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HYDROGEL SYNTHESIS FROM CELLULOSE DERIVATIVES OBTAINED FROM *YUSHANIA ALPINA* BAMBOO FOR WOUND DRESSING APPLICATIONS

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Abstract

The extraction of cellulose was obtained from the bamboo plant *Yushania alpina,* a native species of Ethiopia, and synthesizing of hydrogel from carboxymethyl cellulose, and cross-linked polyethylene glycol with different concentrations for medical application in chronic wound dressings. New cellulose was prepared from a bamboo plant in different mesh sizes powder. Optimization of cellulose has been done by using different techniques like double and single string alkalization treatment recovering 45.5% of cellulose. Carboxymethylcellulose was synthesized from cellulose and monochloroacetic acid by the process called carboxymethylation or etherification. Prepared carboxymethylcellulose is water-soluble, unlike native cellulose. Fourier transformed infrared spectroscopy (FT-IR), and x-ray diffraction (XRD) characterization techniques were employed to investigate the functional group and structure of processed cellulose. Additionally, the moisture content of cellulose was analyzed by using a moisture analyzer and porosity test has been implemented. The hydrogel was synthesized from carboxymethyl cellulose (CMC) and with different concentrations of cross-linker polyethylene glycol (PEG). 15 and 20 ml of polyethylene glycol were used to enhance the properties of the hydrogel. Hydrogel has been characterized by FT-IR and XRD techniques. The water absorption capacity of hydrogel was also investigated.

Keywords: Wound Healing, Cellulose, Carboxymethyl cellulose, Cross-linker Hydrogel, Porosity test, Moisture analysis, FT-IR and XRD analysis, porosity test and water absorbing capacity.

1. Introduction

Wound healing is a complicated process involving various interactions between growth

factors, extracellular matrix, and cells (Aarabi et al., 2007; Capanema, Mansur, de Jesus, et al., 2018). If tissue integrity and homeostasis are to be recovered in the seconds after an injury, numerous intracellular and intercellular pathways must be engaged and coordinated. Acute wounds are normal to regenerate tissues, but chronic wounds are difficult to regenerate injured tissues in the usual way (Huang et al., 2022; Jirawitchalert et al., 2022). In addition, immune system cellular components, the blood coagulation cascade, and inflammatory pathways are all activated. Immune cells (neutrophils, monocytes, lymphocytes, and dendritic cells), endothelial cells, and other gene expression and phenotypic alter dramatically in cells. Keratinocytes, and fibroblasts, results in cell proliferation, differentiation, and migration (Aarabi et al., 2007).

The wound healing process requires growth factors to eliminate microbes and regenerate damaged tissues. The growth factors can easily migrate throughout the wound area if the wound keeps under moisture conditions. The dressing shields the injury and aids in the regeneration of dermal and epidermal tissues during the wound-healing process, (Capanema, Mansur, Mansur, et al., 2018). Hydrogels are playing a vital role to keep the wound in moisture condition. Bamboo is a grass that belongs to the Poaceae family, which has over 1200 varieties and grows in tropical and subtropical climates around the world (Nigatu et al., 2020). Ethiopia has two native bamboo species: highland bamboo *Yushania alpina* and lowland bamboo *Yushania alpina* and *Oxytenantheria abyssinica* (Desalegn & Tadesse, 2014). The growth of the bamboo stand is marked by an increase in length, the number of culms, and biomass (Mulatu et al., 2013), as well as an increase in weight and length from the underground rhizome system and the above-ground culms.

Cellulose is a polysaccharide carbohydrate molecule, which is made up of carbon, hydrogen, and oxygen. A long chain of D-glucose linked by a β 1,4-glycosidic bond provides cellulose. Cellulose is insoluble in water due to hydrogen bonds and requires a high temperature for decomposition (Berndes et al., 2003), but it is the largest and most renewable source of environment-friendly raw material (Kabir et al., 2018). Cellulose meets for the production of green and biobased products (Dara et al., 2021). Cellulose and its derivative are used for the synthesis of hydrogel for different applications such as diapers, cosmetics, corneal, cartilage and contact lenses, water reservoirs in agriculture (Chang & Zhang, 2011; Klein & Poverenov,

2020). Wound dressing, drug delivery, and tissue engineering (Carvalho & Mansur, 2017; Mogoşanu & Grumezescu, 2014) Civil engineering (Fitrah et al., 2019). Hydrogel materials have a number of incredible characteristics like biodegradability, porosity, biocompatibility, eco-friendly, high water absorbance capacity, renewability, and Good mechanical strength (Ahmed, 2015), and all of these properties enable the hydrogel to be highly unique in various applications. Due to the presence of hydrophilic groups in their polymer networks, such as NH_2-OH, COOH, and SO_3H, hydrogels continue to absorb water and swell to form 3D structures. Physical or chemical crosslinking helps prevent hydrogels from dissolving in the solvent and allows them to maintain an unmodified 3D structure during swelling (Rizwan et al., 2017). Solid molded shapes, pressed powder matrices, microparticles, coatings, membranes or sheets, encapsulated solids (in osmotic pumps), and liquids (that form gels upon heating or cooling) are all examples of hydrogels (Husain et al., 2018).

Hydrogels synthesized from natural biopolymers such as cellulose, cellulose derivatives, chitin, and chitosan by using the methods of physical crosslinking and chemical cross-linking have more advantages in the aspect of renewable (Liu et al.,2016), non-toxicity, biodegradability low cost, and biocompatibility (Kabir et al., 2018; Kabiri et al., 2003). Physical cross-linking interaction is based on ionic and hydrogen bonding whereas chemical cross-linking interaction is based on covalent bonding. The biopolymer can be categorized into two groups, Natural biopolymer, and synthetic biopolymer, and based on this classification hydrogels are also classified as hydrogels synthesized from natural biopolymer and synthesized biopolymer. Natural-based hydrogel is preferable because of its biodegradability, low cost, and biocompatibility. Synthetic-based polymeric hydrogel is more difficult than natural polymeric based in terms of techniques, sources, and applicability. Hydrogels that have a high swelling capacity can use for personal hygiene and agriculture (Enawgaw et al., 2021). The objective of this project is to synthesize hydrogel from cellulose derivatives.

2.Materials and methods

2.1. Sample Collection

Yushania alpina Bamboo sample was obtained from Bio and Emerging Technology Institute (BETin), Addis Ababa, Ethiopia. Sieve Analyzer, Grinder, 1M, 2.5M, 3M, and 10% sodium hydroxide (NaOH), 10% potassium hydroxide (KOH) Hydrogen peroxide (H_2O_2), Ethanol, Sulfuric acid (H_2SO_4), moisture analyzer, Fourier transform infrared (FTIR) spectroscopy, X-

ray Diffractometer.

2.2. Extraction of cellulose from bamboo plant

The bamboo sample was ground into powder by using a grinder machine. The obtained powder sieved with the help of a 30, 60,100,180, 250 and 325 mesh size analysers. 1M, 2.5M and 3M NaOH aqueous solution was prepared. 10% potassium and NaOH were prepared as a belching agent. 25g bamboo powder was mixed with 750ml of NaOH in the ratio of 1:30 W/V and stirred for 2 hours at 90℃. The sample was filtered and washed with 1 litter of distilled water. The obtained cellulose cake was dried at 50℃ for 8-12 hours using the oven. The process should be carried out for 2 cycles (Liew et al., 2015)

2.3. Whiting

Bleaching was done using 10% NaOH and 7.5% H_2O_2 at 1:10 (W/V) molar ratio of cellulose to bleaching solution in g/ml. the bleaching agents were added at (1:1) v/v molar ratio and then the mixture was kept at room temperature for 30 min followed by heating at 70°C in the water bath for 30 minutes. Finally, after filtration washing was done warm distilled water and dried at 60°C for 8 hrs the cellulose was extracted from the bamboo tree. (Wang & Zhao, 2021).

2.4. Alkalization

5gm of the extracted cellulose was weighed and dissolved in 100ml of distilled water in 250 ml flask. 10 ml of 30% NaOH was added in a dropwise into the flask. Then the mixture was kept in shaker incubator for 1hr at 25°C (Abdul Hameed et al., 2020).

