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Synthesis of a novel superabsorbent hydrogel by copolymerization of acrylamide and cashew gum modified with glycidyl methacrylate

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Abstract

Novel superabsorbent hydrogels were manufactured using chemically modified cashew gum (CGMA) and acrylamide (AAm) as reactants. The route for the synthesis was feasible due to the incorporation of glycidyl methacrylate (GMA) into structure of cashew gum (CG) to form the cashew gum-methacrylated (CGMA), in an appropriate mixture water-DMSO, as solvent, and using TEMED as catalyst. Thereafter, the CGMA was copolymerized with AAm yielding (CGMA-co-AAm) hydrogels. The main characteristics of raw and the modified materials are reported in this paper. ^{13}C NMR, ^{1}H NMR and FTIR spectroscopies confirmed the incorporation of vinyl groups, from GMA, into CG structure. By the spectrophotometry analyses, it was found that, ca. 82% of GMA was incorporated to the CG after 24 h of reaction. The cross-linking of CGMA or co-polymerization of CGMA with acrylamide leads to a hydrogel formation. Their gelation was characterized by FT-IR analysis. Alkaline hydrolysis at 40 °C for 3 and 4.5 h increased the water uptake (WU) capacity. Hydrolyzed CGMA-co-AAm hydrogels present higher values of WU (up to 1500) and may be classified as water superabsorbent material. Applications in agriculture, as soil conditioner, and in biomedical field, as biomaterial (scaffold) are being investigated.

Keywords: Cashew gum; Acrylamide; Methacrylated-polysaccharide; Aqueous-DMSO; Superabsorbent hydrogel

1. Introduction

Searching superabsorbent hydrogels is directly associated with the synthesis of new materials to be applied either as soil conditioner or as controlled release matrices for agrochemicals (Chatzoudis & Rigas, 1999; Kabiri, Omidian, Hashemi, & Zohuriaan-Mehr, 2003). The use of these materials in biomedical field by applying in the treatment of edemas for body water elimination has been reported by Sannino et al. (2003). Superabsorbent hydrogels are formed of highly hydrophilic cross-linked polymers, which present high water absorption (Karadag, Üzüm, & Saraydin, 2002). This propriety is due to the flexibility and hydrophilicity of the polymer network, which may be high or low, depending on chemical composition of the hydrogel (Bell & Peppas, 1995; Gutowska, Bae, Jacobs, Feijen, & Kim, 1994; Muniz & Geuskens, 2001).

For applications in soil, a superabsorbent hydrogel should present appropriate characteristics such as low cost and biodegradation (Bouranis, Vlyssides, Drossopoulos, & Karvouni, 1995; Chatzoudis & Rigas, 1998; Chatzoudis & Rigas, 1999). To synthesize a new hydrogel, we have been used the cashew gum (CG), that is a polysaccharide extracted from the cashew tree *Anacardium occidentale* (Mothe & Rao, 2000; Silva et al., 2004; Zakaria & Rahman, 1996). The cashew tree raises at semi-arid regions (de Paula & Rodrigues, 1995; Menestrina, Iacomini, Jones, & Gorin, 1998; Mothe & Rao, 1999) i.e. low water amount is enough to grow and be alive. However, it was observed in works previously performed in our research group that this raw polysaccharide is not highly hydrophilic and it is not possible to synthesize hydrogels from this raw material. An alternative to overcome this problem would be to modify the structure of CG, by reaction with glycidyl methacrylate

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GMA (van Dijk-Wolthuis et al., 1995; van Dijk-Wolthuis, Kettenes-van den Bosch, van der Kerk-van, & Hennink, 1997) The CG is composed of arabinose, glucose, glucuronic acid, galactose, mannose and rhamnose. Its structural scheme was reported by Sarubbo et al. (2000). Double bonds from GMA are attached into CG structure by that reaction (Reis, Cavalcanti, Rubira, & Muniz, 2003). This allowed to perform a co-polymerization reaction with hydrophilic vinyl monomers, as the acrylamide, AAm. It has been used in synthesis of hydrogels (Bajpai & Giri, 2003; Guilherme, Silva, Girotto, Rubira, & Muniz, 2003; Kwok, Prime, Qiao, & Solomon, 2003; Muniz & Geuskens, 2000) and on biomaterials (Yang, Mendelsohn, & Rubner, 2003) due their water affinity and their good mechanical proprieties (Bell & Peppas, 1995; Gutowska et al., 1994). Researchers have investigated superabsorbent hydrogels formed from hydrolyzed polyacrylamide (Drubovskii, Lagutina, & Kazanskii, 1994; Okay & Sariisik, 2000). These hydrogels swell up to 400 times their own dry weight (Bouranis et al., 1995). After the hydrolysis, the amides groups of the polyacrylamide are changed to carboxyl groups. The aim of this paper is to prepare and characterize superabsorbent hydrogels, associating AAm monomers and the modified CG. The methodology developed in this work for the modification of CG to obtain the CGMA and the respective GCMA-co-AAm hydrogels was recently applied for a Brazilian Patent (Guilherme, Rubira, Feitosa, & Muniz, 2004).

