

# Synthesis and Characterization of Carboxymethyl Cellulose (CMC) from Water Hyacinth Using Ethanol-Isobutyl Alcohol Mixture as the Solvents

Asep Handaya Saputra, Linnisa Qadhayna, and Alia Badra Pitaloka

**Abstract**—Water hyacinth, a free-floating aquatic weed originating from South America has become a major floating weed of tropical and subtropical regions of the world. The plant has the tendency to regenerate from seeds and fragment allowing rapid increase in plant population. Water hyacinth is however a fiber that is rich in its cellulosic compounds, which can be derivated into somewhat multifunctional properties. Carboxymethyl Cellulose (CMC) is a derivated cellulose that is used in food products as a thickener agent or non-food products such as detergents, paints, and others. The research has investigated further on how one would synthesize CMC from water hyacinth as well examining the characterization of the CMC samples that is originated from the plant. The production of CMC involves four main processes: the isolation of alpha-cellulose from the water hyacinth and the synthesis by the alkali-catalyzed reaction (alkalization) of cellulose with chloroacetic acid (carboxymethylation) and finally the purification of the CMC itself to remove undesirable compounds. In this research, the variations are comprised of the NaOH added (5 M - 35 M) during alkalization and the ratio of the solvent between isobutanol and ethanol (ratio 80:20, 50:50 and 20:80) thus fifteen samples are obtained. The characterization is based on two testing methods: FTIR and Degree of Substitution (DS). During FTIR test, it is proven that CMC is produced in the experiment. This is verified from the spectrum transmitting 1400 cm<sup>-1</sup> 1600 cm<sup>-1</sup> indicating ether and carboxyl functional group consecutively. On the other hand, the highest DS is obtained in sample CMC-28-10 with a value of 1.76 with the highest purity of 93.24.

**Index Terms**—Carboxymethyl cellulose (CMC), water hyacinth, ethanol, isobutyl alcohol

## I. INTRODUCTION

Cellulose is a long chain of linked sugar molecules that can provide strength for all kinds of plants [1]. It is the main component of plant cell walls, and the basic building block for many commercial products such as textiles, packaging, explosives, paper and many more [1]. The cellulosic compounds from plants can then be integrated into complex cellulosic compounds. These cellulose derivatives are developed so that it can be soluble in water or dilute alkali and thus can be utilized as finishing and sizing agents for textiles, as absorbable surgical gauze, protective colloids, adhesives, thickening agents for foods, creams, ointments

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and pastes and many other industries [4]. Water hyacinth for example in nature is a type of plant that is very rich in its cellulose property and thus it can be obtained for the purpose of a more valuable product. The celluloses can subsequently be integrated into a derivative of a cellulosic compound that is, CMC (carboxymethyl cellulose).

CMC is a water-soluble fiber at room temperature. In this research, the focus will be generating carboxymethyl cellulose from water hyacinth, a fast growing wild weed in water that is highly problematic. In order to see which would provide the highest quality of production, one can differentiate from the values in their degree of substitution from having different solvent medium. There are two main processes when synthesizing CMC. First of all, the cellulosic compounds in the water hyacinth are required to be isolated so that it would not interrupt the production of CMC. Other than celluloses, water hyacinth is also comprised of hemicelluloses as well as lignin. These two undesirable compounds needed to be removed so that only solely celluloses will be acquired. When the celluloses are finally obtained, the process of converting cellulose into a derivative compound can be assembled. There are two main reactions in order to convert cellulose into CMC: the first procedure is alkalization and the second reaction is followed by carboxymethylation. The goal and objectives of the research are to investigate further whether water hyacinth cellulose can be synthesized into CMC at various assorted variables during the processes (various concentrations of NaOH and the ratio of the isobutyl-ethanol mixture) as well as to characterize the CMC samples based on its degree of substitution (DS), Fourier Transform Infra Red (FTIR) and its purity to identify which CMC has the highest quality out of all.

