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Research Article



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Green superabsorbent hydrogel derived from activated charcoal and glycerol with maleic acid as a cross-linker

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ABSTRACT

Superabsorbent hydrogels characterize a set of polymeric materials with three-dimensional structures capable of absorbing large amounts of water due to their hydrophilic functional groups on their surface. Their application in industries, agriculture, and the environment is of primary significance. This study reports the synthesis and characterization of green superabsorbent hydrogels derived from activated charcoal. The process involved a polymerization reaction between activated charcoal (AC) with glycerol (G) using sodium hydroxide as an initiator in the absence and presence of maleic acid as a crosslinker to synthesize HCG-1 and HCG-2 superabsorbent hydrogel respectively. Characterization of the hydrogels was done using Fourier transform infrared (FT-IR) spectroscopy, scanning electron microscope (SEM), and X-ray diffraction (XRD). Optimization conditions were done by synthesizing hydrogel with varying dosages of both activated carbon and maleic acid as well as swelling time. The FT-IR results showed the appearance of strong sharp peaks at 1591.34 cm^{-1} and 1400.28 cm^{-1} in HCG-1 associated with $-COO^-$ symmetric stretching and asymmetric bending vibrations, indicating interlink between reacting monomers. A new absorption band at 1639.48 cm^{-1} associated with $-COO^-$ bending in non-conjugated ester indicates ester-crosslink in HCG-2 hydrogel. XRD analysis showed a phase shift from semi-crystalline to crystalline structure upon crosslinking. SEM analysis showed a crystalline intact, rigid structure without voids and pores on its surface in HCG-1 compared to the smooth irregular pores and lamina structure observed in HCG-2 hydrogel. The dosage ratio of AC: G: maleic acid of 8:5:1 produced hydrogel with an optimal water absorption capacity of 1255.80±0.70%. Maleic acid was found to improve the water absorption capacity of the superabsorbent. The study is an eye opener towards the application of biodegradable hydrogels in agriculture, especially in semi and arid regions.

KEYWORDS: Activated charcoal, Characterization, Crosslinking, Glycerol, Superabsorbent hydrogel

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INTRODUCTION

Superabsorbent hydrogel is a three dimensional lattice polymer material with hydrophilic functional groups in its network. They are biodegradable crystalline polymers that can absorb and retain water at least 400 times its original weight and avail at least 95 percent of the stored water for absorption by crops (Gandini *et al.*, 2016). Hydrogels are commonly prepared from polar materials and can be classified as physically or chemically cross-linked gels. In the first case, the networks are held together by physical forces, including ionic, H bonding, or hydrophobic forces, while in the second case, the gel has covalently crosslinked networks. Superabsorbent hydrogels find their applications mostly in technological fields including medicine (Koetting *et al.*, 2015), agriculture and horticulture (Senna *et al.*, 2015), food packaging (Farris *et al.*, 2009), and wastewater treatment (Khan & Lo, 2016).

Most of the hydrogels reported in the literature are synthetically prepared or from petroleum by-products, crosslinked with organic cross-linkers (Guilherme *et al.*, 2015). Mostly, synthetic cross-linkers used in super-absorbent hydrogels are poly (vinyl alcohol) (Martens *et al.*, 2003), poly (amido-amine) (Ferruti *et al.*, 2005), polyacrylamide (Gao *et al.*, 2007), poly (N-isopropyl acrylamide) (Nayak *et al.*, 2004) and poly (ethylene glycol) (Nagahama *et al.*, 2008). These are non-biodegradable and are bound to remain in the environment for long. This becomes the baseline for researchers towards synthesizing superabsorbent hydrogels from locally available renewable green materials which are cost-effective and degradable, especially from charcoal, starch and chitosan (Mahmoudian & Ganji, 2017). Hydrogels from these materials have the capacity to absorb water more than 20 times their weight and hence referred to as superabsorbent (Abedi-Koupai & Sohrab, 2004). This work aims towards synthesizing superabsorbent through a polymerization reaction between carboxylate and hydroxyl functional groups in AC and glycerol monomers respectively. Maleic acid was used as a binder molecule through an ester-crosslinkage between polymer units of activated charcoal

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glycerol polymer (HCG-1) to form a gel (HCG-2). The resulting hydrogel has increased hydrophilic groups as well as crosslinker points resulting in increased water absorption capacity. The low cost of monomeric materials coupled with the high swelling capacity of synthesized superabsorbent hydrogels has attracted more emphasis in the field of agriculture (Kasimu *et al.*, 2022).