2. Materials and methods

2.1. Materials

The CG powder was kindly supplied by CNPCa-EMBRAPA, Brazil. The isolation process of the CG is the same used by De Paula & Rodrigues (1995) and the M_w is 2.6×10^5 g mol^{-1}. Ethanol and nitric acid, both PA, were supplied by Synth (Brazil). Acrylamide (AAm) 99% and dimethyl sulfoxide (DMSO) were purchased from Aldrich (14,866-0) and. Mallinckrodt AR®(ACS, M4948-04), respectively. *N*,*N*,*N*′,*N*′-Tetramethylethylenediamine (TEMED) 99% was purchased from Invitrogen (15524-010) and glycidyl methacrylate (GMA) 97% from Across Organics (106-91-2). Buffer solution, at pH=10, was supplied by Across Organics (25860-0010) and cerium (IV) sulfate by Vetec, Brazil (723). Membrane (JP41) having permeability to air: 55 l/s m^2 and 28 μm in average pores size was used for CG filtration.

2.2. Preparation of chemically modified cashew gum

The CG was purified by precipitation from aqueous solution pouring ethanol. Recent prepared aqueous solution of DMSO at 8.4 mmol L^{-1} concentration was added to TEMED as catalytic. After complete homogenization, it was mixed with the requested amount of GMA and stirred for 24 h at 40 °C. Amount level of TEMED, CGMA and CG used for polysaccharide modification were summarized in Table 1. The modified CG, noted as CGMA, was purified by precipitation in excess ethanol. The precipitate was dissolved in water and re-precipitated with ethanol, filtrated using low-pressure. Just after, the precipitate was lyophilized by a freeze dryer (Christ gefriertrocknungsanlagen) for 48 h and stored on dark at room temperature.

Table 1
Amount of GMA, TEMED and CG added in 10 mL of aqueous DMSO for modification of the polysaccharide

GMA (mmol)	TEMED (mmol)	Cashew gum (CG) (μmol)	Weight ratio (g CG / g GMA)
7.53	27.80	3.85	(1/1)
22.58	27.80	3.85	(1/3)
45.16	27.80	3.85	(1/6)

2.3. Synthesis of CGMA-co-AAm hydrogels

Requested amount of CGMA and AAm monomers were dissolved in 10.0 mL of distilled water. After dissolving, 0.5 mL of Cerium (IV) sulfate and 0.5 mL of nitric acid were added to obtain 2 mmol L^{-1} and 0.4 mol L^{-1}, respectively. The gelation was observed after around 45 min of heating at temperature 75 °C. Afterwards, the swollen hydrogels were hydrolyzed in buffer solution at pH 10, for 3.0 or 4.5 h. After washing of the hydrogels in abundant water for 24 h at ambient temperature, the hydrogels were dried at 60 °C for 72 h. In the present work, the CGMA-co-AAm hydrogels obtained by copolymerization reaction were labeled as [*C*/*A*/*h*], where *C* and *A* mean, respectively, the concentrations (g mL^{-1}) of CGMA and AAm in the feed solution. The *h* is the hydrolysis time, in hours. To characterize the modification of polysaccharide with GMA by FT-IR, hydrogel composed of pure CGMA was obtained. It was synthesized by the same procedure used to obtain the CGMA-co-AAm hydrogels but in absence of AAm.

2.4. FT-IR spectroscopy

FT-IR spectra of CG, CGMA, and CGMA-co-AAm hydrogels were taken on a Bomen FT-IR model MB100 spectrometer in the range from 4000 to 450 cm^{-1}. Powdered samples were prepared with KBr to make pellets. To achieve a 2 cm^{-1} resolution, 128 scans were made in each spectrum.

2.5. ^{1}H NMR and ^{13}C NMR spectroscopy

The ^{1}H NMR and ^{13}C NMR spectra were obtained by a Varian spectrometer, model Oxford 300 working at 300 MHz. The spectra of CG and CGMA were obtained using solutions in D_2O, at concentration 150 mg mL^{-1}.

