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Preparation and Characterization of Cellulose Based Superabsorbent Hydrogel from Rice Husk Cross-Linked With Ethane-1, 2-Diamine Using a Microwave

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Abstract

Superabsorbent hydrogels are being increasingly used as soil conditioners to enhance soil water retention, reduce the rate of irrigation and improve plant growth during drought. In the present work, superabsorbent hydrogel was prepared from cellulose based material with ethylenediamine as the crosslinking agent and evaluates it impact as water reservoir on maize growing in greenhouse. The cellulose isolated from rice husk, which has a basis to modify and obtain carboxymethylcellulose (CMC) using sodium hydroxide (NaOH) and monochloroacetic acid (MCA). The superabsorbent hydrogel was characterized by fourier transform infrared (FTIR) and x-ray diffraction (XRD). The percentage swelling attained by optimum conditions of time, power and amount of cross-linker required for the production of most desirable, stable and high water absorptivity were investigated, the optimum swelling capacity was found to be 1175%. The control pot (no superabsorbent hydrogels) revealed a significant difference in plant growth parameter and growth yield parameters compared to the pots treated with superabsorbent hydrogels. Increase in hydrogel dose significantly affects the growth and yield parameter of the maize. The optimum were recorded at 5grams for the superabsorbent hydrogels.

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1.0 Introduction

Superabsorbent hydrogels are 3-dimensional hydrophilic (water-loving) polymers, which have the ability of absorbing and retaining a high amount of water, and the releasing it under stress conditions. In dry state, its network is in form of coil, when exposed to water, they significantly expand to a larger size. The ability of the hydrogel to absorb water is as a result of the hydrophilic functional groups (OH, CONH, CONH2, COOH, and SO3H) attached to the polymer backbone while their resistance to dissolution arises from the cross-linker between networks (Raju *et al.*, 2003). The swelling abilities depend on the polymer and the degree of crosslinking which can either be covalent, ionic, hydrogen, van der Waal interaction existing between the polymeric networks. Superabsorbent hydrogels are widely used in various fields such as hygiene napkins, disposal diapers, soil for horticulture and agriculture, drug delivery system, water and food purification, (Wang *et al.*, 2008). Nowadays, they are widely used in tissue engineering (Khan *et al.*, 2009), sensor (Sorber *et al.*, 2008), drug delivery (Wu *et al.*, 2008), soft contact lense (Park, 1997), blood-contacting biomaterials (Michalek *et al.*, 2010). Most of the existing superabsorbents are made

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from petroleum based products such as acrylic acid, methacrylic acid and acrylamide, which are known to be non-renewable materials (Hacker and Mikos, 2011). Unites state environmental agency reported that, the petroleum based product used in making diapers are carcinogenic when exposed to human (U.S, EPA, 1994). There is need to develop superabsorbent hydrogels that are eco-friendly, especially from natural materials such as cellulose, starch and chitosan. Consequently, they have attracted much attention due to their cost-effective, degradability and availability (Mahmoudian and Ganji, 2017).

Cellulose is the most abundant renewable, biocompatible and degradable linear, long-chain, water-insoluble, natural polymer on earth. It is broadly circulated in the plant such as grass (Sutiya *et al.,* 2012), straw (Xu *et al.,* 2013), wood (Sunardi *et al.,* 2016) and cotton (Li *et al.,* 2014). Cellulose has been isolated from several agricultural wastes such as, orange peels (Arslan, 2007) palms oil (Palamae *et al.,* 2017), banana (Adinugraha *et al.,* 2005), cotton waste (Haleem *et al.,* 2014), durian (Rachtanapun *et al.,* 2012), and sago waste (Pushpamalar *et al.,* 2006), rice husk (Abdulhameed *et al.,* 2019).