MATERIALS AND METHODS

Reagents and Chemicals

The Coconut shells were obtained from Mombasa County - Kenya and transported to the Kenyatta University laboratory. Glycerol, potassium manganate VII, Sodium hydroxide, and sulphuric acid were obtained from Kenya Science Chemical Limited (Kenya), while maleic acid was purchased from Sigma Aldrich Company (Germany).

Preparation of Activated Charcoal

The synthesis technique of activated charcoal was adopted from Papita (2010). Pieces of dry coconut shell were placed into a 20 L aluminum container and excess air was removed by warming. The container was then tightly closed and heated at a temperature of 473 to 553 K for 8 hours after which cooling was done for 12 hours, and the container was opened to obtain fully carbonized charcoal (Papita, 2010). The charcoal was crushed and ground into powder using a Sheller machine (Honda ESB 501). Accurately weighed 200 g of the powdered charcoal was mixed with 500 mL of 2 M solution of potassium permanganate in a 1 L Erlenmeyer flask. The mixture was allowed to stand for 12 hours for carbon to oxidize. Oxidative reaction introduces oxygen functional groups on the surface of carbon materials to facilitate polymerization reaction with hydroxyl groups of glycerol (Meng & Ye, 2017). The mixture was filtered and the residue washed with deionized water to remove the purple color of the oxidizing agent. The residue was air-dried until a constant weight was attained to obtain activated carbon then coded (AC).

Synthesis of Hydrogel from Activated Charcoal and Glycerol (HCG-1)

The synthesis of HCG-1 from AC and glycerol was adopted from Lakshmi (2011). An electronic weighing balance (CZ 200) was used to accurately weigh 80.0 g of powdered oxidized carbon which was transferred into 1000 mL Erlenmeyer flask. The addition of 200 mL of distilled water was done as heating and stirring of the mixture continued until carbon suspensions were formed.

To the boiling carbon suspension, 37.5 mL of 13.6 M glycerol and 10 mL of 5.0 M Sodium hydroxide were added with stirring as monomer and activator respectively. The resultant mixture was then heated until it became viscous, after which it was allowed to cool at room temperature to form 123 g of solid hydrogel polymer.

Synthesis of Cross-linked Superabsorbent Hydrogel (HCG-2)

The process of HCG-2 synthesis was carried out by preparing HCG-1 as per the procedure described. To the viscous gel obtained, 25.0 mL of 3.5 M maleic acid was added with stirring. The stirring and heating of the mixture was done continuously at a temperature of 373 K to 423 K until a viscous gel was formed followed by cooling to obtain 125 g solid HCG-2 superabsorbent hydrogel. Figure 1 shows the preparation scheme of HCG-2 hydrogel. The structure of activated charcoal was adopted from (Sugumaran *et al.*, 2012).

Characterization of the Superabsorbent Hydrogel

Fourier Transform Infrared (FT-IR) analysis of the superabsorbent hydrogels

The procedure was adopted from Krauklis *et al.* (2018) to determine functional groups present in the superabsorbent hydrogels. The crystals of the superabsorbent hydrogels HCG-2 and HCG-1 were dried in the open air for 12 hours until they attained constant weight. one mg of each of the superabsorbent hydrogel was mixed with 25 mg of dry spectroscopic grade potassium bromide. The mixtures were ground using pestle and mortar to form a fine powder. The powder was compressed into thin pellets then functional groups were determined using FT-IR (Shimadzu IR Tracer-100) at a wavelength range of 4000-200 cm^{-1} (Hammond *et al.*, 2005).

Phase composition analysis of the superabsorbent hydrogels

The procedure for phase composition of the hydrogels (HCG-1 and HCG-2) was adopted from Aikawa *et al.* (1998). Analysis was done using XRD (model Hossein Beygin) at scattering angle (2θ) ranging from 10 to 90° and at a scan rate of 5°/min.