The relaxation time length and the angle pulse used were 30 s and 90°, respectively, for ^{1}H NMR spectra and 30° and 1 s for the ^{13}C NMR spectra. The ^{1}H NMR and ^{13}C NMR spectra of GMA were obtained in CD_3Cl solvent. The 3-(trimethylsilyl) propionic acid-d_4 sodium salt was used as intern reference. The chemical shift was given at ppm.

2.6. Measures of GMA incorporated into CG

Amount level of GMA incorporated to CG was investigated by spectrophotometry at 308.5 nm that is the wavelength for maximum absorption of GMA dissolved in the same solvent, as blank, used for the modification of CG. It was done using a calibration curve of photometric signal, done using the blank as reference, which correlates the absorbance to the concentration of GMA in that solvent. After the precipitation of the polysaccharide with acetone, the calibration curve was then used to assign the unreacted GMA, liquid phase, as a function of time. it was collected an aliquot of 5 mL from reaction mix (200 mL) and precipitated with 10 mL acetone. Afterwards, the solvent was extracted from polysaccharide by centrifugation at least 20 min for removing the residues of polysaccharide. It is important to notice that the polysaccharide was precipitated with acetone instead of ethanol for eliminating any possibility of the reaction of GMA with this solvent. The readings of absorbance were only performed in fully transparent solutions. Every procedures, including the centrifugation and readings of absorbance, were carried out in closed recipients to avoid the loss of any component from mix. The amount of incorporated GMA was determined by difference between the initial and found concentrations of GMA.

2.7. Water uptake measurements

The water uptake capacities (WU) of CGMA-co-AAm hydrogels were determined at temperature 25 °C through the following equation

$$\mathrm{WU} = \frac{W_\mathrm{w}}{W_\mathrm{d}} \tag{1}$$

where W_w is weight of swelled hydrogel in water at each time and W_d is the weight of starting dried hydrogel (in peaces that the averaged weight is ca. 0.09 g). It was used two samples of dried hydrogel for each measure.

3. Results and discussion

There are two different pathways that could be considered for chemicals modifications involving polysaccharides when GMA is used as the modifier (Chen & Park, 2000; Ferreira, Vidal, Geraldes, & Gil, 2000; Sarubbo et al., 2000). In DMSO, an aprotic solvent, the GMA was introduced into the polysaccharide through transesterification reaction (Fig. 1a). However, using protic solvent, e.g. aqueous environment, the polysaccharide could react with GMA by opening the epoxy ring (Fig. 1b).

The modification of dextran by GMA addition has been described by van Dijk-Wolthuis et al. (1995, 1997). In those works, the dextran reacted with GMA in DMSO, as a solvent, and dimethyl amino pyridine (DMAP), as an accelerator. Using a corresponding reaction, sucrose was modified by Ferreira et al. (2000).

In the present work, the CG was reacted with GMA in aqueous-DMSO media using the TEMED as catalytic agent this has not yet been reported in the literature. As mentioned

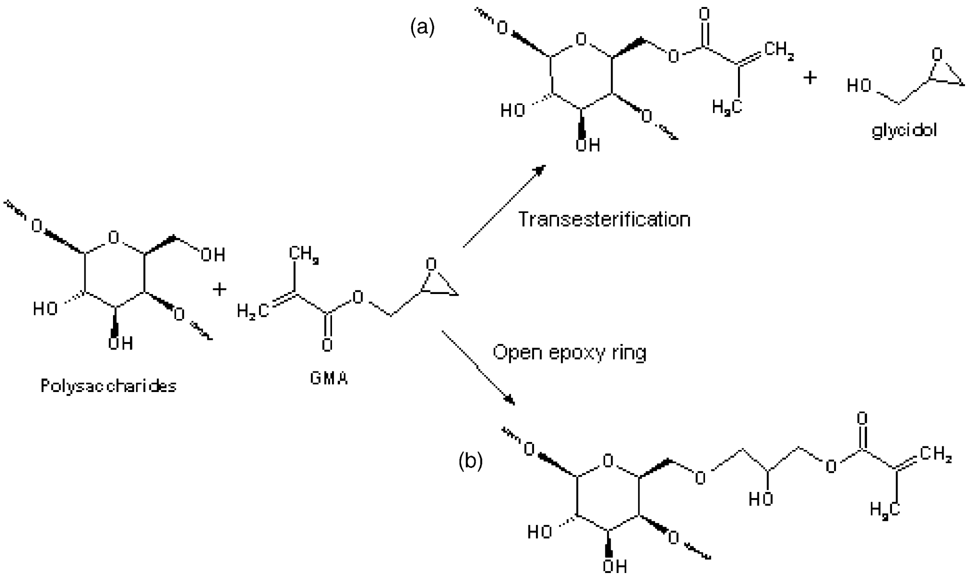

Fig. 1. Schematic of the feasible pathways of modification of polysaccharides with GMA: (a) transesterification, (b) open epoxy ring mechanism.

