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REGENERATED KENAF CORE CELLULOSE PRODUCTS PREPARED VIA PRE-COOLED METHOD

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ABSTRACT

Cellulose was prepared from kenaf core (KC) powder by a series of bleaching and alkali treatment processes. The extracted cellulose was dissolved using different the alkali/urea solvents (NaOH/urea and LiOH/urea) via pre-cooled method to form cellulose solution. Cellulose solution produced from KC cellulose was regenerated to form cellulose hydrogel. Hydrogel produced from cellulose with lower cellulose content possessed higher porosity, hence increased the water swelling of the hydrogel. Cellulose solution also was mixed at different percentage of polyvinyl alcohol (PVA) to produce regenerated transparent membranes. The addition of 10 % of PVA has increased the porosity of the regenerated membrane. The regenerated cellulose/PVA membranes were color-printed using a laser printer and the membranes were recycled, in which the printed ink was removed using distilled water and printed repeatedly for several times. Crosslinking of the KC cellulose solution with aldehyde based crosslinker (glyoxal and glutaraldehyde (GA)) were studied using different percentages of the aldehyde. Tensile strength of the membranes increased with increasing crosslinker percentages. However, the porosity decreased due to the crosslinked cellulose structure. Moreover, magnetite (Fe₃O₄) nanoparticles was synthesised and were embedded in cellulose solution for the preparation of magnetic cellulose membrane (MCM). The formed cellulose solution was mixed with Fe₃O₄ nanoparticle at different percentage of Fe₃O₄ i.e., 10, 20 and 30 % to produce regenerated MCM. From XRD result, it shows that the CrI of cellulose decreased with the addition of magnetite nanoparticles. However, it is obtained from VSM test that the magnetic properties of the regenerated membrane increases as the percentages of magnetite nanoparticle increases.

Keywords: Cellulose hydrogel; cellulose membrane; crosslinking; kenaf cellulose; pre-cooled method
INTRODUCTION

Kenaf is one of the important industrial crops cultivated in Malaysia after wood and bamboo (Mohd Edeerozey et al. 2007). Recently, various studies have been reported in utilizing kenaf for different applications due to its advantages such as renewable resources, fast growing rate, and annually available. Kenaf core with shorter fiber length are less used compare to kenaf bast which gives advantages to the pre-cooled system using alkali/urea solvent. This will ease the cellulose dissolution to be occurred. Alkali/urea system is used due to the green process which promises non-toxic process, cheaper, biodegradable and rapid dissolution process (Zhang et al. 2010). The produced regenerated cellulose products were used in the form of fibers, films, powders, beads, and membranes. The cellulose morphology changes after dissolution and regeneration process occurred where the processes have affected the properties of the regenerated cellulose produced.

In this study, cellulose was isolated from kenaf core powder undergoes bleaching and alkali treatment. The extracted cellulose was dissolved in LiOH/urea solvents. Cellulose hydrogel was produced using different cellulose content. Regenerated cellulose membrane was mixed with PVA solution to form transparent and printable membrane. Crosslinked cellulose membrane with aldehyde based (glutaraldehyde and glyoxal) were produced to study crosslinking effect on chemical, physical and mechanical properties of the membrane. Magnetite was synthesized using in-situ method and cellulose was dissolved in NaOH/urea solvent to produce cellulose solution for the production of magnetite cellulose membrane (MCM) at different magnetite content. The properties of hydrogel and membranes produced were characterized using UV-Vis, SEM, FTIR and XRD. Water uptake of both hydrogel and crosslinked membrane was also investigated.

METHODOLOGY

Kenaf cellulose (KC) (3 wt %) were dissolved in LiOH/urea aqueous solution and frozen at −13 °C. Then, ECH (5 %) was dropped wisely into the cellulose solution and stirred until the formation of hydrogel achieved. Cellulose/polyvinyl alcohol (CS/PVA) solutions were prepared in different ratios of PVA and CS. The CS/PVA solution was stirred for 30 min and centrifuged and cast on a glass plate and coagulated in 5% H2SO4 solution to form the CS/PVA film. Two different types of crosslinker (glutaraldehyde (GA) and glyoxal) were used as coagulant and prepared at different percentages of crosslinker. The prepared CS was cast on a glass plate and immersed in different coagulating baths. The crosslinked membrane produced were freeze dried and air dried for further characterization. Magnetite nanoparticles were synthesis by using of Fe(II), Fe(III) and NaOH dissolved in distilled water at 50 to 60 °C. The solution turned black color after the addition of NaOH which indicates the formation of magnetite. To form magnetite cellulose membrane (MCM), the synthesized magnetite was added into the cellulose solution at different magnetite content (10, 20 and 30 %) and stirred homogeneously, cast on a glass plate and coagulated in acid bath to form MCM.

RESULTS AND DISCUSSION

Production of Cellulose Hydrogel

Figure 1 shows the SEM images of hydrogel prepared using different percentage of cellulose. The sample coded as HKCR2, HKCR3 and HKCR3.5 represent the 2 %, 3 % and 3.5 % of KCR cellulose dissolved in LiOH/urea solvent to form hydrogel respectively. It shows clearly that by increasing the cellulose content from 2 to 3.5 %, the average pore size of the hydrogel has decreased. This may be due to the entanglement between the cellulose chain is greater at higher cellulose content (Chang et al. 2010).
Figure 1 SEM images of the cross-sections of the hydrogels (a) HKCR2 (b) HKCR3 and (c) HKCR3.5.