There had been various researches that had executed a similar experimental approach (i.e. synthesis of carboxymethyl cellulose) using alternate raw materials other than wood. Most of these researches selected isopropanol as the solvent during their experiment. The reason for this is because isopropanol have proven to provide the highest value of DS. Barai et al [2] for instance selected water hyacinth as the raw material and they concluded that the best set of conditions for carboxymethylation for  $\alpha$ -cellulose was by utilizing isopropyl alcohol as the solvent medium. In addition, 10% of aqueous NaOH solution had proven to give the highest DS in comparison to other NaOH concentrations. Other than the solvent, most of the researches used other variations such as different temperature profiles during carboxymethylation, distinctive configurations of the solvent (a mixture of two or three different solvents within different compositions) and many more.

## II. EXPERIMENTAL PROCEDURE

### A. Materials and Apparatus

The materials used in the research are  $\alpha$ -cellulose of water hyacinth, distilled water, sodium hydroxide, sodium monochloroacetate, isobutyl alcohol, ethanol, ethanol 96%, acetic acid, toluene, sodium chlorite and acetone.

On the other hand the utilized used are beaker glass, Erlenmeyer flask, graduated cylinder, volumetric pipettes, petri dish, porcelain evaporation lab dish, glass lab dish, filter paper, digital scale, fumehood, oven, thermometer, soxhlet, vacuumed pump, pH meter, pH paper, water bath and stirring rod with motor.

### B. Experimental Procedure

In order to generate carboxymethyl cellulose from the water hyacinth there are two main stages that is needed to be achieved (this include both physical and chemical reactions). The first procedure is initiated by obtaining the cellulosic parts of the plant and secondly derivate the cellulose obtained into carboxymethyl cellulose. In this section, one would discuss the research method of producing carboxymethyl cellulose (CMC) from water hyacinth as the raw material.

The first part of the research method involved the preparation of water hyacinth cellulose. The purpose of dewaxing is to remove wax as well as pectin content in the water hyacinth. The first process is followed by delignification process by bleaching the water hyacinth powder with 1%  $\text{NaClO}_2$  solution. However, other than lignin, hemicellulose has to be removed by alkylation using 17.5%  $\text{NaOH}$ . After the two main processes have been done, the water hyacinth cellulose is washed and neutralized using acetic acid and warm water. The second part involved synthesizing the cellulose into CMC, which is comprised of three main processes: alkalization, carboxymethylation and neutralization. The solvent used during alkalization is ethanol and isobutanol mixture (ratio 20:80, 50:50 and 80:20). Sodium hydroxide is added drop by drop during this process and the variations of  $\text{NaOH}$  are 5%, 10%, 20%, 30% and 35%. Sodium Chloroacetic Acid is added during carboxymethylation at a temperature of  $60^\circ$ . Consequently each sample is then purified and neutralized using ethanol 96%. There will be three characterizations: FTIR, the degree of substitution and its purity.

The degree of substitution is determined by a sequence of procedures. First of all 0.5-0.7 g (weight in dry basis) of the sample is weighed and wrapped in a filter paper. It is then ignited in crucible. After cooling, the crucible is then placed into a 500 ml beaker and 250 ml of water added. Afterwards 35 ml of N/10  $\text{H}_2\text{SO}_4$  by a pipet, it is then boiled for 30 minutes. The solution is then titrated with N/10  $\text{KOH}$  and shaking it gently using phenolphthalein as the indicator. Separately, the alkalinity or acidity of the sample is measured by the method below and the degree of substitution is calculated as followed:

$$D.S. = \frac{(162)(A)}{10000 - (80)(A)} \quad (1)$$

$$A = \frac{af - bf_1}{\text{Weight of Sample (g) in Dry Basis}} - * \text{alkalinity (or + acidity)} \quad (2)$$

where:

$a$ : The volume (ml) of N/10  $\text{H}_2\text{SO}_4$  used

$f$ : The factor of N/10  $\text{H}_2\text{SO}_4$

$b$ : The volume (ml) of N/10  $\text{KOH}$  required

$f_1$ : The factor of N/10  $\text{KOH}$

(\*): Measuring Alkalinity or Acidity

One gram of the sample of the sample (dry basis) is measured accurately and dissolved by adding about 200 ml of water.