2.5. Carboxymethylation or Etherification

5g of monochloroacetic acid was weighed and added to the previously prepared mixture and the mixture was heated at 65°C for 3 hrs. The sample was then filtered and soaked for 24 hrs. with 100 ml Methanol. Next, the sample was neutralized using glacial acetic acid. And finally, it was filtered again and dried in oven at 60°C for 8hrs (Abdul hameed et al., 2020).

2.6. Synthesis of (CMC/CA) Hydrogel

2 gm of synthesized Caroboxymethylcellulose (CMC) was weighed and added into 100 ml of distilled water. The solution was continuously stirred at room temperature until it was

homogenized. Citric acid (10, 15, 20, and 25%) was added in in the W/W of CMC and homogenized for 20min. 10 ml of solution was then poured into a 60 mm diameter of the petri dish, and allowed for 24hrs at 40℃ to remove the water. Then the sample was heated for 24 hrs at 80 ℃ let for cross-linking reaction (Capanema et al., 2018a).

2.7. Synthesis of CMC: PEG hydrogels

2 gm of the synthesized CMC was weighed and added into 250 ml of beaker. Then 100 ml of distilled water was added into it. The solution was continuously stirred at room temperature until it was completely dissolved. 10ml, 15ml, 20ml, and 25ml of PEG was added into the solution and stirred for 20 min until it is homogenized. After that 10 ml of solution was poured into a 60 mm diameter of the petri dish, and allowed for 24hrs at 40℃ to remove the water. Then the sample was heated for 24hrs at 80℃ to let for cross-linking reaction. (Capanema et al., 2018b).

2.8. Water absorption capacity of the hydrogel

The water absorption capacity of the obtained hydrogel was measured by weighing the hydrogel sample using a weighing balance. Put it on aluminium foil and distilled water was added to the hydrogel and let it be absorbed for some time. Later on, removed excess water from the surface of the hydrogel and weighed the swelled hydrogel again. Then water absorption was calculated using the following formula:

$$\text{Water absorption (\%)} = [(W_2 - W_1)/(W_1)] * 100\%$$

Where, W_1 - the initial weight of the hydrogel,

W_2 - final weight of the hydrogel (after immersion in water)

2.9. Porosity of the hydrogel

Porosity of the hydrogel was measured by the concept of solvent replacement method. Weighed discs of the hydrogel and placed/immersed in absolute ethanol for 24 hrs. When the hydrogel immersed into the ethanol, there was a change in volume and that volume taken/considered as the volume of disc hydrogel. Then discs removed of the ethanol and blobbed with blotting paper to remove extra ethanol present on the surface of the hydrogel disc and weighed again. Porosity of the hydrogel was calculated using the formula given below.

Porosity =$[(M_2-M_1)/(\rho V)] * 100\%$

Where, M_1 and M_2- weight of the hydrogel disc before and after ethanol immersion,

V - volume of hydrogel disc in the solvent,

ρ - density of absolute ethanol (798 kg/m^3)

3. Result and Discussion

3.1. Extraction and optimization of cellulose

Cellulose was extracted from the bamboo plant by using different organic solvents and belching reagents. The basic solvent NaOH with different concentrations (1M, 1.5M, and 3M) has been involved to dissolve cellulose. Additionally, 7.5% H_2O_2 and 10% NaOH have been used as a belching reagent. There are some parameters that can affect the extraction of cellulose such as temperature, stirring rate, string time, concentration ratio of bamboo sample and solvent, and mesh size of the bamboo powder. Different mesh size bamboo powder has been used during the production of cellulose. The selection process of mesh size to give optimum cellulose yield was a very tedious process. After multi string processes have implemented, it has been observed that 60 mesh size bamboo powder gives a better yield of cellulose than others. 45.5 % yield of cellulose has been produced from 5g of 60 mesh size (250 microns) bamboo powder. The standard cellulose value is found to be in the range of 40-60 % yield.

Table 1:Effect of NaOH concentration on cellulose production.

Amount of bamboo powder used in (gm)	Concentration of NaOH used in Molar(M)	Yield of cellulose produced in percent (%)
5	1	38
5	1.5	40
5	3	45.5

Single stirring method of extraction was applied to separate hemicellulose and lignan from cellulose. 5g of bamboo powder was measured by using balance and added to a 250 ml beaker. An aqueous solution of NaOH (150 ml) was measured based on a 1:30 W/V ratio and added to

a 5gm sample containing beaker then stirred at 90^0C for 2 hours. After 2 hours of the stirring process, the impurities (hemicellulose and lignin) were separated by the filter sheet. The extracted cellulose was washed and dried for 24 hours in an oven at 50^0C. The stirring process is one of the parameters to get the optimum yield product of cellulose.

In the double stirring process, more qualitative cellulose was obtained. If impurities are left during the single stir process, it is better to do again the process to eliminate hemicellulose and lignin impurities from cellulose. During the double stirring extraction process, 5g of 60 mesh size bamboo powder was measured by using an electrical balance.150ml of aqueous sodium hydroxide was added to a 250 ml conical flask with 5g of bamboo powder, the sample and the solvent stirred at 90℃ for 2 hours. After 2 hours, hemicellulose and lignin impurities have been filtered by using the filter sheet. Cellulose cake was dried at 50℃ for 24 hours in the oven and repeated the process. By comparing the percentage yield of cellulose for both single stirred and double stirred extraction methods using 3M NaOH it has been observed that the optimum cellulose was produced in the double stirring extraction process. In conclusion, the double stir process is preferable for the quantitative and qualitative production of cellulose yield.

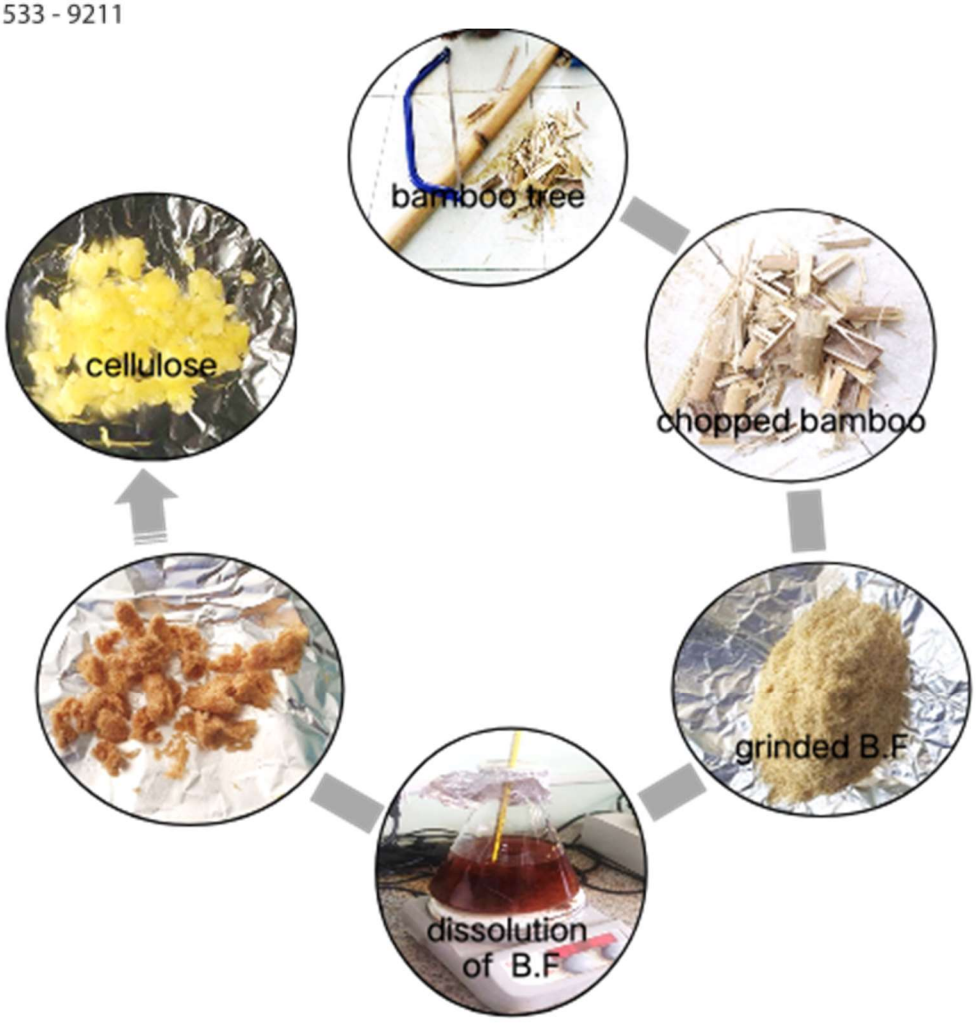


Figure 1: Production of cellulose

3.2 Moisture content analysis of cellulose.

The moisture content of the extracted cellulose was measured using a moisture analyser. The temperature of the moisture analyser was 120℃. Cellulose, which extracted from different mesh size bamboo samples has different moisture content. Table 2 shows the moisture content of processed cellulose. 1gm of extracted cellulose was measured for moisture analysis.