Microstructural analysis of the superabsorbent hydrogels

The surface morphology of (HCG-1 and HCG-2) superabsorbents was determined by soaking them in buffer solutions of pH 12 for 8 hours. Filtration of the mixture was done using filter paper no 42 and residue dried in the oven at 40 °C until a constant weight was obtained. Dried samples were coated with gold to become conductive before placing them in a SEM chamber (ZEISS SUPRA 60) at an accelerating voltage of 250 kV to obtain micrographs (Zhu *et al.*, 2017).

Equilibrium Water Content (EWC)

The procedure for determining equilibrium water content (EWC) was adopted from (Zhu *et al.*, 2017). Accurately weighed 2.0 g of superabsorbent hydrogel polymers were put in polyester bags followed by determining their weight then recorded as (Q_s). The polyester bags containing hydrogels were then immersed in 500 mL of distilled water for 24 hours to swell. After the swelling period, the samples were removed carefully and excess water on the surface of the polyester bags was removed using filter paper.

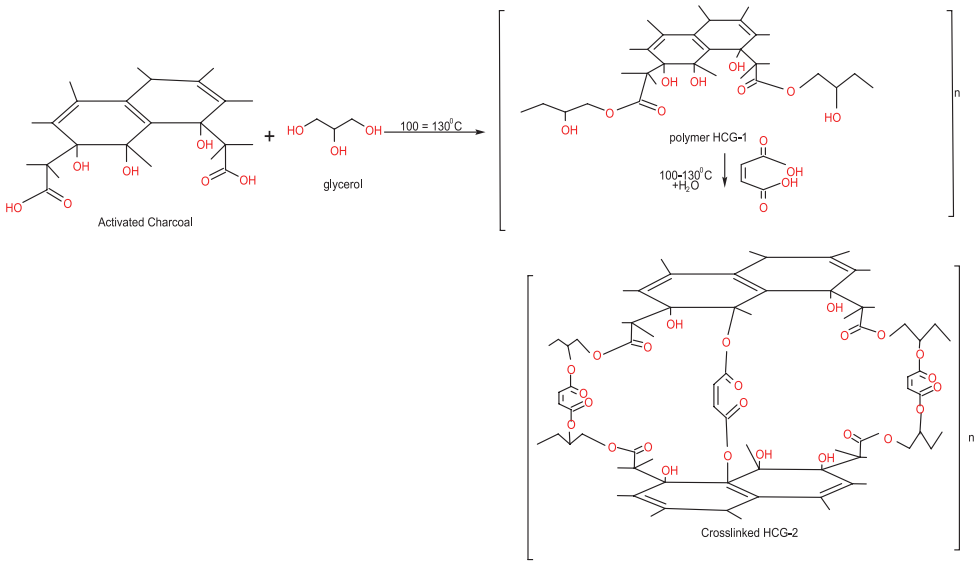


Figure 1: Scheme of preparation of HCG-1and HCG-2 hydrogel

The weight of the fully swollen superabsorbent was determined and recorded as (Q_d) (Zhu *et al.*, 2017). Equation 1 was used to determine the equilibrium water content.

$$EWC(\%) = \frac{Q_s - Q_d}{Q_d} \times 100 \qquad 1$$

Where Q_s is the initial weight of hydrogel before swelling and Q_d is the final mass of hydrogel after swelling

Effect of Activated Charcoal Dosage on Swelling Capacity of the Hydrogel (HCG-2)

The hydrogels were prepared by varying dosages of activated charcoal from (1, 2, 4, 6, 8, and 10 g) while maintaining the mass of glycerol at 3.75 mL 13.6 M. Maleic acid 2.5 mL 3.5 M and 1 mL 5.0 M sodium hydroxide was added as cross-linker and initiator respectively. The swelling capacity of the superabsorbent was determined using Equation 1 by immersing 2.0 g of each prepared sample in 500 mL of deionized water for 24 hours.

Effect of Maleic Acid Dosage on the Swelling Capacity of the Hydrogel

The effect of the maleic acid dosage was studied by varying dosage (1.25, 2.5, 3.75, 5.0, 6.25, and 7.5 g) while maintaining the dosage of activated carbon as 6 g, the volume of glycerol as 3.75 mL 13.6 M, and sodium hydroxide as 1 mL 5.0 M. Accurately weighed 2.0 g of each prepared sample was then immersed in water for 24 hours to determine the swelling capacity using Equation 1.