Afterwards, 5 ml of N/10  $\text{H}_2\text{SO}_4$  is added by a pipet and the solution is boiled for 10 minutes. After cooling, it is titrated with N/10  $\text{KOH}$  (S ml) using phenolphthalein as the indicator. A blank test (without CMC) is pursued at the same time (B ml) and the following formula is used for calculation:

$$\text{Alkalinity (or acidity)} = \frac{(B - S)(f_1)}{\text{Weight (g) of sample on dry basis}} \quad (3)$$

Remarks: When the absolute value is (+), it is alkalinity and when the absolute value is (-), it is acidity. On the other hand the purity is determined by:

$$\text{Purity} = 100(\%) - \text{NaCl}(\%) + \text{Sodium Glycolate}(\%) \quad (4)$$

The sodium chloride content is determined by a series of procedure: one gram of the sample is weighed accurately in a 300 ml flask, which is dissolved by adding about 200 ml of distilled water. The solution is then titrated with N/10  $\text{AgNO}_3$ , using potassium chromic acid as the indicator. The result is calculated by the following formula:

$$\text{NaCl}(\%) = \frac{0.5846(f)(\text{required volume (ml) of } \frac{N}{10} \text{ AgNO}_3)}{\text{Weight of Sample in Grams (dry basis)}} \quad (5)$$

$f$  = the factor N/10  $\text{AgNO}_3$

## III. RESULTS AND DISCUSSIONS

The nature and composition of the organic solvents selected as the reaction media plays an important part in the structural transformation of cellulose during alkalization. The formation of alkali cellulose occurred in this stage where the crystalline structure is modified and thus its accessibility to chemicals increases by swelling. Cellulose can be swollen in concentrated  $\text{NaOH}$  but not dissolved [3]. The change of crystallinity and polymorphism is due to the partition of sodium hydroxide between the reaction medium and the cellulose chain. The partition occurs when suspending cellulose in mixtures of isobutanol, ethanol water and sodium hydroxide where the organic medium plays the role of uniformly distributing  $\text{NaOH}$  with water in cellulose. Whereas  $\text{NaOH}$  will be fully miscible in ethanol and water, it does not so in isobutanol, therefore there will be two different layers of solution, the upper layer solution (ULS) and the lower layer solution (LLS) [4]. According to Olaru, Olaru since  $\text{NaOH}$  is dissolved in ethanol completely, the concentration of the base will be lowest near the polymeric chains thus there will be a minimum modification of the crystalline structure of cellulose. However, since the

presence of isobutanol exist where it plays a role of a poor solvent for sodium hydroxide would favor higher concentrations of NaOH in the vicinity of macromolecules and thus give rise to substantial decrystallization and change of polymorphism from cellulose to alkali cellulose during alkalization. Since there are two different solvent media, the change of the crystalline structure is intermediary and thus there would be different proportions of amorphous material.

Sodium carboxymethyl cellulose is produced from a heterogeneous reaction where its rate is dependent upon the diffusion rate of the reagents NaOH and  $\text{ClCH}_2\text{COONa}$  inside the cellulose particles [5]. This means that the aggregation state of the cellulose particles act as a decisive role. The crystalline structure of the particles should be destroyed adequately so that the particles become looser thus increasing the reaction rate. If the crystalline aggregation cannot be destructed then the NaOH and  $\text{ClCH}_2\text{COONa}$  will remain in the solvent rather than diffuse into the crystalline aggregation when carrying out the carboxymethylation for cellulose. That is the reason as to why, NaOH as well as  $\text{ClCH}_2\text{COONa}$  is added slowly and is to be stirred at a very high speed so that the reaction will take place. In addition, by having complete reactions, it would lessen the production of byproducts such as sodium glycolate and also decreasing the availability of monochloroacetic acid.

#### A. Analysis of CMC by Its Yield

There are fifteen different samples with two different variations i.e. three distinctive compositions of the organic solvent (isobutanol and ethanol) and five different concentrations of NaOH. The yield is obtained by dividing the amount of dry CMC obtained by the amount of the cellulose. The results are tabulated in Table I, Table II, and Table III consecutively.