Table 2:Moisture content of extracted cellulose in precent.

Bamboo powder (g)	% Yield	Moisture content in (%)
5	38	7.2
5	37	7.64
5	45.5	7.5
3	33.8	8.22
25	42.72	7.22

Moisture content analysis is very important process to know the change of cellulose structure after water absorption. During moisture content analysation of processed cellulose value found to be in the range of 7.22 to 8.22%. According to (Liu et al., 2020) work the moisture content of cellulose was in the range between 1.29 to 4.39%. Based on the comparison moisture content of processed cellulose from bamboo sample is higher than the moisture content of the previous work. Table 2 shown the moisture content of extracted cellulose from bamboo plant. Based on table-2 information, it has been observed that 8.22 is the highest moisture content. The moisture content controls the product quality and physical properties of all substances and materials at all stages of processing and final product existence (Liu et al., 2020).

3.3. Production of carboxymethyl cellulose

After the first part (extraction of cellulose) has done, synthesis of CMC was performed. 5.0 g of monochloroacetic acid was then Weighed to previously prepared mixture and the mixture was heated at 65°C for (2-3) hrs. this step can be done using an oven by setting power at 6 and heating for 2 min. after that the sample was filtered and soaked for 24 hrs. with 100 ml Methanol. Next, the sample was neutralized using glacial acetic acid. And finally, it was filtered again and dried in oven at 60°C for 8hrs. Fig.2 shows produced CMC from cellulose. CMC has been produced by etherification method and it is soluble in water unlike native cellulose (Zainal et al., 2021). The replacement of hydroxyl group of native cellulose to carboxymethyl group has a vital role to enhance water holding capacity of the hydrogel.

Figure 2:synthesised carboxymethylcellulose (CMC)

Figure 1: CMC powder

3.4 Synthesis of hydrogel from CMC and cross-linker PEG with 15- and 20-ml content and agar.

The 2gm of CMC was measured and dissolved in 100 ml of distilled water with continuous string process. Different concentration (10,15,20 and 25%) of citric acid were added in W/W of CMC and homogenized for 20 minutes. 10 ml of solution was poured into 60 mm diameter of the petri dish, then it was allowed for 24 hours at 40℃ to remove the water. The sample was heated for 24 hours at 80℃ for cross-linked reaction. Fig 5 shows the synthesis of hydrogel from carboxymethylcellulose and 15 and 20 ml of cross-linker polyethylene glycol. PEG is very important cross-linker and network modifier during the production of supper water absorbent hydrogel for wound dressing application. PEG can dissolve in water and organic solvent(Capanema et al., 2018).

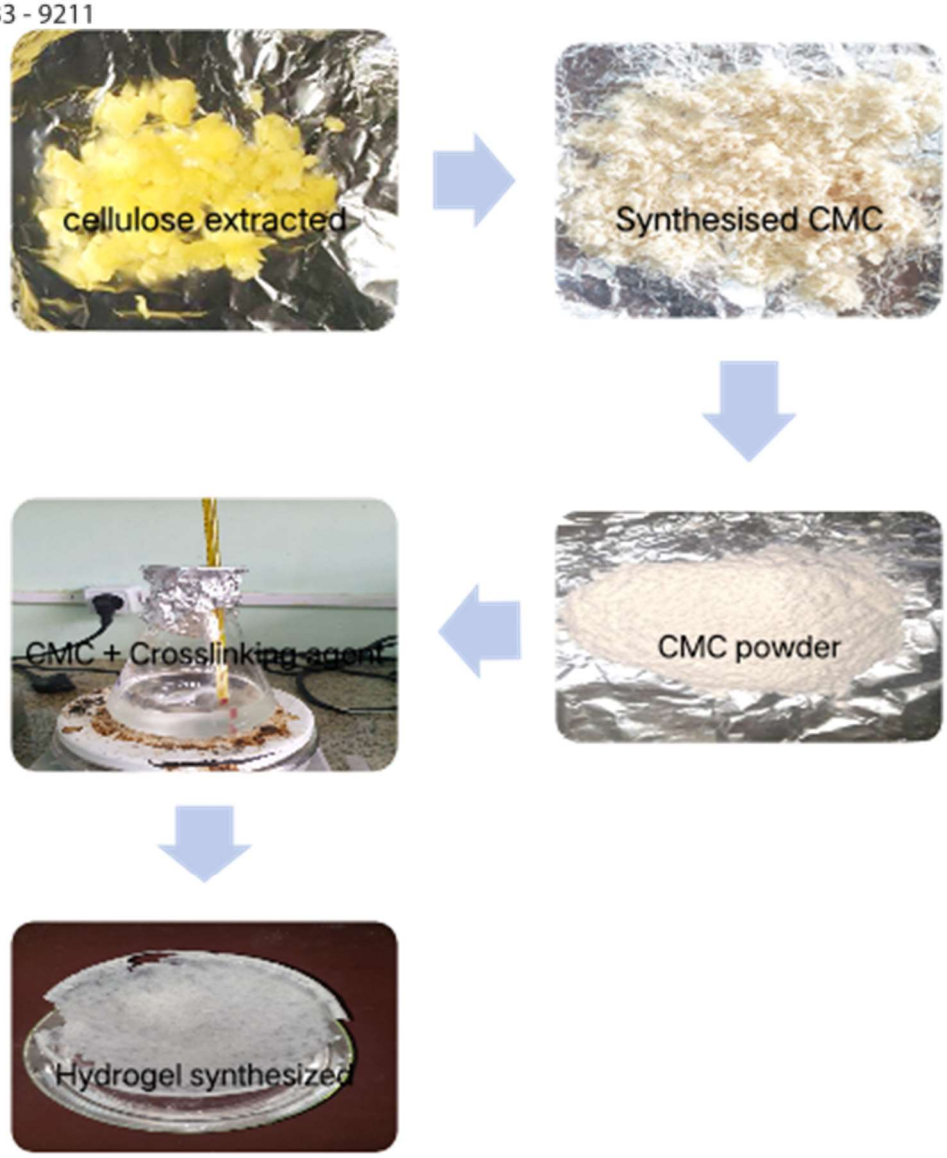


Figure 4:schematic diagram showing method of production of CMC-based Hydrogel

Cellulose, which extracted from bamboo sample was used as raw material to produce environmentally friendly hydrogel. As shown in fig 4 Cellulose has been produced from Ethiopian highland bamboo (*Yushania alpina)* plant by using NaOH solvent. Extracted cellulose OH functional group has modified by carboxymethyl to enhance the properties of cellulose. CMC is soluble in water and it has various applications.