Carboxymethylcellulose can be obtained when cellulose is reacted with monochloroacetic acid (MCA) or its sodium salt under alkaline condition in the presence of an organic solvent (Putri and Kurniyata, 2016). Carboxymethylcellulose has several hydroxyl (OH) and carboxyl (COO-) groups, depending on the degree of substitution. Due to its low toxicity and immunogenicity, carboxymethylcellulose is widely used in pharmaceutics and drug delivery, cosmetics, toothpastes and food additives.

Several studies have been done on the synthesis of carboxymethylcellulose from cellulose obtained from agricultural waste materials such as sugarcane bagasse (Alizadeh *et al.,* 2017), palm carnel cake (Bono *et al.,* 2009) and cotton ginning (Haleem *et al.,* 2014), sago waste (Pushpamalar *et al.,* 2006), mulberry paper waste (Rachtanapun *et al.,* 2015), papaya peel (Rachtanapun *et al.,* 2010), sugar beet pulp (Togrul and Arslan, 2003), rice husk (Abdulhameed *et al.,* 2019).

Crosslinking is a broadly used method for the modification of polymer properties. This process involves the formation of three dimensional structures causing substantial changes in material properties, thereby avoiding dissolution in the polymer network. Ethylenediamine has been used by several researchers as crosslinkers. Vanderck *et al.,* 2010 used ethylenediamine to crosslinked polyiimide membrane in methanol environment, the crosslinker can either be crosslinked by chemical reaction or UV irradiation. Bifunctional amine such as ethylenediamine and para-xylylethylenediamine were also used as crosslinkers for a gas separation membrane to avoid plasticization (Okamoto *et al.,* 1999). Yang *et al.,* 2010 reported that the aqueous solution of ethylenediamine was primarily used to crosslinked the pore walls and active surface of polymer membrane. Li *et al.,* (2014) reported that using ethylenediamine as crosslinker during the synthesis of few-layer reduced grapheme oxide, is cost effective and was tested as electrolyte for a Li+ ion battery which showed advantages with 346m Ah g-1 capacity at a charge discharge current density. Luan *et al.,* (2013) also used ethylenediamine as crosslinker during the synthesis of robust highly conductive 3D GO hydrogel, it showed high electrical conductivity of 1351 S m-1 and specific surface area of 745 m2 g-1 with 10.3 MPa break strength. Therefore ethylenediamine has been proved to be an effective crosslinker. So far, there is limited information on crosslinking of carboxymethylcellulose with ethylenediamine, we therefore hypothesized that ethylenediamine can be used to crosslinked carboxymethylcellulose using a microwave, thereby forming an amide linkage. Electrostatic repulsion between COO- in carboxymethylcellulose ions create more spaces with the hydrogel matrix, thereby absorbing high amount of water. In our previous studies, we used 1, 2-ethanediol as crosslinker in the synthesis superabsorbent hydrogel from carboxymethylcellulose without dispersing in any solvent.

Microwave heating has been used by many researchers for various applications (Ekezie *et al.,* 2017). It provides volumetric heating process and improves heating efficiency as compare to other conventional heating techniques. Microwave heating is simple to use, reduce the required process energy, enhances production yield and reduces the generation of bye product and is environmentally friendly. Therefore, microwave heating has become an attractive heating technique.

Therefore, this work aimed at synthesizing and characterizing a superabsorbent hydrogel from rice husk as cellulose derivatives, cross-linked with ethylenediamine, optimized condition for synthesis and evaluate its impact on growing of maize in greenhouse.

2.0 Materials and Methods

2.1 Materials

The rice husk was obtained from Euro rice mills in Mwea, Kirinyaga County – Kenya. Sodium hydroxide, ethylenediamine and glacial acetic acid were prepared from Merck Chemical Co. (Darmstadt, Germany). Nitric acid, Ethanol and methanol were provided from the local market. Monochloroacetic acid (MCA) was purchased from Daejung Co. (South Korea) and an

RM 240 microwave with a maximum power output of 700 watts was used in the synthesis process.