above, the aim of this work is the synthesis of water superabsorbent hydrogel using the CG modified with GMA. So, it is beyond of this paper the investigation, in details, whether transesterification and/or opening epoxy ring pathways occurs. For the moment, our main purpose is to incorporate vinyl groups from GMA in CG structure, and their respective characterization.

3.1. FT-IR spectroscopy analysis

The FT-IR spectra of GMA, CG and CGMA are shown in Fig. 2. The intense band that appears at 1720 cm^{-1}, in spectrum of CGMA, was attributed to C=O stretching frequency of conjugated ester groups. The weak band at ~1700 cm^{-1} in CG spectrum was assigned to C=O group from glucuronic acid, present as 6% in the original gum. It was also observed in CGMA spectra change in the shape and intensity of the bands at 1637 cm^{-1}, indicative of C=C stretching frequency. The corresponding band in CG spectrum is attributed to wavenumber of carboxylate group from glucuronic acid. The introduction of C=C groups into polysaccharide chains is confirmed by the appearance of the intense band at 813 cm^{1} in FT-IR spectrum of CGMA, indicative of the =C–H out-of-plan bending. The increase in the amount of C–O groups could be noted by the more intense band in 1200–1100 cm^{-1} range in CGMA spectrum, compared to CG spectrum one. Reaction of CG with different amount of GMA was followed through FT-IR and the spectra are shown in Fig. 3. Increasing the GMA amount in the feed solution raises the intensity of bands at 1720, 1637 and 813 cm^{-1} in the CGMA spectrum. So, the modification of CG is enhanced by increases of the amount level of GMA in the reaction vessel.

FT-IR spectra of GMA, CG, CGMA and hydrogel of pure CGMA are depicted in Fig. 4. The cross-linking reaction was characterized by the shift in the C=O stretching band, from 1720 to 1729 cm^{-1}, that indicates the lack of the conjugated ester groups. The decreases in the intensity of the band at 1637 cm^{-1} and complete disappearance of the band at 813 cm^{-1}, corresponding to the change from vinylic to methylenic carbons. Those bands are evidences of the occurrence of cross-linking reaction.

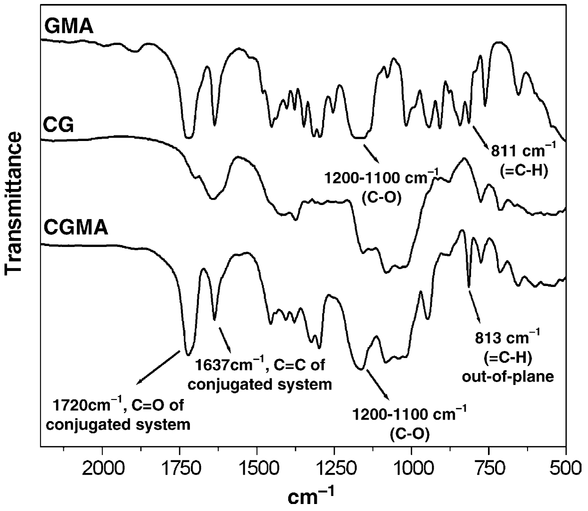

Fig. 2. Transmittance FT-IR spectra of GMA, CG and CGMA.

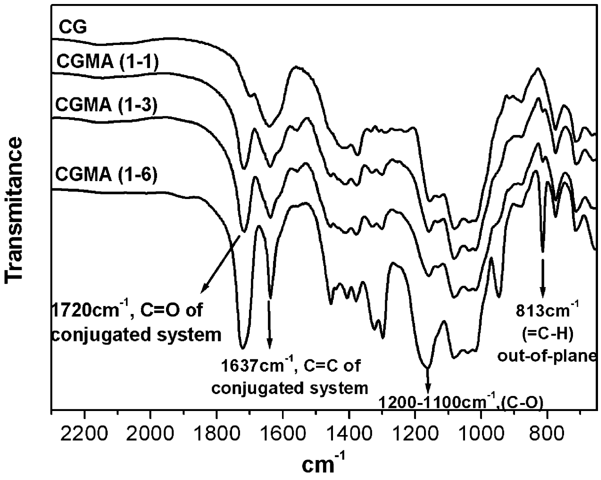

Fig. 3. Transmittance FT-IR spectra of GMA and CGMA. The labels 1:1, 1:3 and 1:6 indicate the weight ratio of CGMA and GMA used in reaction.