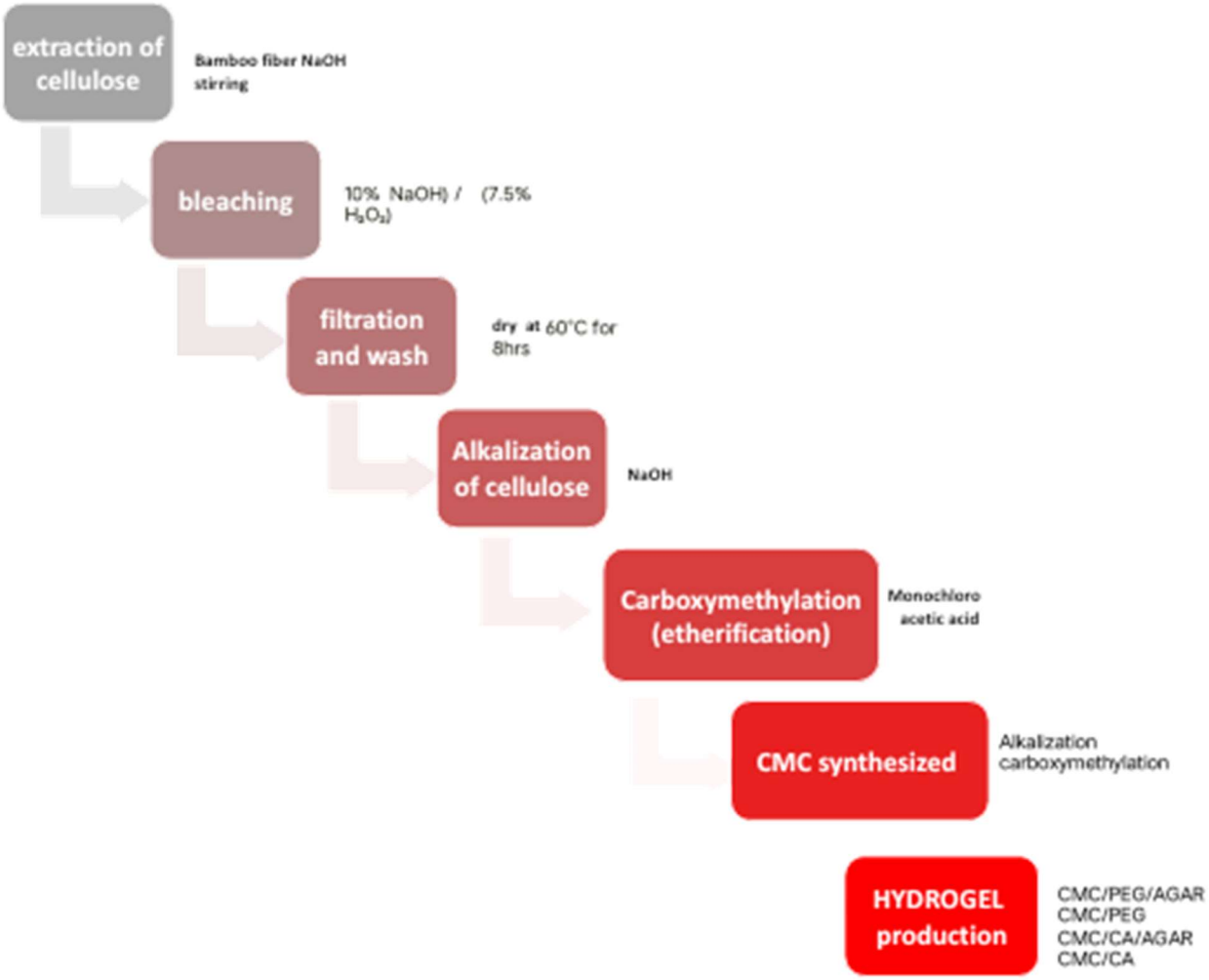


Figure 5:Schematic diagram showing general methodology of Hydrogel production from Bamboo fibre

4. Characterizations

4.1. Fourier transform infrared analysis of Cellulose.

FTIR analysis can be used to determine the chemical structure of compounds and to establish the identity of organic and inorganic materials (functional groups). The fingerprints of the functional groups obtained for cellulose are labelled in fig 6 and they were extremely comparable to the absorbance observed for standard cellulose. The C-H rocking vibration, C-O stretching vibration, and symmetric CH_2 bending vibration, respectively, were connected with the absorbance bands at around 895.01, 1018.46, and 1155.41 cm^{-1}, where these peaks are referred to cellulose form of the carbohydrates. Aside from the peaks mentioned above, the peak at 2885.63 cm-1 corresponds to the stretching O-H groups and the peak at 3279.13 cm^{-1} corresponds to the stretching vibration of the C-H groups. Furthermore, the peak at 3,325.42 cm^{-1} is allocated to the O-H stretching vibration of the hydroxyl groups. based on the results, it was clearly confirmed that the extraction of cellulose from bamboo plant was successfully accomplished. Choi, M. et al., has also said that the C-H rocking vibration, C-O stretching vibration, and symmetric CH2 bending vibration were connected with the absorbance bands at around 898, 1,057, and 1,430 cm^{-1}, respectively, where these peaks are referred to cellulose form of the carbohydrates. Aside from the peaks indicated above, the peak at 1,640 cm^{-1} belongs to the stretching O-H groups and the peak at 2,900 cm^{-1} refers to the stretching vibration of the C-H groups. Furthermore, the peak at 3,410 cm^{-1} is allocated to the adsorbed water and is typical of the O-H groups. As a result of the findings, it was determined that the extraction of cellulose from wastes of Graviola (*Annona muricata*) leaf had been completed effectively which is comparably same with our research finding.

4.2. FTIR analysis of Carboxymethylcellulose (CMC).

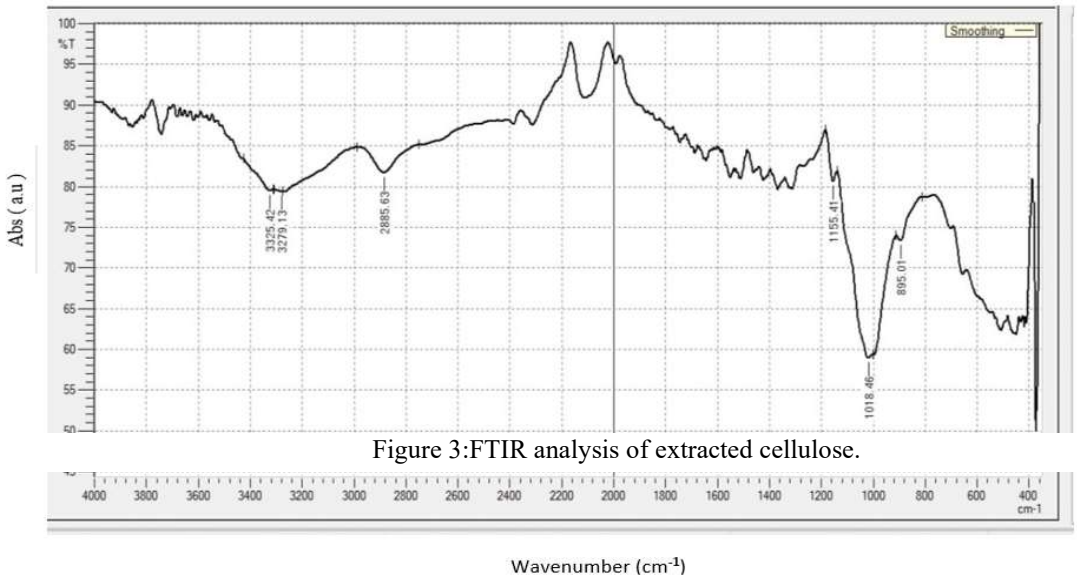


Figure 3:FTIR analysis of extracted cellulose.

The CMC samples were analysed by FTIR which is shown in Fig 7. The x-axis represents the wave number (cm^{-1}) and y-axis shows the light transmittance through the sample (absorbance). The graph shows the FTIR spectrum of CMC from 400 to 4000 cm^{-1}. The characteristic transmission band at 3473.95 cm^{-1} shows the hydrogen bonding OH stretching region. The small hump at 3065.02 cm^{-1} shows the attributable to C-H stretching vibration. The peak observed at 1683.93 cm^{-1} confirms the presence of COO- is assigned to stretching of the carboxyl group. Hydrocarbon groups ($–CH_2$ scissoring) was shown at 1425.46 cm^{-1}. The band observed at 1307.79 cm^{-1} and 1058.97 cm^{-1} are assigned to OH stretching in-plane and C-H stretching in symmetric (primary alcohol) of CMC. The bands at around 650- 750 cm^{-1} is due to the deformation vibration of hydrogen bonds. Based on the spectra it is believed that the cellulose has undergone carboxymethylation and CMC has been successfully synthesised (Saputra et al., 2014).