2.2. Methods

2.2.1. Extraction of Cellulose

About 5.00g of rice husk was weight in 250mL Erlenmeyer flask and 100mL of 80% glacial acetic acid, 10mL of 70% nitric acid was added. The flask was covered using aluminium foil and heated in an oven at 120°C for 20 minutes. The sample mixture was allowed to cool and 60mL of distilled water was added, the mixture was filtered and washed with distilled water and 95% ethanol. The residue was dried in an oven at 60°C for 19hrs. (Brendel *et al.*, 2010)

The extract yield of cellulose was calculated using the formula below

$$\text{The yield of cellulose } (\%) = \frac{\text{Dry weight extract of RH}}{\text{Weight of RH}} \times 100\%$$

2.2.2. Synthesis of carboxymethylcellulose (CMC)

The synthesis of carboxymethylcellulose (CMC) involves two stages: alkalization and carboxymethylation (etherification). About 5.00g of the extracted cellulose was added to 100mL of distilled water in a 250mL Erlenmeyer flask. Then 10mL of 30% of sodium hydroxide solution was added dropwise. The alkalization process was carried out for 1hr at 25°C on a fitted shaker. Then 5.00g of monochloroacetic acid were added to the mixture and heated in a microwave at the power of 6 for 2 minutes, the mixture was filtered. Neutralization obtained residue by soaking with 100mL of methanol for 24hrs, then the mixture was neutralized using glacial acetic acid. The mixture was filtered again and the residue was dried in an oven at 60°C.

The yield of carboxymethylcellulose was calculated using the formula,

$$\text{The yield of CMC } (\%) = \frac{\text{Weight of CMC } (g)}{\text{Weight of Cellulose } (g)} \times 100$$

2.2.3. Synthesis of Superabsorbent Hydrogel

About 2.00g of carboxymethylcellulose (DS-0.79) was dissolved in distilled water by stirring at room temperature using a magnetic stirrer for 24 hrs, after obtaining a solution, a variable amount of ethylenediamine and water with ratio of 1:5 was added as the cross-linker. The solution was heated in a microwave at various power outputs and time. To remove the unreacted chemicals, the contents were washed several times with distilled water until the hydrogel was perfectly transparent. It was then dried in an oven at 45°C until it maintains a constant weight.

2.2.4. Swelling Ratio

The equilibrium swelling capacity was measured by weighing the sample before and after immersion in distilled water for 24hrs followed by removal of the excess water on the surface with a syringe and filter paper. The swelling ratio is obtained using the following equation:

$$\%Swelling\ (\%S) = 100\left(\frac{m_t - m_o}{m_o}\right) \quad (3)$$

Where m_t is the initial weight and m_o is the final mass of the hydrogel.

2.3. Characterization

2.3.1. Fourier Transform Infrared (FTIR)

The Functional groups of the cellulose, carboxymethylcellulose and superabsorbent hydrogel were investigated using infrared spectroscopy spectrum (Shimadzu IR Tracer -100). Pellets were made from cellulose, carboxymethylcellulose and superabsorbent hydrogel using KBr and measured for wavenumbers of 3800-400 cm^{-1} respectively.

2.3.2. X-ray Diffraction (XRD)

The X-ray diffraction patterns of the carboxymethylcellulose and superabsorbent hydrogel were recorded on a D2 PHASER Bruker AXS X-ray diffractometer. The powder samples were dried in a hot air oven at 105°C for 3 h before testing. The scattering angle (2θ) ranged from 10 to 60° at a scan rate of 5°/min.

2.4 Application of superabsorbent hydrogel

This was carried out to determine the possibilities of applying superabsorbent hydrogel to reserve water in agriculture, and its effect on corn grown in Darazo, Bauchi state, Nigeria using a greenhouse mechanism. The experiment was completely randomized design with 3 replications. The sandy soil was obtained from farmers of Darazo town. In this study, 18 pots were used and each pot contained 12kg of the soil. The dried powder of superabsorbent were mixed with the soil (sandy) closer to the plant roots. A variable amount of the superabsorbent hydrogel (1, 2, 3, 4 and 5g) was mixed closer to the plant. The doses were set in triplicate, soil without amendment of the superabsorbent hydrogel was considered as a control.