Figure 2 shows the kinetics of water uptake (WU) by the hydrogel after freeze dried. The WU at higher cellulose content is less than the WU at lower cellulose content. The graph shows that the rate of WU is faster at the first 10 min and increase gradually until 80 min. Above 80 min of exposure, the increasing of WU has reached the equilibrium point in which the hydrogels has absorbed maximum of water. Increase the time after the saturation process will not increase the percentage of WU.

Figure 2 Rate of water uptake of cellulose hydrogel (a) HKCR2, (b) HKCR2.5, (c) HKCR3 and (d) HKCR3.5

Production of Cellulose Membrane
Figure 3 shows the morphology of the top side (contacting the coagulant) of the membranes casted on glass plate at different % of PVA. It is observed that the porosity of the membrane’s surface increased with increasing of PVA content (Sawatari & Kondo 1999).

Figure 3 SEM images of the top side of membranes having CS/PVA in ratio (a) PVA0, (b) PVA1, (c) PVA5, and (d) PVA10

The scotch tape test is employed for estimating the adhesion strength of the ink on membranes. Figure 4(a) shows the printed membrane of PVA0 and PVA10; the ink on both printed membranes was removed using the tape
test method. As can be seen in Figure 4(b) and 4(d), the ink was easier be removed from PVA10. This may due to the pore size of PVA10, which is larger than that of PVA0. The pore size will affect the ink detachment because larger pore size makes the ink detachment easier when contact with water.

![Figure 4 Photograph of (a) printed PVA0 membrane, (b) ink detached from PVA0, (c) printed PVA10 membrane, and (d) ink detached from PVA10](image)

Figure 5(a) shows the water uptake (WU) of the membranes against percentage of crosslinker for both GA and glyoxal crosslinked cellulose membranes. It shows that as percentage of crosslinker increased, the value of WU has decreased. Cellulose membrane without crosslinker (DI) has the highest value of WU which is 256 % and reduced by 27 % and 26 % for both GA and glyoxal respectively at 20 % crosslinker. Figure 5(b) shows the pore volume ($V_p$) of cellulose membrane without crosslinker, GA crosslinked cellulose membrane and glyoxal crosslinked cellulose membranes at different crosslinker percentages. The $V_p$ of DI membrane is 2.54 cm$^3$g$^{-1}$ and the value has decreased to 1.64 and 1.93 cm$^3$g$^{-1}$ which is decreased for both GA20 and GLO20 respectively. These results correspond to the water uptake where crosslinking process decreased both $V_p$ and WU. This is due to the movement of molecular chain has been restricted by the crosslinking process (Li et al. 2014).

![Figure 5 (a) Water uptake and (b) pore volume of cellulose membranes crosslinked with GA and glyoxal against percentage of crosslinker](image)

Figure 6(a) and (b) show the stress versus strain curves of materials tested. As can be seen from the test, the higher the crosslinker content the higher the strength of the membranes. DI membrane possessed the lowest tensile strength which is 56 MPa. However, for glyoxal and GA crosslinked cellulose membranes, both show increment in tensile strength with increasing the crosslinker percentage up to 20 %. This has resulted in the increase in tensile strength up to 68 MPa and 75 MPa for both glyoxal and GA crosslinked cellulose membranes.

![Figure 6(a) and (b) Stress versus strain curves of materials tested](image)
Figure 6 Stress-strain curve of cellulose membranes crosslinked with GA and glyoxal against percentage of crosslinker

Figure 7 shows the hysteresis loops of magnetic cellulose membrane (MCM) at different magnetite content i.e., 10%, 20% dan 30% measured under VSM. MCM show that the membranes possessed ferromagnetic properties where there was a linear relationship between the magnetic properties and magnetite content. Therefore, increasing the magnetite content up to 30%, saturation magnetization (Ms) becomes greater from 5.5 emu/g (MCM10) to 25.6 emu/g (MCM30). Figure 8 shows the XRD pattern of CM where three main diffraction patterns at 2θ = 12.4°, 20.3° and 22.2° which corresponding to the cellulose II crystal at plane (1 1 0), (1 1 0) and (2 0 0), respectively. Meanwhile, the XRD pattern for Fe₃O₄ show six diffraction peaks at 2θ = 30.19°, 35.58°, 42.91°, 53.46°, 57.24° and 62.63° respectively. These peaks correspond to the crystalline plane of Fe₃O₄ at (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0). However, the crystallinity of the samples were decreased as increased the magnetite content from 71.4 % for CM to 44.7 % for MCM30.

Figure 6 Hysteresis loops of magnetic cellulose membrane at different magnetite content

Figure 7 XRD patterns of cellulose membrane and magnetic cellulose membrane at different magnetite content
CONCLUSION
In summary, cellulose also can be extracted from kenaf core and others biomass plants. The extracted cellulose from biomass still can be a new natural biopolymer sources. This research used a low-cost production of regenerated cellulose materials and can be formed into biodegradable products. Research in regenerated cellulose materials using natural biopolymers can be extended into more applications such as membrane filtration for wastewater treatment, hydrophobic properties for water resistant products, coating and etc.

REFERENCES