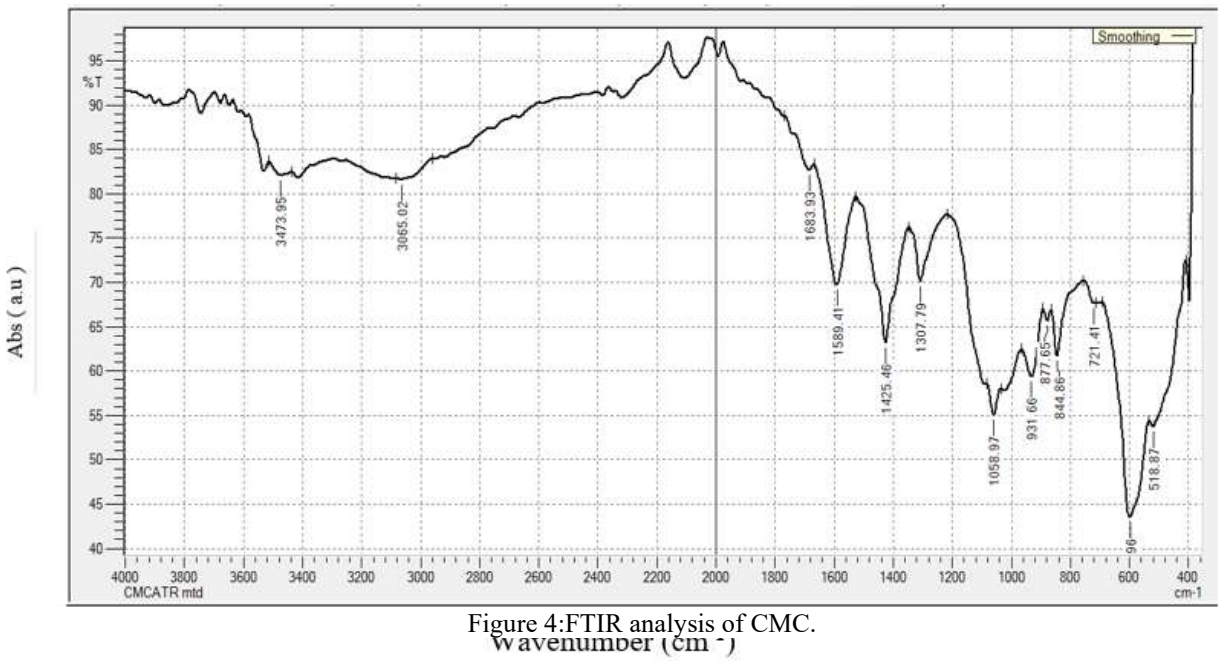


Figure 4:FTIR analysis of CMC.

4.3. FT-IR analysis of Hydrogels

4.3.1 FTIR analysis of hydrogel synthesised from CMC and cross-linker PEG (15 and 20 ml)

As it shown in fig 8 & 9, FTIR spectroscopy result clearly show that there was a crosslinking between carboxymethyl cellulose, and polyethylene glycol (PEG) of 15ml and 20ml respectively. Different band peaks were observed at different wave number values. The bands

of CMC/PEG (15 ml) observed at wave number values of 3269,48, 2893.35, 1591.34, 1413.88, 1319.37 and 1016.53, were assigned to O-H stretching vibration, C-H stretching vibration, carboxylate (-COO) asymmetric stretching vibration, scissoring bending vibration of -CH: carboxylate (COO) symmetric stretching vibration and C-O stretching vibration, respectively. The bands of CMC/PEG (20 ml) observed at wave number values of 327141, 2920 35, 1593 27, 1415.15, 1319.37 and 1053.18 were demonstrated that O-H stretching vibration, C-H stretching vibration, carboxylate (-COO) asymmetric stretching vibration, scissoring bending vibration of -CH_2, carboxylate (-COO) symmetric stretching vibration and C-O stretching vibration, respectively. The intensity of the bands observed for CMC PEG (20ml) were slightly increased as compared to those of CMC PEG (15ml) hydrogel thus indicated that intensity of the band of the hydrogel can enhanced with the increase in feed amount of the concentration of PEG in the preparation of CMC hydrogel. Therefore, a high concentration of PEG prompted

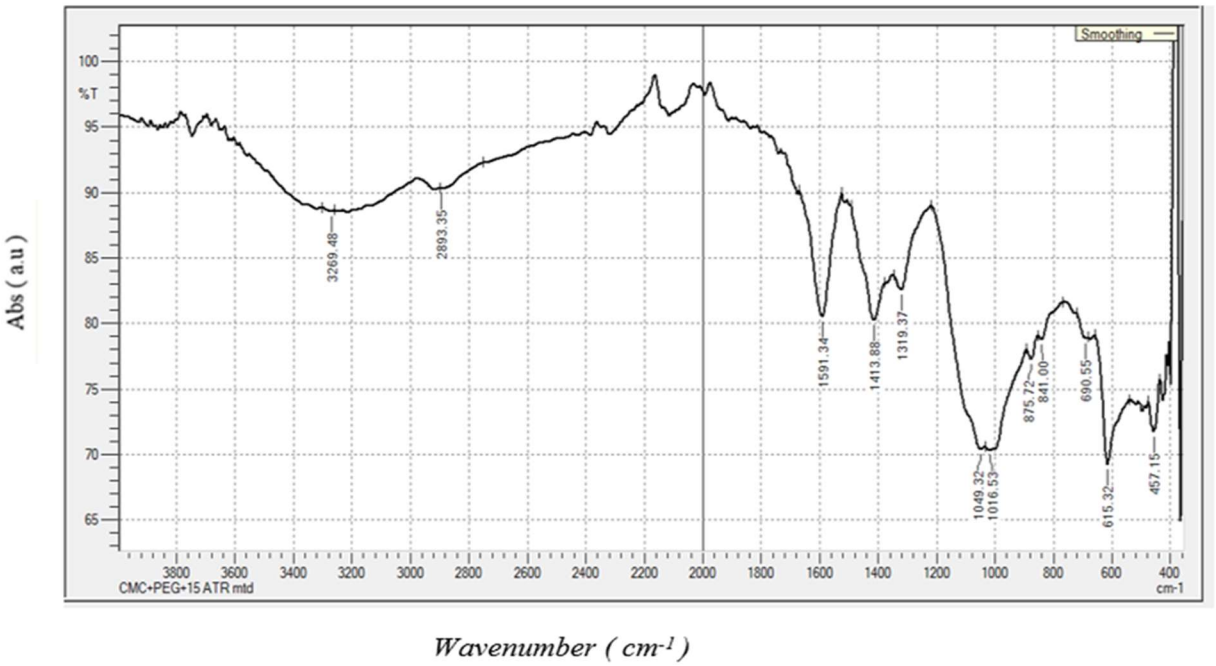


Figure 5:FTIR spectrum of hydrogel (CMC+PEG 15ml).

the crosslinking of CMC by PEG.