##### 1) The composition of isobutyl-ethyl alcohol with a ratio of 80ml: 20ml

In this variation there are five different sets of data with different concentrations of NaOH (i.e. 5%, 10%, 20%, 30% and 35%) with a similar composition of isobutyl-ethyl alcohol as the solvent (20 ml: 80 ml ratio). The Table below exemplifies the physical features (weight, yield and pH) of each sample:

TABLE I: CMC SAMPLES WITH A MIXTURE RATIO OF 80:20 (ISOBUTYL: ETHYL ALCOHOL)

Concentration of NaOH	Weight of Cellulose (grams)	Weight of NaMCA (grams)	Weight of Dry CMC (grams)	Yield (%)	Initial pH
5%	5.20	6.20	4.95	95.29%	7
10%	5.20	6.20	4.59	88.27%	7.5
20%	5.20	6.20	4.45	85.58%	10
30%	5.20	6.20	4.73	90.96%	12
35%	5.20	6.20	6.43	123.65%	14

##### 2) The composition of isobutyl-ethyl alcohol with a ratio of 50 ml: 50 ml

In this variation there are another five different sets of data with different concentrations of NaOH (i.e. 5%, 10%, 20%, 30% and 35%) with a similar composition of isobutyl-ethyl alcohol as the solvent (50 ml: 50 ml ratio). The Table below exemplifies the physical features (weight, yield and pH) of each sample:

TABLE II: CMC SAMPLES WITH A MIXTURE RATIO OF 50:50 (ISOBUTYL: ETHYL ALCOHOL)

Concentration of NaOH	Weight of Cellulose (grams)	Weight of NaMCA (grams)	Weight of Dry CMC (grams)	Yield (%)	Initial pH
5%	5.20	6.20	5.27	101.35%	7
10%	5.20	6.20	7.66	147.31%	8
20%	5.20	6.20	9.31	179.04%	9
30%	5.20	6.20	7.59	145.96%	12
35%	5.20	6.20	6.70	128.85%	14

##### 3) The composition of isobutyl-ethyl alcohol with a ratio of 20ml: 80 ml

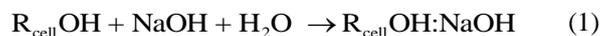
The last variation is consisted of another five different sets of data with different concentrations of NaOH (i.e. 5%, 10%, 20%, 30% and 35%) with a similar composition of isobutyl-ethyl alcohol as the solvent (80 ml: 20 ml ratio). The Table in 4.6 exemplifies the physical features (weight, yield and pH) of each sample:

TABLE III: CMC SAMPLES WITH A MIXTURE RATIO OF 20:80 (ISOBUTYL: ETHYL ALCOHOL)

Concentration of NaOH	Weight of Cellulose (grams)	Weight of NaMCA (grams)	Weight of Dry CMC (grams)	Yield (%)	Initial pH
5%	5.20	6.20	6.54	125.38%	7
10%	5.20	6.20	7.75	149.04%	8
20%	5.20	6.20	8.55	164.42%	10
30%	5.20	6.20	7.72	148.46%	12
35%	5.20	6.20	6.95	133.65%	14

From the Tables depicted above, the yield of the CMC is approximately ranging from 85.58% (CMC-82-20) until 179.04 % (CMC-55-20). The reaction of CMC involved a two-step process:

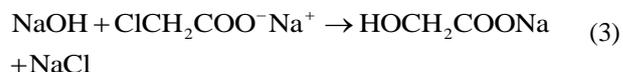
Step One:



Step Two:



In addition, there is also a by-product (sodium glycolate):



The first step, cellulose is suspended in alkali to open the bound cellulose chain, allowing water to enter. The treatment of exposing cellulose with caustic soda is to break crystalline clusters and obtain an alkali-cellulose complex, a substrate that allows an access to the following reaction: carboxymethylation. During the next stage, the alkali cellulose is reacted with sodium monochloroacetate to obtain sodium carboxymethyl cellulose.

The reason as to why the yield is larger than the raw material itself (cellulose) is due to the addition of monochloroacetic acid during carboxymethylation. The amount of monochloroacetic acid added for each sample is 6.20 gram. The calculation of the percentage yield is computed by dividing the weight of the dry CMC by the weight of cellulose solely (weight of CMC/weight of cellulose). The monochloroacetic acid reacted is not taken

into account for the yield percentage and that is the reason as to why the percentage yield is very high. From the Table above, there are no correlations between any of the results (The amount of NaOH added with the percentage yield). There are also no correlations to other characterizations (such as DS, FTIR or purity) towards the yield of the CMC. The reason for this is because with the calculation of the yield itself, one cannot conclude whether all of the cellulosic compounds have actually been mercerized into CMC.

However according to BPPK, one can identify whether a reaction have actually occurred in the research. CMC is soluble in water where it would form a gelled-like substance when water is added.