Fig. 5 shows the FT-IR spectra of GMA, AAm, CGMA and CGMA-co-AAm hydrogel. It was observed a broad band in the region 1780–1680 cm^{-1} in CGMA-co-AAm hydrogel spectrum, attributed to the overlap of the C=O stretching bands of esters (originated from CGMA), primary amides and N–H deformation of primary amides groups (originated from AAm), indicative of copolymerizing reaction of CGMA with AAm. The absence of the band at 813 cm^{-1} in the spectrum of CGMA-co-AAm hydrogels attributed to vinyl groups from AAm and CGMA, confirms the consumption of this group and the reaction of copolymerizing.

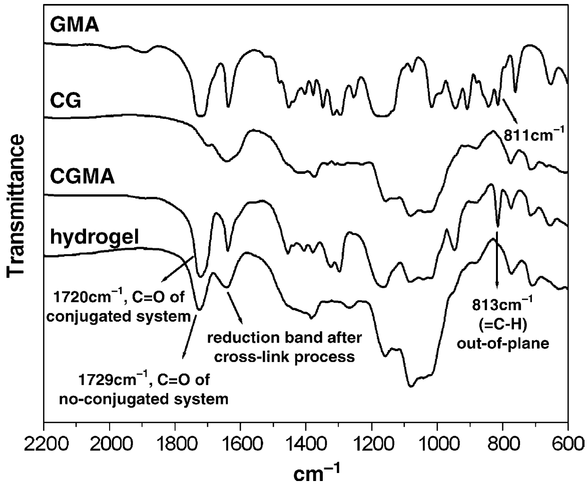

Fig. 4. Transmittance FT-IR spectra of GMA, CG, CGMA and hydrogel of pure CGMA.

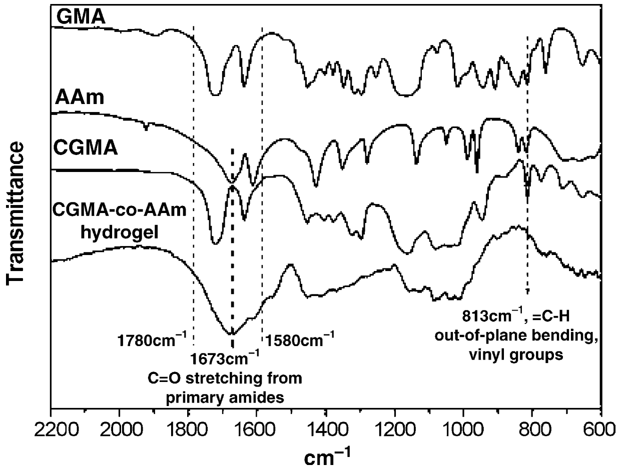

Fig. 5. Transmittance FT-IR spectra of GMA, AAm, and CGMA and CGMA-co-AAm hydrogel.

3.2. Nuclear magnetic resonance (^{1}H NMR and ^{13}C NMR)

Fig. 6 shows the ^{1}H NMR spectra of CG, GMA and CGMA. The signals observed in CGMA spectrum at δ 6.18 and δ 5.76 ppm were attributed to the *cis* vinyl hydrogen from the GMA. The signal appeared at δ 1.95 ppm was correlated to the methyl hydrogen. This finding is an additional evidence of GMA groups introduction into polysaccharide chains. Signals at δ 139 and δ 130 ppm, attributed to the vinyl carbons, and signal at δ 19 ppm, that correspond to the methyl carbons, were observed in the ^{13}C NMR spectra of CGMA, Fig. 7. The FT-IR and NMR results confirm the presence of vinyl groups in CGMA structure.