Effect of Contact time on the Swelling Capacity of the Hydrogel

The effect of contact time on swelling of superabsorbent hydrogels HCG-2 was studied by varying contact times (0.5, 1, 2, 4, 6, 12, and 24 hours). 2.0 g of each hydrogel prepared at the optimum ratio of AC: G: MA of 8:5:1 was immersed in 500 mL of distilled water for a specified contact time. The samples are then periodically removed after the expiry of contact time and their new mass is determined. The swelling capacity was then determined using Equation 1.

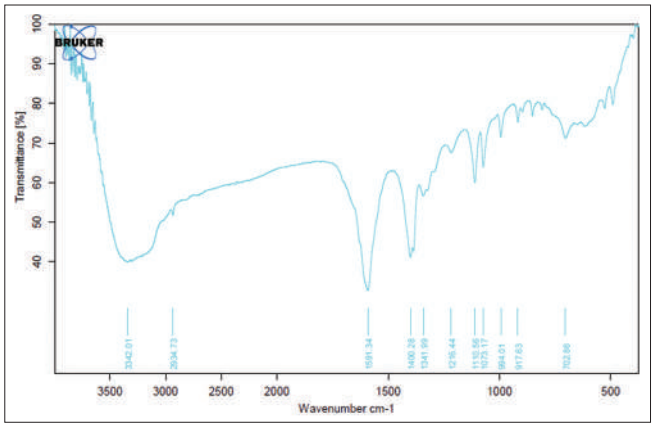


Figure 2: FT-IR spectrum of HCG-1 uncross-linked

RESULTS AND DISCUSION

Fourier Transform Infrared of HCG-1 and HCG-2 Superabsorbent Hydrogel

FT-IR analysis was carried out to identify the functional groups present in the superabsorbent before and after cross-linking. Figure 2 shows the IR spectrum of the uncross-linked hydrogel (HCG-1).

The spectra shows a broad peak at 3342.01 cm^{-1} associated with bending vibration of –OH on the surface of activated charcoal while the C-H asymmetric stretch of methylene at 2934.73 cm^{-1} (Viera *et al.*, 2007). Strong peaks at 1591.34 cm^{-1} and 1400.28 cm^{-1}

are accredited to symmetric stretching and asymmetric bending vibration bands of COO^- respectively. Consequently, C–O stretch vibration, alkyl substituted ether C–O stretching vibration and C– O– C stretch of ring ether vibration appeared at 1341.99 cm^{-1}, 1110.56 cm^{-1} and 1073.17 cm^{-1} respectively. This shows a successful ester polymerization reaction between AC and glycerol forming polymer units of (HCG-1). Further small intense peaks at 1216.44 cm^{-1} show the availability of –OH bending groups in phenol (Coma *et al.*, 2003).

Figure 3 shows the FT-IR spectrum of HCG-2 superabsorbent hydrogel after crosslinking with maleic acid. New active peaks rise at 2108.49 cm^{-1}, 1639.48 cm^{-1}, 1409.36 cm^{-1}, 1255.84 cm^{-1} and 619.57 cm^{-1} associated with -CN bending, $-COO^-$ bending in non-conjugated ester, phenol –OH stretch in ester, aromatic ether stretching and alcohol -OH out of plane bending. This is proof of ester crosslinkage between polymer units of HCG-1 to form crosslinked HCG-2 superabsorbent hydrogel (Coma *et al.*, 2003). In addition, Figure 2 shows a significant shift in wavelength as compared with Figure 3, alcohol –OH stretching mode shifts from 3342.01 to 3404.75 cm^{-1}, asymmetric C-H stretch shifts from 2934.73 to 2966.33 cm^{-1}, symmetric stretching of $-COO^-$ shifts from 1591.34 to 1527.33 cm^{-1}. Clearly, this shows maleic acid bound polymer units of HCG-1 forming a crosslinked three-dimensional structure of HCG-2. Consequently, alkyl substituted ether C–O stretch vibration shifts from 1110.56 to 1117.79 cm^{-1} while H bond deformation shifts from 702.86 to 745 cm^{-1}. However, IR spectrum peaks in Figure 2 at 1400.28 cm^{-1}, 1341.99 cm^{-1}, 1073.17 cm^{-1}, 994.01 cm^{-1} and 917.63 cm^{-1} disappeared completely. This may be due to hydrogen bond formation, intramolecular and intermolecular hydrogen bond formation in HCG-2 hydrogel (Wingerson, 2002).