2.4.1 Planting condition

In order to improve the soil fertility, N. P. K fertilizer with (20. 10. 10) formulations (Urea, diammonium phosphate, muriate of potash) was mixed with the soil. The pots were saturated with borehole water before planting and excess water was drained from the bottom. About two seeds of DMRESR-Y variety were planted in each of the 18 pots for plant growth. The pots were watered immediately after planting and subsequently. Observations were recorded on plant height, the number of leaves (fresh), area of leaves, the grain and stover/stone yield and the yield attributes of the crop. The grain yield maize of each treatment was weigh using weighing balance (Analytical grade).

2.4.2 Pre-harvest studies

2.4.2.1 Plants height

The plant height of each pot was measured using a meter scale from ground level to the growing tip of the plant at 3, 6, 9 and 12 weeks and expressed in centimeter, and the mean values were worked out.

2.4.2.2 Number of leaves per plant

The numbers of leaves of each pot/plant were counted at weeks 3, 6, 9 and 12 after sorrowing and harvest, the mean values were observed and recorded as number of leaves.s

2.4.2.3 Leaves length and width

The leaves length and width were also measured using a metre rule, they can help determining the leaf area, as shown in subsection below

2.4.2.4 Leaf area

The leaf area were calculated using the following equation,

$$Leaf\ area = leaf\ length \times leaf\ width \times k \qquad \ldots\ldots 3.4$$

Several studies revealed that k = 0.75 referred to as the coefficient for determination of leaf area (Yao *et al.*, 2010; Musa and Usman, 2016; Liliane *et al.*, 2019).

2.4.3 Post-harvest studies

2.4.3.1 Harvesting

The maize was harvested after it reaches maturity, when the leaves and husk turns to yellow and dry, with the help of sickle and digger. The maize was dried under the sun, then threshed, shelled and winnowed. They were packed and labeled properly and transported to the laboratory for further studies.

2.4.3.1.1 Yield attributes of maize

2.4.3.1.1 Number of cobs per plants

The number of cobs for each plant was counted at the harvesting stage, the mean value was calculated and recorded.

2.4.3.2.1.2 Length and width of cob (cm)

The biggest cob of the maize was taken out, the length and width were measured from the base to the tip of the cob using a metre scale, and the average mean was calculated and recorded in centimeter.

2.4.3.2.1.3 Cob weight (g)

The cob weight of the biggest maize was weight in grams using an electronic weighing balanced.

2.4.3.2.1.4 Number of grain per cob

The average number of grain per cob was counted and recorded using electronic weighing balance.

2.4.3.2.1.5 Grain weight (g)

The grain of biggest cobs obtained were also weighed in grams using an electronic weighing balanced

2.5 Statistical analysis

The result obtained from the studies were subjected to analysis of variance (ANOVA) using minitab software version 17, fisher least significance difference (LSD) test to generate mean at 5% confidence level of significance.

3.0 Results and Discussion

3.1. Cellulose FTIR

The fourier transform infrared (FTIR) spectroscopic investigations gave different absorption bands which are characteristics for cellulose (Figure 2). The broad band at 3432.39 cm^{-1} is characteristic of the OH-stretching vibration it gives good information about the hydrogen bonds formation, intramolecular and intermolecular hydrogen bond (Wingerson and Richard, 2002). The band at 2924.13 cm^{-1} represents the C–H stretching vibration. The absorption band at 1631.81 cm^{-1} is assigned to absorbed water. In addition, the absorption band at 1383 cm^{-1}, represents the symmetric CH_2 bending vibration. The hemicellulose and lignin peak at 1508 cm^{-1} and 1459 cm^{-1} (which were observed in figure 1) fully disappeared after extraction (figure 1) implying that cellulose has been isolated. The absorption band at 1104.55 cm^{-1}, represents the C–O–C stretching at β-(1-4)-glycosidic linkages (Kondo, 1997).