### B. Analysis of CMC by FTIR

The CMC samples were analyzed by FTIR, which can be depicted in the two spectrum in Fig. 1. The x-axis represents the wavelength ( $\text{cm}^{-1}$ ) and y-axis whos the light transmittance through the sample. The FTIR spectrum of the sample shows that the carboxyl, methyl and hydroxyl functional groups are found at wavelength of 1617.60, 1454.44 and 1195.72 (for CMC-55-10), 1613.57, 1426.01 and 1214.54 (for CMC-28-10) respectively. However for sample CMC-82-10 only carboxyl and methyl functional groups are shown in the peak at 1654.16 and 1458.13  $\text{cm}^{-1}$ . Consequently for all CMC samples synthesized the IR spectra indicates the typical absorptions of the cellulose backbone as well as the presence of the carboxymethyl ether group at 1654.16, 1617.60 and 1613.57  $\text{cm}^{-1}$  consecutively for each sample. The additional peak at wavelength of 2357.27 at sample CMC-55-10 might be due to the existence of the contamination from impurities or combination band with water. Subsequently the bands around 1458.15 – 1420.05  $\text{cm}^{-1}$  are assigned to  $\text{CH}_2$  scissoring. It is obvious that those in the broad absorption band of approximately above 3500  $\text{cm}^{-1}$  is due to the stretching frequency of the hydroxyl group (-OH).

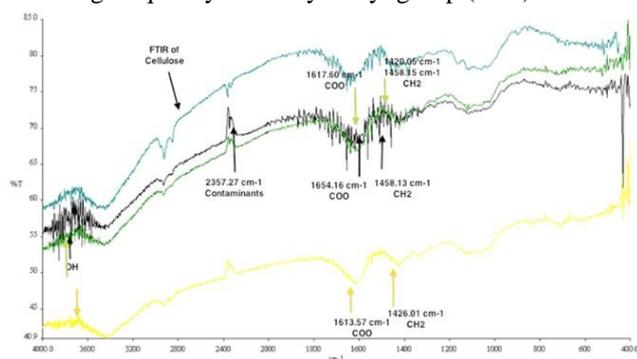


Fig. 1. FTIR representation of sample CMC-82-10 (black), CMC-55-10 (green) and CMC-28-10 (yellow) and a FTIR analysis of cellulose (blue)

### C. Analysis of CMC by the Degree of Substitution

Before moving on with the analysis of CMC by the degree of substitution, notations are used when naming the samples. The terminology of naming of the sample is exemplified as below:

- CMC-82-05

In the nomenclature specified above, CMC directly means the sample, 82 indicates the ratio of composition used in the variable (80% isobutanol, 20% ethanol) and 05 denotes the concentration of NaOH used (in this case 5%).

An analysis of the degree of substitution (DS) is conducted by utilizing Cellogen (DSK International) as the testing method. With the DS of each sample tested, the effect of the solvents can be studied. The solvent used were mixtures of isobutyl alcohol with ethanol within different compositions (80:20, 50:50 and 20:80 respectively). As one can observe there are undetected degree of substitution seen in Table IV. Thereason as to why the degree of substitution is undetected is due to each of these samples' conditions are not yet fully neutralized. This probably also happened with the degree of substitution that is lower than expected. In addition, the test was only performed once instead of more, due to lack of samples; therefore multiple tests cannot be preceded. However looking at the trend in CMC-28-05 through CMC-28-35 (CMC samples with a mixture of 20% ethanol, 80% isobutanol), the highest DS obtained was in sample CMC-28-10 with a value of 1.67. Initially with a NaOH concentration of five percent, the value of the DS is only 0.79 until it rises almost twice as high with a NaOH concentration of 10% (DS: 1.67) and it falls again to 0.79 in sample CMC-28-20.

With the trend exemplified in between CMC-28-05 through CMC-28-35 one can conclude the effect of various concentrations of sodium hydroxide. At particular alkali strength, the DS was maximum after which it started declining. The observation can be explained by considering the carboxymethylation process that actually occurred during the experiment. The first involve the reaction of the cellulose hydroxyl with sodium monochloroacetate in the presence of sodium hydroxide to give CMC. On the other hand, the second reaction involves sodium hydroxide reacting with sodium monochloroacetate to form sodium glycolate. For sample CMC-28-10, the first reaction stood out more than the second reaction, therefore more CMC is formed rather than the byproduct, sodium glycolate. However as it CMC-28-20 to CMC-28-35 is preceded, glycolate formation pre-dominates which means inactivation of mnochloroacetate and its consumption by the side reaction. A similar approach had been reported with [6].