Phase Composition of Uncross-linked HCG-1 and Cross-linked HCG-2 Hydrogel

Figures 4 and 5 represents analysis of the diffraction patterns of the uncross-linked HCG-1 and cross-linked HCG-2 hydrogel respectively. The sharp peaks represent the crystalline phases while the hallo/broad semi crystalline phase.

The semi crystalline nature of uncross linked HCG-1 hydrogel is highly noticeable due to the presence of a broad halo peak at

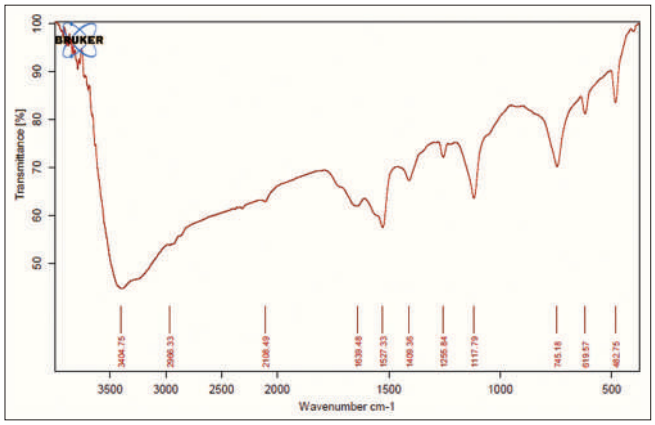


Figure 3: FT-IR spectrum of HCG-2 cross-linked

angles between (2θ) 16-30 and few crystalline peaks at 39.37, 42.52, 50.09 (Varaprasad *et al.*, 2010). Upon crosslinking with maleic acid, the polymeric hydrogel was converted to crystalline as shown in Figure 5.

The phases changed significantly as the 2θ angle peaks of HCG-1 at 39.37, 42.52, 50.09, 59.96 and 68.01° disappeared as compared with HCG-2. This may be attributed to the polymerization reaction initiated by cross linking between polymer units of HCG-1 to form the crystalline phase of HCG-2 (De Silva *et al.*, 2007). The sharp well-resolved new peaks that appeared on HCG-2 at (2θ) 18.21, 21.59 and 25.48 are associated with crystalline phases of ($CaC_2O_4.H_2O$), graphite and unburned carbon respectively (Pal & Pal, 2006; Bouchelta *et al.*, 2008). Moreover, the increased number of sharp peaks in HCG-2 is proof of crystalline phase formation in addition to the binder (Hajimohammadi *et al.*, 2011). The diffraction peaks at 2θ between 62.88 and 77.11 in HCG-2 were associated with face-centered cubic packing of graphite while angle 2θ at 28.8 was due to graphite peak arising on tubular structure of carbon atoms was clear proof of crystalline structure (De Silva *et al.*, 2007; Bouchelta *et al.*, 2008).

Scanning Electron Microscope (SEM) of HCG-1 and HCG-2 Superabsorbent Hydrogel

From the micrographs, it is clear that HCG-1 showed a crystalline intact and rigid structure without voids and pores on its surface (Figure 6a). This may be associated with the conditions that polymerization between activated charcoal and glycerol was successful (Mohan *et al.*, 2010). However, after crosslinking with maleic acid HCG-2 showed surface modification different from that of HCG-1. A well-developed fibrous with smooth irregular pores and lamina structural nature occurred on the surface of the powdered HCG-2 superabsorbent hydrogel as shown in Figure 6b. This clearly showed an ester crosslink between polymer units of HCG-1 with maleic acid forming a 3-dimensional crystalline structure (HCG-2) polymer (Buikliskii *et al.*, 2012).