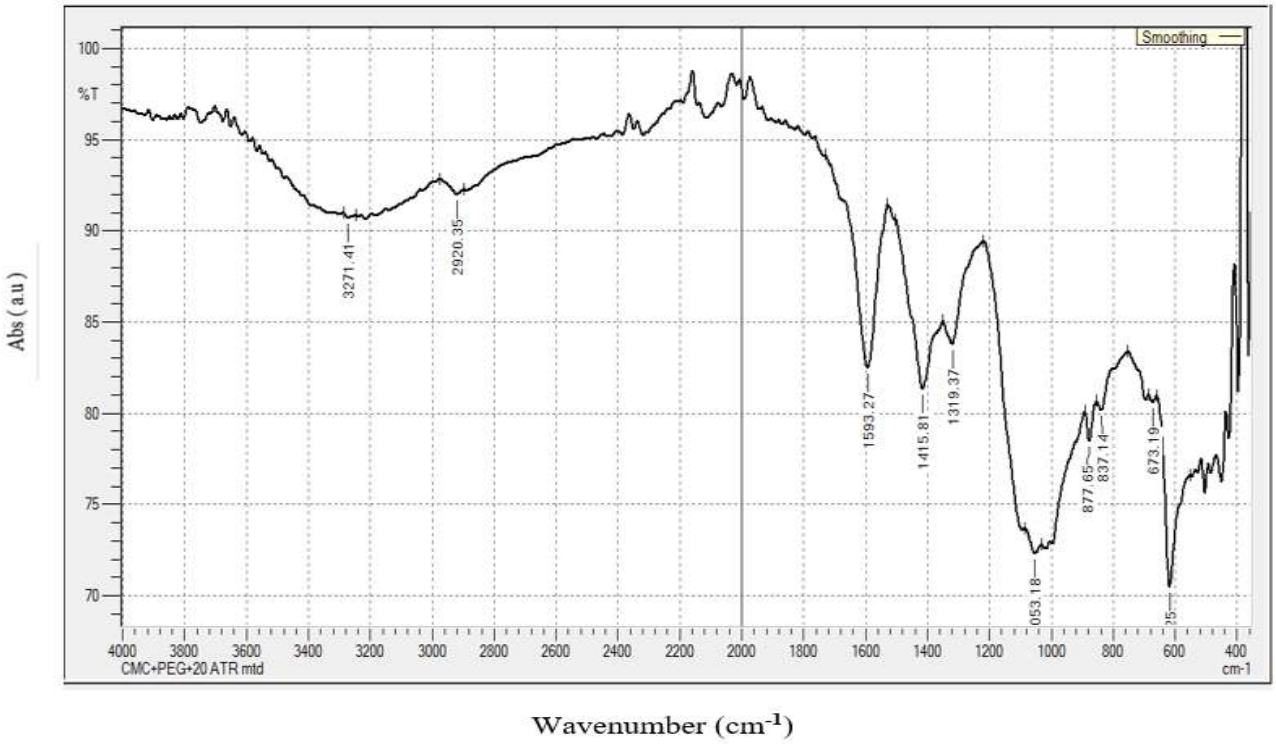


Figure 6: FTIR spectrum of hydrogel (CMC+PEG 20ml)

4.3.2 FT-IR analysis of hydrogel synthesised from CMC, cross-linker PEG (15 ml) and Agar.

Fig (10) shows FT-IR analysis of hydrogel synthesized from CMC, cross-linker PEG, and agar. The band was observed at 3267.55 cm^{-1} and assigned to a hydroxyl group(O-H) stretching vibration. Based on the observation of the FTIR fig (10) there is a broad peak because of the presence of a different hydroxyl group on the agar sample. The peak at 2891.42 cm^{-1} was associated with C-H stretching vibration, and 1593.27 cm^{-1} was assigned with C-O stretching vibration from the peptide group. The peak at 1411.95 cm^{-1} was corresponding with O-H bending vibration. The two bands at 1323.22 cm^{-1} and 1039 cm^{-1} correspond to bending motions of the terminal =CH2 and stretching vibrations of the carboxylic group's C-O bond, respectively. At 877.65 cm^{-1} C-O stretching was observed. The addition of agar increased the swelling capacity of hydrogels compared to hydrogels made without agar.

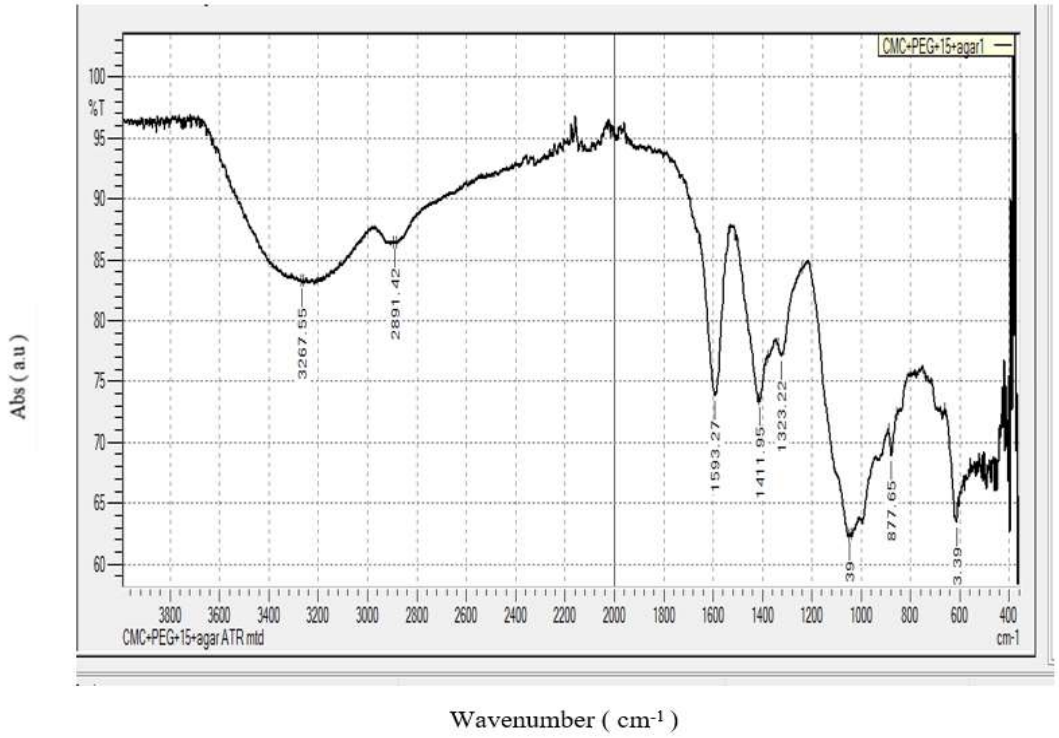


Figure 10:FTIR analysis of hydrogel (CMC +PEG + agar)

4.4.XRD analysis of Cellulose, Carboxymethylcellulose and Hydrogel

4.4.1. X-ray infrared (XRD) analysis of cellulose

measurement of cellulose CI by XRD provides a qualitative or semi-quantitative evaluation of the amounts of amorphous and crystalline cellulosic components in a sample.it helps to know the structure of cellulose. Unlike hemicellulose and lignin, which have an amorphous structure, cellulose has a crystalline structure. According to Zhang and Lynd , hydrogen bonding and Van der Waals interactions between adjacent molecules give cellulose a crystalline structure.

The typical peaks of cellulose I, according to Klemm et al. and Mahadeva et al. , are commonly recorded at 2θ values of roughly 15° and 22.6°. The XRD pattern of the synthesised cellulose is characterized by a well-defined principal peak at 22.6 (2θ) at the (110) plane and two secondary peaks , one at 18 (2θ) in the (200) plane which is moderate peak and the other at 35 (2θ) in the (004) plane giving the smallest peak. The peaks observed for cellulose which are produced in this research are almost the same with peaks observed for standard cellulose. A larger degree of crystallinity in the cellulose structure was linked to a sharper diffraction peak, according to Alemdar and Sain. The sharper diffraction peak was correlated with a high degree

of crystallinity in the cellulose structure. The smooth areas in the graph where sharp peaks are not observed indicate the amorphous structure of cellulose. Based on the data observed on XRD, We can

conclude that the given cellulose is semi-crystalline and semi amorphous.

4.4.2. XRD analysis of Carboxymethylcellulose (CMC)

The characteristic peaks at 2θ= 20°,25° 33.1°, and 45.53° in the X-ray diffraction (XRD) patterns of CMC reveal the amorphous and crystalline structure. CMC compounds exhibit absorption peaks, as shown in Figure 9, at 2θ= 20°, 33.1°, and 45.53°. CMC compounds can be identified at absorption of 2θ =20°. This is consistent with the findings of Huang et al., who stated that the XRD CMC diffractogram was observed at 2θ 20°. The absorption at 2θ 33.1° and 45.53°, which suggest that there are still impurities in CMC compounds, indicates that the results of this study's CMC compounds are not 100%