TABLE IV: THE DEGREE OF SUBSTITUTION FOR ALL SAMPLES

Sample	Polarity Index	Degree of Substitution
CMC-82-05	4.16	0.14
CMC-82-10		Undetected
CMC-82-20		0.28
CMC-82-30		0.10
CMC-82-35		0.01
CMC-55-05	4.55	0.14
CMC-55-10		Undetected
CMC-55-20		Undetected
CMC-55-30		0.02
CMC-55-35		0.80
CMC-28-05	6.78	0.79
CMC-28-10		1.67
CMC-28-20		0.79
CMC-28-30		0.76
CMC-28-35		0.79

Overall, the reactivity of cellulose depends on the compositions of the organic solvent (ethanol and isobutyl

mixture) due to the change of crystallinity and polymorphism caused during the alkalization step. In addition the role of the solvent in the carboxymethylation reaction is to provide accessibility of the etherifying reagent to the reaction centers of the cellulose chain. The highest degree of substitution obtained when the presence of ethanol is at its highest (80 percent). According to Barai et al, the polarity index of the solvent affects the reaction efficiency. It is concluded that when the polarity of the solvent decreases, the reaction efficiency increases. However this does not relate with the research, the reason is because isobutyl alcohol has three bulky methyl groups surrounding the primary carbon atom, which may provide steric hindrance to the reacting groups. This would then account for the lower DS attained in CMC-82-05 to CMC-82-35 where the majority of the solvent is isobutyl alcohol (80%). Thus, the highest DS acquired will be when the composition of the solvent is 80% ethanol and 20% isobutanol.

#### D. Analysis of CMC by the Purity

Other than FTIR and DS analysis, the samples are also studied in terms of its purity by taking the three highest DS of each varied composition. The value of the purity is tabulated in Table V.

TABLE V: THE PURITY OF THE THREE MOST SIGNIFICANT SAMPLES

Sample	Purity
CMC-82-20	91.51%
CMC-55-35	90.12%
CMC-28-10	93.24%

Despite, the low value of DS, CMC is proved to be formed due to the purity test acquired above. CMC-82-20 with a DS of only 0.28 has the a purity value of 91.51. This does not differ much with CMC-28-10 with a DS value as high as 1.67 but a purity content of 93.24 (only differ by approximately 2 percent). However despite the high purity content, DS plays a larger role when it comes to the manufacture of CMC. The higher the degree of substitution, the more the CMC would provide improved compatibility with other soluble components such as salts and non-solvents.

#### IV. CONCLUSION

It is concluded that water hyacinth can be derived into CMC by applying the method that had been executed during the research. The obtained samples of CMC are attained within two different variations: concentrations of NaOH and the isobutyl-ethanol mixture. The highest degree of substitution obtained is sample CMC-28-10 when the composition of ethanol is 80% and isobutanol at 20% with a value of 1.76. However, for the purity test, within three samples tested (CMC-82-20, CMC-55-35 and CMC-28-10), the results are 91.51%, 90.12% and 93.24% respectively. This proves that CMC has actually been formed.

From the experiment designated above, it is suggested that other solvent should be selected instead of isobutanol since the alcohol has three bulky methyl groups surrounding the primary carbon atom, which prevents further reaction from taking place (i.e. conversion of alkali cellulose into

carboxymethyl cellulose).

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**Linnisa Qadhayna** was born on December 6, 1989 and has just recently obtained her bachelor degree from Universitas Indonesia in August 2013 majoring in Chemical Engineering. Her research was centered on the development of integrated cellulose from other alternative natural resources (i.e. water hyacinth).



**Alia Badra Pitaloka** was born on August 2, 1978. She is a lecturer of chemical engineering at Universitas Sultan Ageng Tirtayasa. She is currently a Ph.D. student at Chemical Engineering Department Universitas Indonesia where her research focuses on the development of superabsorbent material based on celluloses and their derivate specifically CMC from water hyacinth