Effect of Amount of AC used on the Swelling Capacity of Hydrogel (HCG-2)

Figure 7 shows the mean percentage swelling capacity attained when 2.0 g of prepared superabsorbent hydrogel was immersed in distilled water.

From Figure 7, it is evident that increasing the amount of AC in synthesized HCG-2 hydrogel from 1.0 g to 6.0 g increased the percentage swelling from 390.43±0.15 to 1020.13±0.75%. This may be due to the high ionization constant of increasing carboxylate groups, the hydrophilic properties of the gel in deionized water, and the improved chain expansion of the gel (Zain *et al.*, 2018). However as the dosage of AC increases from 6.0 to 10.0 g, the swelling capacity decreases from 1020.13±0.75 to 850.63±0.76%. This may be attributed to a highly branched arrangement which lowers the physical entanglements in the structure of HCG-2 hydrogel as well as a probable breakdown

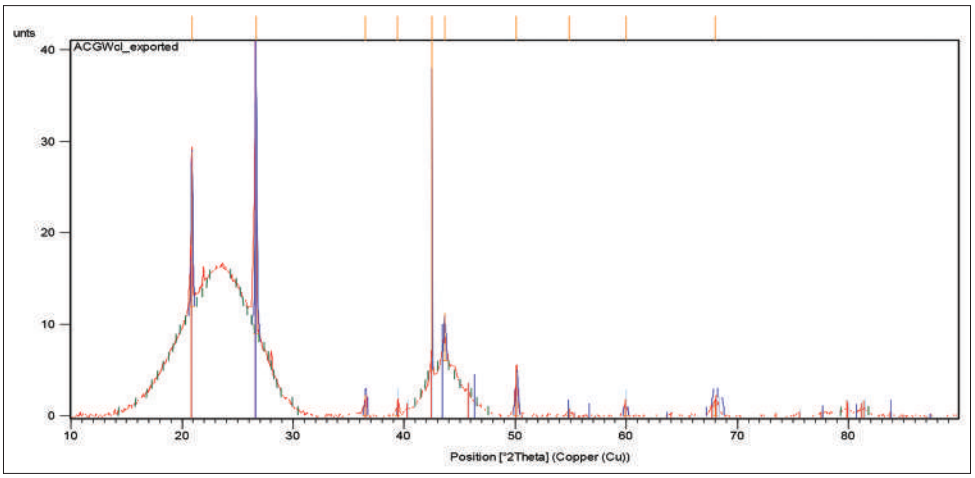


Figure 4: Powdered diffraction pattern of uncross- linked HCG-1

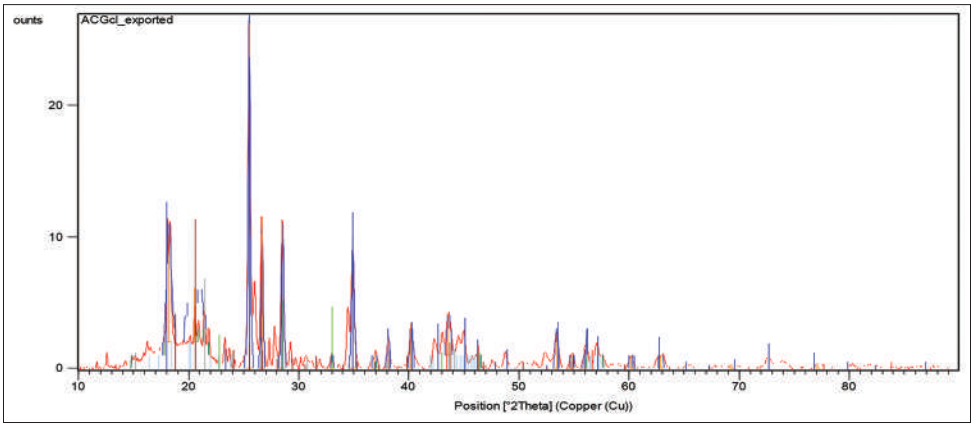


Figure 5: Powdered diffraction pattern of cross-linked HCG-2

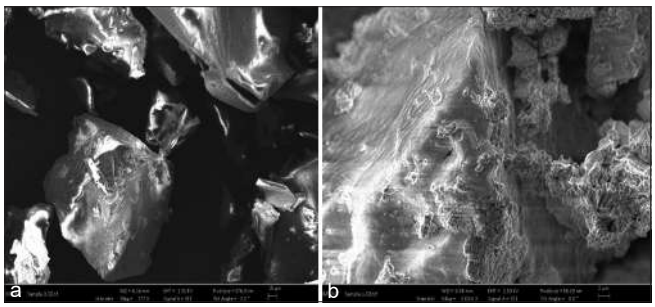


Figure 6: SEM micrographs of the (a) Uncross linked HCG-1 and (b) cross linked HCG-2 hydrogels

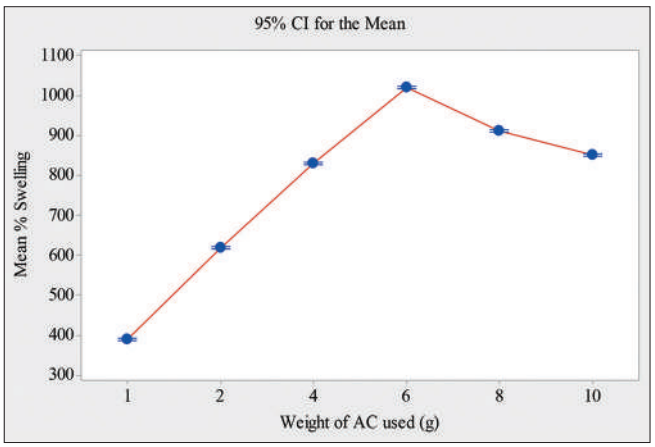