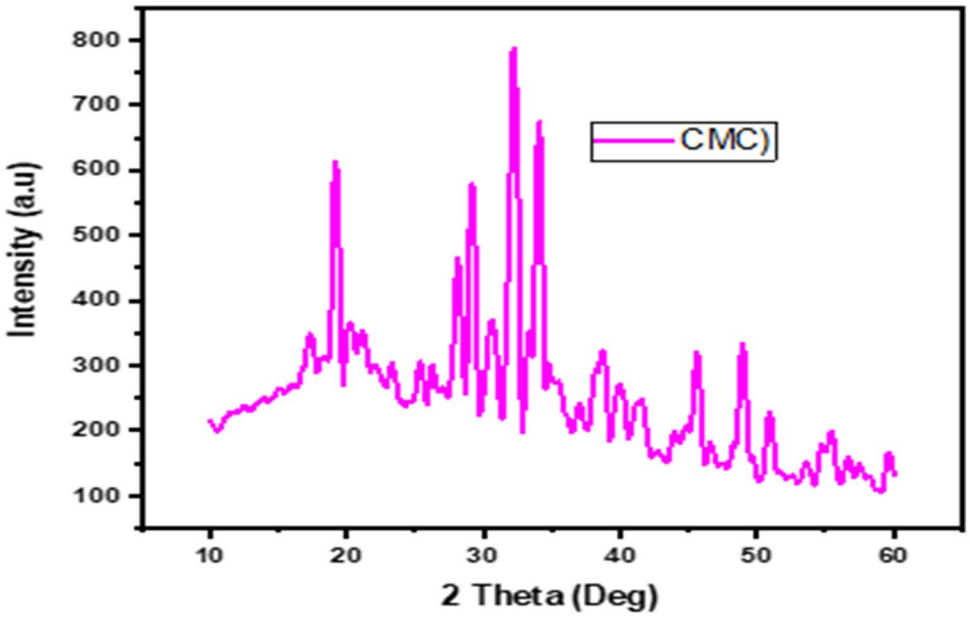
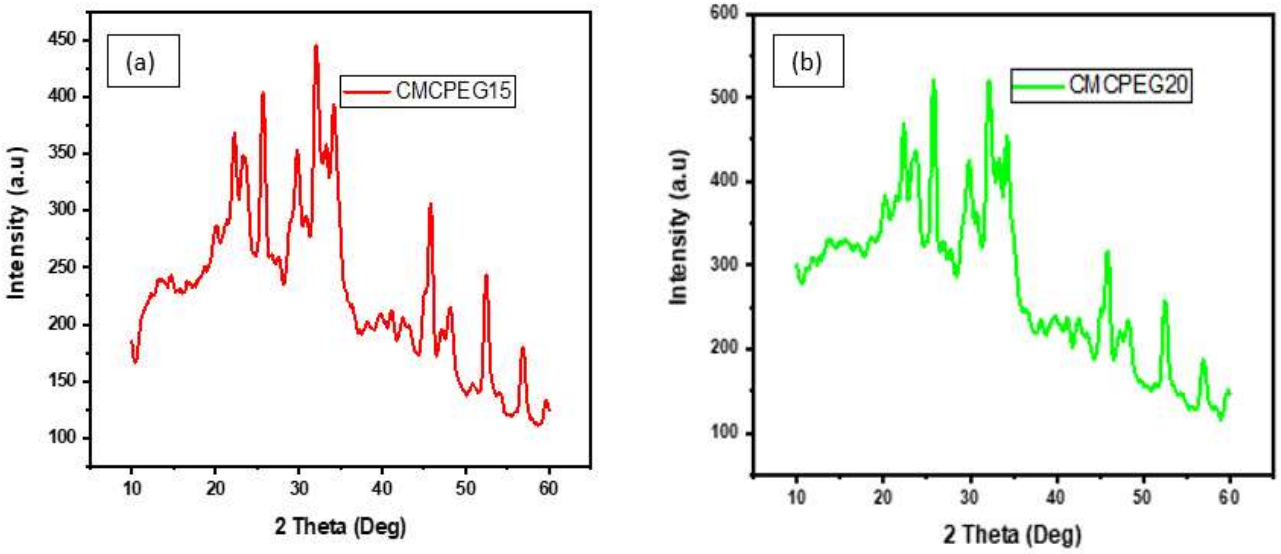


Figure 12:XRD analysis of CMC

4.4.3.XRD analysis of Hydrogels

The XRD patterns of CMC/PEG (15 ml) and CMC/PEG (20 ml) hydrogel blends are shown in fig 13 (a) and (b), respectively. It is found as they have nearly the same diffraction pattern. The crystalline nature of these hydrogels are evident with high intensity peak at 2θ of 25 and 35°.



The other diffraction peaks of the hydrogel are observed at 21, 32, 45, 53 and 57°. The peak intensity of CMC/PEG blend hydrogel is decreased as compared to CMC, and it might be due to hydrogen bond interaction between the carboxylic group of CMC and hydroxyl group of PEGS which resulted in change in the crystallographic position of two polymers after blending.

The XRD graph drawn on the x-axis is 2θ and on the y-axis is intensity. The crystallinity of hydrogel was examined by the x-ray diffraction technique. The XRD diffractogram of this hydrogel assigned to diffractions 2θ values at 20^0, 25^0, 31.2^0 33.4^0, 45.2^0, 53.6^0 and. From fig 13 (b) it has observed that the highest peaks have appeared at 2θ values at 20^0 ,25^0 and 33.4^0. The peaks are indicating the crystallinity of the processed hydrogel.

3.6. Analysis of porosity and water absorption capacity of hydrogel

The results showed that superabsorbent hydrogels (SAP) were created with swelling degrees ranging from around 200 to 600 %, with the concentration of CA crosslinker and the addition of PEG as a network modifier having a substantial impact.

The maximum water absorption capacity was found to be 570% which was % absorption of hydrogel synthesized from (CMC/Agar/ PEG 20ml) and the minimum was 284% and it was hydrogel synthesised of (CMC/ Agar/ CA 15%). Based on these values we can conclude that the Hydrogels synthesised from CMC and PEG have higher water absorption capacity than that of hydrogels synthesised from CMC and CA. the addition of Agar in the CMC/PEG hydrogel blend has subsequently increased the water absorption capacity of the hydrogel. So that

Figure 7:(a) and (b)XRD analysis of hydrogel synthesised from CMC ,15and 20 ml of PEG respectively

CMC/Agar/PEG blend hydrogel was suggested as an ideal material with good water absorption capacity in this research work.

The hydrogels synthesised have shown % porosity ranging from 50 % to 80 %. The maximum % porosity was found to be 78.87% which was calculated from hydrogel synthesised using CMC/Agar/ PEG 20ml and the minimum value was 58.5% which was the porosity of CMC/Agar/ CA 15% hydrogel. The addition of agar had increased the viscosity of the synthesised hydrogel. The increase in porosity of the hydrogels is exhibited during the addition of agar. The porosity of the generated hydrogels is influenced by the viscosity of the polymers. Zia et al., has also showed that, as the viscosity of the fluid increases, the removal of bubbles from polymeric hydrogels decreases to some extent, resulting in pores in the fabricated hydrogels and an increase in porosity. It was concluded that the porous hydrogel has more water absorption capacity.

(a) (b) (c)

Figure 14: (a) CMC/Agar/PEG 15ml Hydrogel ; (b) CMC/Agar /PEG 20ml Hydrogel; (c) water absorbed CMC/ Agar/ PEG 20ml hydrogel

Table 3 shows %porosity and water absorption % of different blends of s hydrogel synthesized

Table 3:porosity % and water absorption % values of different blends of synthesized hydrogel

Hydrogel	Porosity %	Water absorption %
CMC/PEG	69.70 %	550 %
CMC/ Agar / PEG 15ml	78.58 %	567%

CMC/Agar/ PEG 20ml	78.87%	570 %
CMC/Agar/ CA 15%	58.5%	284%

4.Conclusion

Bamboo tree is naturally abundant material that can be used as a source for cellulose based hydrogel production. Extraction of cellulose from bamboo tree was both challenging and imperative. In this research work, single and double stirring methods of extractions using different molarity of NaOH and KOH were applied and the double stirring method of extraction using 3M NaOH was found to be the best method. The effects of several variables, including temperature, concentration, ratio(w/v and v/v), mesh size of the bamboo powder, time of heating and stirring were investigated by analysing the yield of the cellulose obtained. As these parameters changed the cellulose yield also differs. the maximum yield obtained for the cellulose was 45.5% and we can say that the percentage yield found on this research was good when compared to the general abundance of cellulose which is 40% - 60%. Among the different mesh size bamboo powders used the 60 mesh size was found to be more available and cost effective to be used as a source of cellulose extraction. And cellulose extracted was modified to carboxymethyl cellulose. The success of the cellulose extracted and the modified cellulose ,CMC, was confirmed by using FTIR and XRD characterization methods. Finally a superabsorbent and porous hydrogel blend having 570% water absorption capacity and 78.87 % porosity was successfully synthesised using CMC / Agar / PEG(20ml).