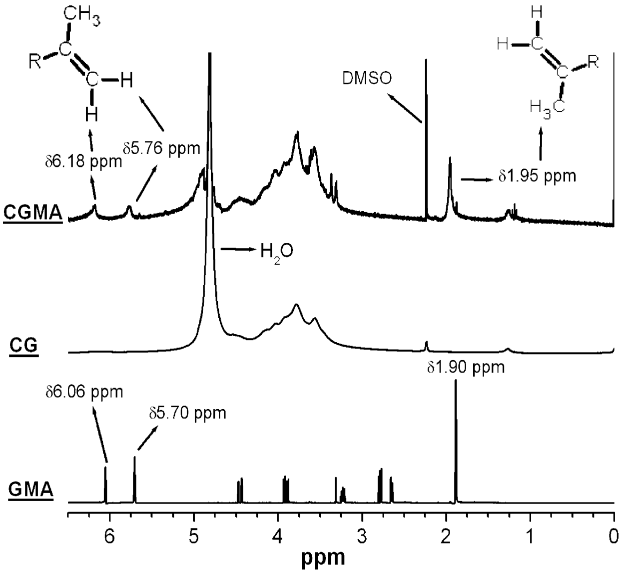

Fig. 6. ^{1}H NMR spectrum of GMA, recorded in CD$_3$Cl. Spectra of CG and CGMA were recorded in D$_2$O (methyl hydrogens: δ 1.95; vinyl hydrogens: δ 6.18 and δ 5.76 ppm).

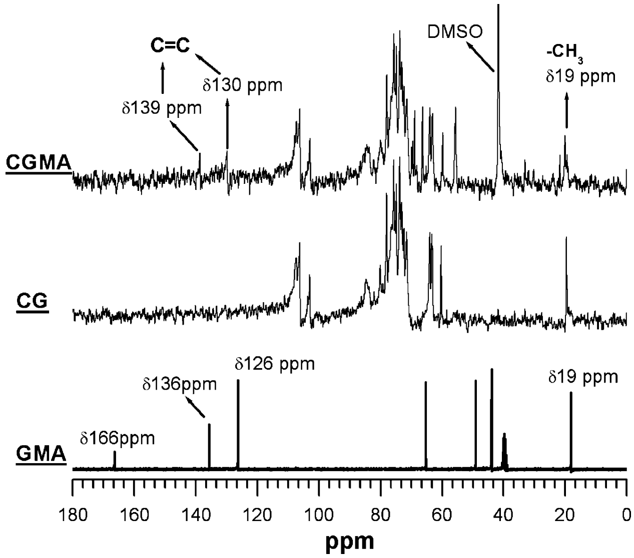

Fig. 7. ^{13}C NMR spectrum of GMA, recorded in CD$_3$Cl. Spectra of CG and CGMA were recorded in D$_2$O.(methyl hydrogens: δ 1.95; vinyl hydrogens: δ 6.18 and δ 5.76 ppm).

3.3. Amount level of GMA reacted with the polysaccharide

The extent of the vinyl groups that has been incorporated in the CG cannot be evaluated by NMR analyses. The reason for this is that the CG is a branched acidic heteropolysaccharide of complex structure that generates large signals in the ^{1}H NMR spectra and, therefore, it is not possible to determine the degree of modification by these signals. According to the literature, this parameter has already been determined by ^{1}H NMR analyses, however, the studied polysaccharides, e.g. dextran and inulin, have macromolecular structure lesser complex than the CG.

The amount of GMA incorporated into CG was obtained from linear dependence of absorbance to the concentration of GMA, as displayed in Fig. 8. The values of reacted GMA were then plotted against the time in different temperatures, which are shown in Fig. 9. It was verified that the amount of unreacted GMA, present in liquid phase, decreases with

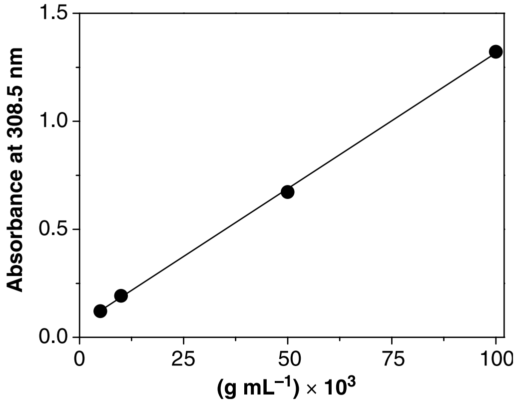

Fig. 8. Absorption curve for photometric calibration which correlates absorbance to the concentration of GMA.

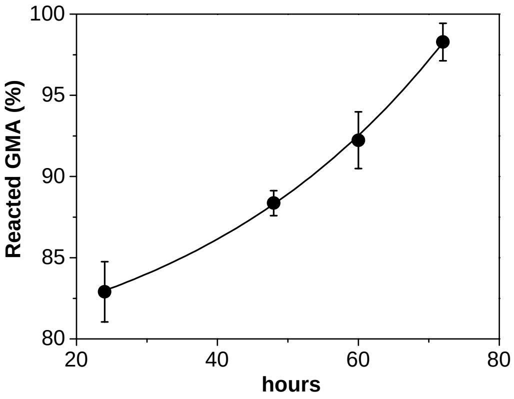

Fig. 9. Amount level of GMA reacted with the polysaccharide in dependence of time in different temperatures.

