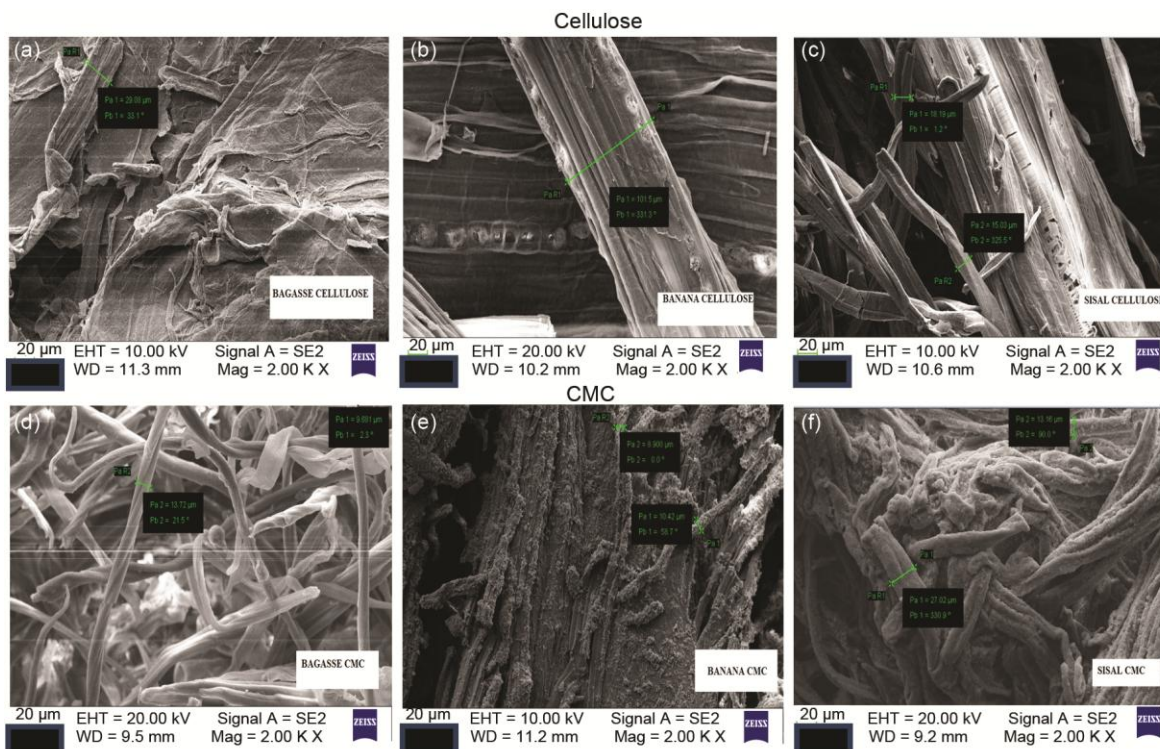


Fig. 4 — SEM image of (a) bagasse cellulose, (b) banana cellulose, (c) sisal cellulose, (d) bagasse CMC, (e) banana CMC and (f) sisal CMC

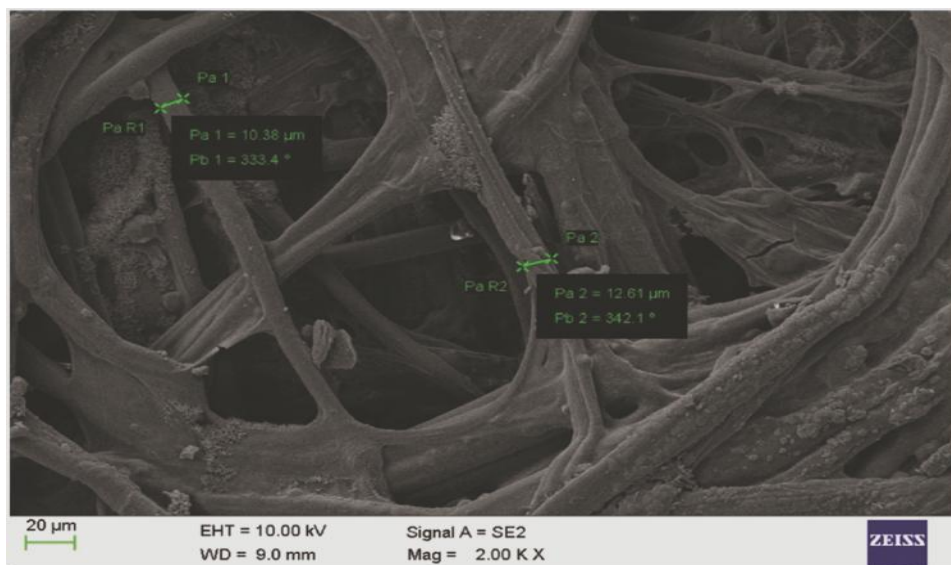


Fig. 5 — SEM results of banana SAP

0.6 % respectively. While comparing all the three synthesized SAP varieties, it is found that SAP from banana has a film like linked structure, but those from sisal and bagasse still maintain powder form. The swelling behaviour of the SAP synthesized from the commercial CMC is much lower than those of the SAP synthesized from the agro-waste fibres. Among the agro-waste fibres, though the cross-linking has not occurred in the case of bagasse and sisal varieties of SAP, the swelling behaviour has been higher, with bagasse SAP having the highest, closely followed by that of sisal.

4 Conclusion

The superabsorbent polymers developed from banana, bagasse and sisal fibres, are found to have improved in terms of absorbency as evidenced from the experimental results. The cellulose yield has been high for banana fibres followed by sisal fibres and the least for bagasse fibres. Sisal CMC shows the least immersion time of 2.67 and the maximum of 55.67 is recorded by sisal raw fibres. The synthesis of CMC has shown a great reduction in immersion time in all the three agro-waste fibres, namely bagasse, sisal and banana. The absorbency% has been high for CMC from banana fibres, but the increase in the absorbency between raw and CMC stages has been higher in the case of sisal fibres. Banana fibres have recorded the highest degree of substitution, and the least by CMC from bagasse fibres. The SAP synthesized from banana fibres has shown an increase in absorbency, swelling rate and reduction in immersion time. This

owes to the higher yield of cellulose and the higher degree of substitution attained in the case of banana fibres. This has been proved by chemical composition, SEM and FTIR analyses.

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