Figure 7: The effect of amount of activated charcoal on the percentage swelling of 2.0 g HCG-2 (at 5 g glycerol, 2 g maleic acid, 1 g NaOH)

of the H-bonds or may be ionization of the extra- functional groups which affects H-bonds within the network of HCG-2 gel lowering its swelling ability (Zheng *et al.*, 2012). The optimal mass ratio of 6:5 of AC to glycerol was found to have maximum absorption water capacity.

Effect of Amount of Maleic Acid (g) used on the swelling Capacity of Hydrogel (HCG-2)

Figure 8 shows the effect of varying dosages of maleic acid on the swelling capacity of superabsorbent hydrogel HCG-2.

As the cross linker dosage increases from 1.25 to 2.5 mL, a rapid increase in the percentage swelling capacity of HCG-2 hydrogel was noted from 430.67±6.03 to 1120.33±5.51%. This may be attributed to the increased mechanical strength of the gel, and increased cross linker point which leads to more pores per unit area in the 3D hydrophilic structure of the hydrogel network leading to a high rate of water absorption ability (Katime &

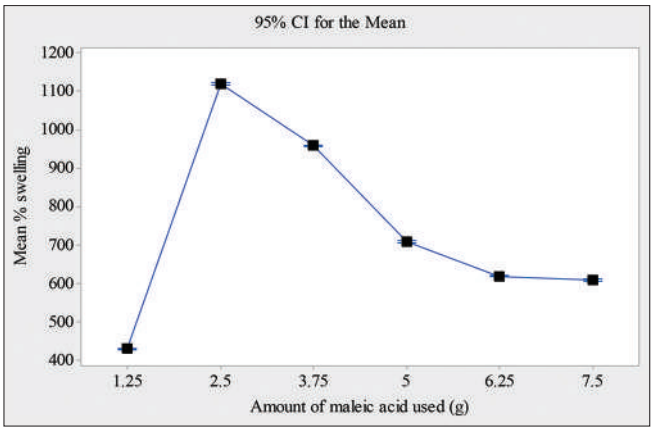


Figure 8: The effect of amount of maleic acid on the percentage swelling of 2.0 g HCG-2 hydrogel immersed in 500 mL of distilled water and swelling period of 24 hours (at 6 g AC, 5 g EA, 1 g NaOH)