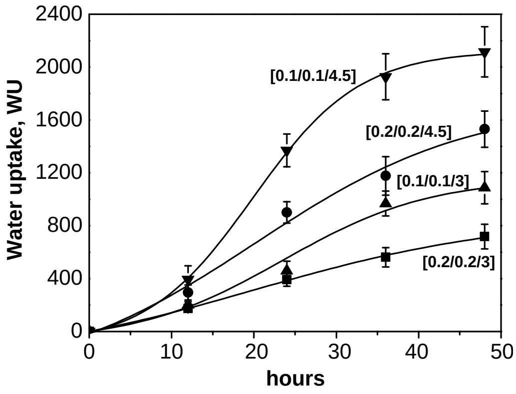

Fig. 10. Water uptake capacity for (0.2–0.2–*h*) and (0.1–0.1–3) CGMA-co-AAm hydrogels as a function of time of immersion in water.

the time. This finding is an indicative of that the amount of GMA in reaction mix is not excessive. Each value of unreacted GMA was averaged from three measures. After 24 h of reaction, it was found that ca. 18% of GMA was still present in the liquid phase. It is a strong evidence of that the difference in the initial and found amounts of GMA has been incorporated in the CG. In this reaction time, each 1 g of polysaccharide reacted with ca. 0.82 g of GMA and this value increased to 0.98 g for 72 h of reaction. From these results, it was point out that the extent of the vinyl groups that has been incorporated in the CG is above ca. 82%. As the CG reacted for 24 h, then, each 1 g of polysaccharide used to synthesis the hydrogels, 0.8 g of it reacts with the acrylamide. As the weights of CGMA and AAm used in the synthesis of hydrogels are equals, then, the content of polysaccharide in the matrix is 28%. In such case, whole molecule of GMA would be incorporated to the CG. But if the reaction occurs by transesterification pathway, it is formed glycidol as a byproduct, so only part of GMA (the methacrylate) is coupled to the CG structure. However, the reaction by open epoxy ring pathway (Fig. 1b) when GMA is used for chemical modifications of polysaccharides has been barely discussed in the literature (van Dijk-Wolthuis et al., 1995, 1997). Furthermore, such papers have been less addressed to branched acidic hetero-polysaccharides of complex structure, like cashew gum. For these reasons, we have considered in this paper the two feasible pathways for synthesis of CGMA. In this case, the percentage of CG on GCMA-co-AAm dry hydogels ranges from 28 to 35%, in weight.

3.4. Water uptake

The water absorption of CGMA-co-AAm hydrogels was investigated by water uptake capacity, WU. The hydrogels composed of pure CGMA were highly brittle, hindering their WU measurements. However, hydrogels prepared by addition the AAm and CGMA in the feed solutions presented good stability when submitted to the WU tests. This is consistent with the assumption that CGMA copolymerizes with AAm, according to the FT-IR results. In comparative experiment, the CGMA was reacted with acrylic acid using the same procedure for AAm, in order to eliminate the hydrolysis step. The hydrogels obtained in this run were extremely soft; hindering the water uptake measurements. The advantageous of reacting the CGMA with the AAm is of that water uptake capacity of hydrogels may be adjusted by the hydrolysis reaction conditions.

The curves of WU for [0.1/0.1/*h*] and [0.2/0.2/*h*] hydrogels as a function of time in water are illustrated in Fig. 10. After about 35 h immersed in water, the [0.1/0.1/3] hydrogel swell up to 1000, i.e. absorb mass of water corresponding to 1000 times its dry weight. Another relevant aspect in this hydrogel is that it is manufactured using low amount of CGMA and AAm. It was added only 0.1 g mL^{-1} of each component in the feed solution for obtaining the hydrogel. At the same hydrolysis time, the [0.1/0.1/3] hydrogels present higher WU than the [0.2/0.2/3] one. Higher is the CGMA content in the feed solution more cross-linked is the hydrogel. Obviously, an increase of the vinyl group concentration during the gelation increases the cross-linking density. It means that the cross-linking density of such hydrogels may be controlled by the concentration of CGMA and thus, allowing to control the water absorption of hydrogel.