5. References

Aarabi, S., Bhatt, K. A., Shi, Y., Paterno, J., Chang, E. I., Loh, S. A., Holmes, J. W., Longaker, M. T., Yee, H., & Gurtner, G. C. (2007). Mechanical load initiates hypertrophic scar formation through decreased cellular apoptosis. *The FASEB Journal*, *21*(12), 3250–3261. https://doi.org/10.1096/fj.07-8218com

Abdulhameed, A., Mbuvi, H. M., & Changamu, E. O. (2020). Synthesis of cellulose-based superabsorbent hydrogel from rice husk using a microwave. *American Journal of Materials Science*, *10*(1), 1–8. https://doi.org/10.5923/j.materials.20201001.01

Capanema, N. S. V., Mansur, A. A. P., de Jesus, A. C., Carvalho, S. M., de Oliveira, L. C., & Mansur, H. S. (2018). Superabsorbent crosslinked carboxymethyl cellulose-PEG hydrogels for potential

wound dressing applications. *International Journal of Biological Macromolecules*, *106*, 1218–1234. https://doi.org/10.1016/j.ijbiomac.2017.08.124

Capanema, N. S. V., Mansur, A. A. P., Mansur, H. S., de Jesus, A. C., Carvalho, S. M., Chagas, P., & de Oliveira, L. C. (2018). Eco-friendly and biocompatible cross-linked carboxymethylcellulose hydrogels as adsorbents for the removal of organic dye pollutants for environmental applications. *Environmental Technology (United Kingdom)*, *39*(22), 2856–2872. https://doi.org/10.1080/09593330.2017.1367845

Dara, P. K., Raghavankutty, M., Deekonda, K., Vemu, A. K., Sivam, V., Mathew, S., Rangasamy, A., Chandragiri Nagarajarao, R., & Subramanian, S. (2021). Synthesis of Biomaterial-Based Hydrogels Reinforced with Cellulose Nanocrystals for Biomedical Applications. *International Journal of Polymer Science*, *2021*. https://doi.org/10.1155/2021/4865733

Desalegn, G., & Tadesse, W. (2014). Resource potential of bamboo, challenges and future directions towards sustainable management and utilization in Ethiopia. *Forest Systems*, *23*(2), 294. https://doi.org/10.5424/fs/2014232-03431

Enawgaw, H., Tesfaye, T., Yilma, K. T., & Limeneh, D. Y. (2021). *Synthesis of a Cellulose-Co-AMPS Hydrogel for Personal Hygiene Applications Using Cellulose Extracted from Corncobs*. 1–10.

Fitrah, N., Nan, C., Zainuddin, N., & Ahmad, M. (2019). *Preparation and swelling study of CMC hydrogel as potential superabsorbent SCIENCE & TECHNOLOGY Preparation and Swelling Study of CMC Hydrogel as Potential Superabsorbent*. *January*.

Gurtner, G. C., Werner, S., Barrandon, Y., & Longaker, M. T. (2008). Wound repair and regeneration. *Nature*, *453*(7193), 314–321. https://doi.org/10.1038/nature07039

Huang, L. J., Lee, W. J., & Chen, Y. C. (2022). Bio-Based Hydrogel and Aerogel Composites Prepared by Combining Cellulose Solutions and Waterborne Polyurethane. *Polymers*, *14*(1). https://doi.org/10.3390/polym14010204

Husain, M. S. B., Gupta, A., Alashwal, B. Y., & Sharma, S. (2018). Synthesis of PVA/PVP based hydrogel for biomedical applications: a review. *Energy Sources, Part A: Recovery, Utilization and Environmental Effects*, *40*(20), 2388–2393. https://doi.org/10.1080/15567036.2018.1495786

Jirawitchalert, S., Mitaim, S., Chen, C.-Y., & Patikarnmonthon, N. (2022). Cotton Cellulose-Derived Hydrogel and Electrospun Fiber as Alternative Material for Wound Dressing Application. *International Journal of Biomaterials*, *2022*, 1–12.

https://doi.org/10.1155/2022/2502658

Kabir, S. M. F., Sikdar, P. P., Haque, B., Bhuiyan, M. A. R., Ali, A., & Islam, M. N. (2018). Cellulose-based hydrogel materials: chemistry, properties and their prospective applications. *Progress in Biomaterials*, *7*(3), 153–174. https://doi.org/10.1007/s40204-018-0095-0

Liu, J., Fan, X., Zhang, Y., Zheng, H., & Zhu, M. (2020). Quantitative evaluation for moisture content of cellulose insulation material in paper/oil system based on frequency dielectric modulus technique. *Cellulose*, *27*(4), 2343–2356. https://doi.org/10.1007/s10570-019-02820-3

Mulatu, Y., Environment, E., & Fetene, M. (2013). *Official Journal of the Biological Society of Ethiopia Ethiopian Journal of Biological Sciences Indexed and abstracted by CABI AJOL STAND STRUCTURE, GROWTH AND BIOMASS OF ARUNDINAR... July 2020*.

Nigatu, A., Wondie, M., Alemu, A., Gebeyehu, D., & Workagegnehu, H. (2020). Productivity of highland bamboo (Yushania alpina) across different plantation niches in West Amhara , Ethiopia. *Forest Science and Technology*, *0*(0), 1–7. https://doi.org/10.1080/21580103.2020.1791260

Rizwan, M., Peh, G. S. L., Ang, H. P., Lwin, N. C., Adnan, K., Mehta, J. S., Tan, W. S., & Yim, E. K. F. (2017). Sequentially-crosslinked bioactive hydrogels as nano-patterned substrates with customizable stiffness and degradation for corneal tissue engineering applications. *Biomaterials*, *120*, 139–154. https://doi.org/10.1016/j.biomaterials.2016.12.026

Velnar, T., Bailey, T., & Smrkolj, V. (2009). The wound healing process: An overview of the cellular and molecular mechanisms. *Journal of International Medical Research*, *37*(5), 1528–1542. https://doi.org/10.1177/147323000903700531

Wang, J., Wang, Q., Wu, Y., Bai, F., Wang, H., Si, S., Lu, Y., Li, X., & Wang, S. (2020). Preparation of cellulose nanofibers from bagasse by phosphoric acid and hydrogen peroxide enables fibrillation via a swelling, hydrolysis, and oxidation cooperative mechanism. *Nanomaterials*, *10*(11), 1–15. https://doi.org/10.3390/nano

Abdel-Halim, E. S. (2014). Chemical modification of cellulose extracted from sugarcane bagasse: Preparation of hydroxyethyl cellulose. *Arabian Journal of Chemistry*, *7*(3), 362–371. https://doi.org/10.1016/j.arabjc.2013.05.006

Capanema, N. S. V., Mansur, A. A. P., de Jesus, A. C., Carvalho, S. M., de Oliveira, L. C., & Mansur, H. S. (2018a). Superabsorbent crosslinked carboxymethyl cellulose-PEG hydrogels for potential wound dressing applications. *International Journal of Biological Macromolecules*, *106*, 1218–1234. https://doi.org/10.1016/j.ijbiomac.2017.08.124

Capanema, N. S. V., Mansur, A. A. P., de Jesus, A. C., Carvalho, S. M., de Oliveira, L. C., & Mansur, H. S. (2018b). Superabsorbent crosslinked carboxymethyl cellulose-PEG hydrogels for potential wound dressing applications. *International Journal of Biological Macromolecules*, *106*, 1218–1234. https://doi.org/10.1016/j.ijbiomac.2017.08.124

Liew, F. K., Hamdan, S., Rahman, M. R., Rusop, M., Lai, J. C. H., Hossen, M. F., & Rahman, M. M. (2015). Synthesis and characterization of cellulose from green bamboo by chemical treatment with mechanical process. *Journal of Chemistry*, *2015*. https://doi.org/10.1155/2015/212158

Liu, J., Fan, X., Zhang, Y., Zheng, H., & Zhu, M. (2020). Quantitative evaluation for moisture content of cellulose insulation material in paper/oil system based on frequency dielectric modulus technique. *Cellulose*, *27*(4), 2343–2356. https://doi.org/10.1007/s10570-019-02820-3

Wang, T., & Zhao, Y. (2021). Optimization of bleaching process for cellulose extraction from apple and kale pomace and evaluation of their potentials as film forming materials. *Carbohydrate Polymers*, *253*(October 2020), 117225. https://doi.org/10.1016/j.carbpol.2020.117225

Zainal, S. H., Mohd, N. H., Suhaili, N., Anuar, F. H., Lazim, A. M., & Othaman, R. (2021). Preparation of cellulose-based hydrogel: A review. *Journal of Materials Research and Technology*, *10*, 935–952. https://doi.org/10.1016/j.jmrt.2020.12.012