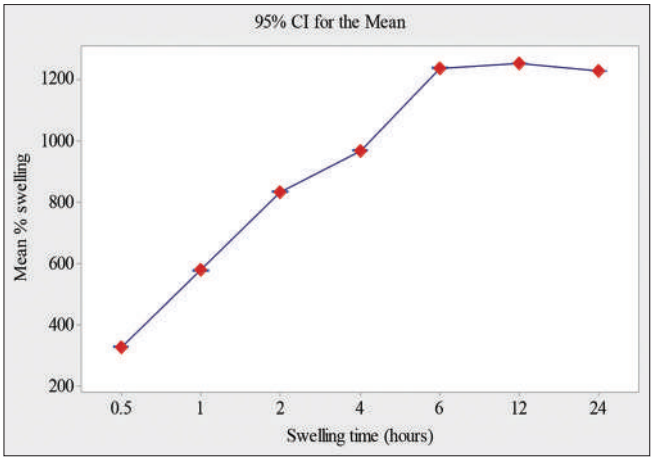


Figure 9: The effect of contact time on the percentage swelling of 2.0 g HCG-2 hydrogel prepared at optimum conditions of AC: G: MA of 8:5:1 immersed in 500 mL of distilled water

Mendizábal, 2010). A gradual swelling percentage capacity decreased from 1120.33±5.51 to 610.01±4.21% as crosslinker dosage increased from 2.5 to 7.5 mL. It is assumed that over crosslinking reinforces the hydrogel structure with amphiphilic groups resulting in increased pressure between smaller newly formed inter-phases. This leads to a reduced rate of hydrogel expansion rate as well as shielding water molecules from being absorbed in the crystalline structure of hydrogel decreasing its swelling ability (Katime & Mendizábal, 2010). A mass ratio of 8:5:1 of AC, glycerol and maleic acid was found suitable to synthesize HCG-2 hydrogel with optimum swelling capacity.

Effect of Time on the Swelling Capacity of the HCG-2 Superabsorbent Hydrogel

Figure 9 shows the effect of time on the swelling capacity of super absorbent hydrogel HCG-2 at different contact times.

The mean swelling percentage increased steadily from 330.43±1.08 to 1255.80±0.70% within the first 12 hours This indicates that as the time interval increases, the porous network of the polymer gets saturated with water hence no vacant spaces for the water molecules (Vimala *et al.*, 2009). Subsequently, the rate of swelling gradually slows to an equilibration between 12 and 24 hours. Again, during the swelling process, hydrogel expands but on saturation, ionization occurs releasing acidic molecules in water which decreases water absorption by the functional group (Ji *et al.*, 2018). The results show that HCG-2 superabsorbent hydrogel is more hydrophilic due to the increased number of hydroxyl functional groups facilitating high water absorption capacity. Jafari and Hamid (2005) reported a swelling percentage of 850% at pH 7.0 for the PAA-based complex, Kasimu *et al.* (2022) reported a swelling percentage of 1090, 925 and 910% for HCE-2, HLE-2 and HLG-2 respectively. The high swelling capacity of HCG-2 makes it a potential hydrogel for agricultural applications.

CONCLUSIONS

The results from this study showed that a mass ratio of 8:5:1 between AC with glycerol and maleic acid produced hydrogel with maximum water absorption capacity. The FT-IR spectra show sharp bands at 1591.34 cm^{-1} and 1400.28 cm^{-1} accredited to $-COO^-$ in symmetric stretching and asymmetric bending. This is a proof of ester polymerization between AC and glycerol monomers forming superabsorbent hydrogel. An increased number of -OH functional groups coupled with a sharp peak at 1639.48 cm^{-1} associated with $-COO^-$ bending in non-conjugated ester showed successful ester crosslinking in HCG-1 polymer units forming crosslinked HCG-2 hydrogel during the synthesis process. XRD showed HCG-2 polymer was crystalline compared to semi crystalline structure in HCG-1. SEM analysis showed an intact, rigid structure in HCG-1 with no pores on its surface compared to the well-developed fibrous with smooth irregular pores and lamina structure in HCG-2. When 2.0 g of HCG-2 were subjected to a contact time of 12 hours in 500 mL deionized water a maximum mean swelling capacity of 1255.80±0.70% was achieved. This showed that HCG-2 has the potential to boost agriculture mainly in arid and semi-arid regions by taming water retainability in soils during dry periods.

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