The value WU encountered for the [0.1/0.1/4.5] and [0.2/0.2/4.5] hydrogels after 48 h of immersion in water was close to 1500 and 2000. Therefore, it may be point out that the WU of CGMA-co-AAm hydrogels is strongly dependent on the time hydrolysis. Higher is the time of hydrolysis higher is the water uptake capacity of and [0.1/0.1/*h*] and [0.2/0.2/*h*] hydrogels. The alkaline hydrolysis of AAm produces the change of amide groups to carboxylic groups. For comparison, WR measurements for the unhydrolyzed hydrogels were investigated. It was found that their WR values (not shown here) were less than 50. This value is practically insignificant when these hydrogels are hydrolyzed for 3 and 4.5 h.

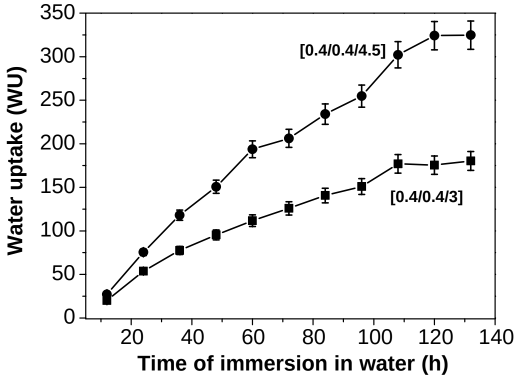

Fig. 11. Water uptake capacity for CGMA-co-AAm hydrogels as a function of time of immersion in water.

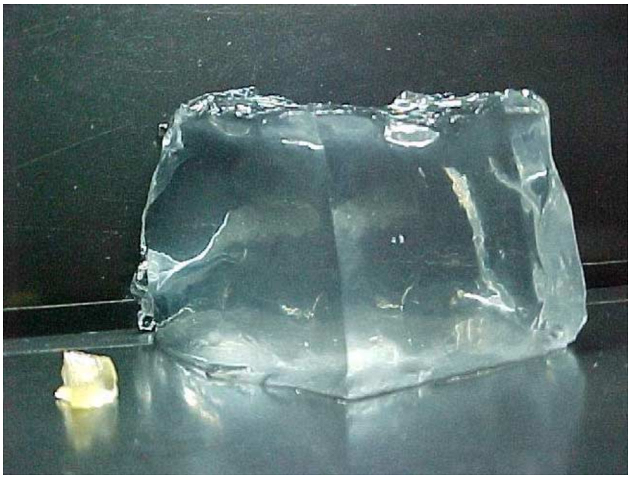

Fig. 12. Photo of (0.4–0.4–4.5) CGMA-co-AAm hydrogel, dried (left side) and swollen (right side).

According to Mahdavinia, Purjavadi, Hosseinzadeh, and Zohuriaan (2004) electrostatic repulsions among the carboxylic groups lead to WU capacity enhancement, or hydrophilicity, on hydrolyzed polyacrylamide hydrogels. The [0.1/0.1/4.5] hydrogel swells larger than the [0.2/0.2/4.5] one. However, after 48 h of immersion in water the either [0.1/0.1/4.5] hydrogel or [0.2/0.2/4.5] one were extremely soft due to the highly absorption of water. For that reason, their water uptake measurements were considered finish after 48 h.

The dependence of WU to the time of immersion in water for [0.4/0.4/4.5] and [0.4/0.4/3] hydrogels is shown in Fig. 11. In that figure, the WU of hydrogel was accompanied from 0 to 130 h of immersion in water. The hydrogel hydrolyzed for 4.5 h swelled ca. 325 times its own dry weight.

Fig. 12 illustrates the photo of the [0.4/0.4/4.5] CGMA-co-AAm hydrogel; dried (left side) and swollen ca. 325 fold its own dry weight (right side). It should be emphasized here that even highly swollen the [0.4/0.4/4.5] CGMA-co-AAm hydrogel maintains good mechanical properties.

4. Conclusions

The cashew gum (CG) was modified by addition of glycidyl methacrylate (GMA) using aqueous-DMSO, as solvent, and TEMED, as catalyst. ^{13}C NMR, ^{1}H NMR and FT-IR spectroscopies confirmed the incorporation of vinyl groups, from GMA, into CG structure. The cross-linking of pure CGMA and its co-polymerizing with acrylamide lead to hydrogel formation, characterized by FT-IR analyses.

After hydrolysis, CGMA-co-AAm hydrogels became more water-superabsorbent and swell up about 1500 times their own dry weight. Their water uptake capacity was highly dependent on the time of hydrolysis, CGMA and AAm amounts. Applications of hydrolyzed CGMA-co-AAm hydrogels in agriculture, as soil conditioner, and in biomedical field, as biomaterial (scaffold), are in course in our laboratory. Also, these hydrogels have characteristics to be used as absorbent-package (for food industries), as absorbent for personal care.

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