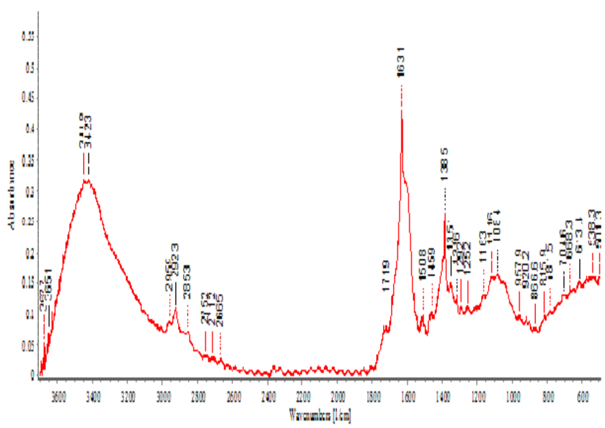

Figure 1: FTIR of rice husk before extraction

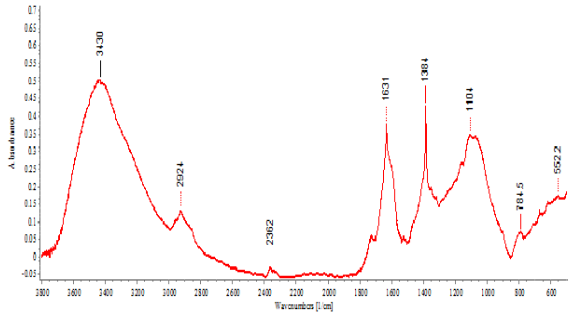

Figure 2: FTIR of the extracted cellulose from rice husk

3.2. FTIR of carboxymethylcellulose (CMC)

The FTIR spectra of the carboxymethyllcellulose sample prepared to show the typical absorptions of the cellulose backbone as well as the presence of the carboxymethyl group (figure 3). The broad absorption band at 3337 cm^{-1} is due to the stretching frequency of the –OH group as well as intramolecular and intermolecular hydrogen bonds. The band at 2948 cm^{-1} is assigned to CH_2 stretching vibration (Viera *et al.*, 2007). The new and strong absorption band at 1593 cm^{-1} confirms the presence of COO^- group. The bands around 1459 cm^{-1} are assigned to $–CH_2$ scissoring and –OH bending vibration (Yeasmin and Mondal, 2015). The band at 1086 cm^{-1} is due to $CH–O–CH_2$ stretching vibration. 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.

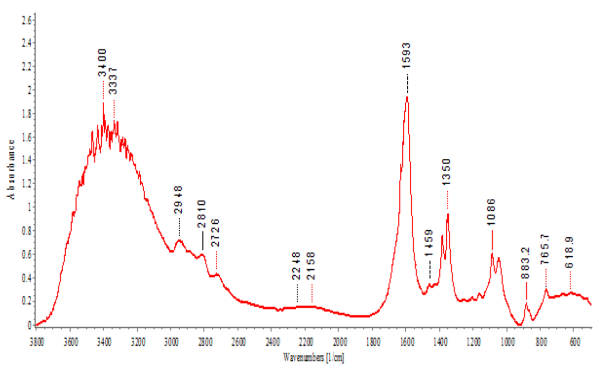

Figure 3: FTIR of Carboxymethylcellulose

3.3. Cellulose-Based Hydrogel

The spectrum of the hydrogel indicated peak at 3379 cm^{-1} which is due to stretching of hydroxyl (OH) groups. The peak at 2928 cm^{-1} indicated C-H stretching vibrations (Abdulhameed *et al.*, 2019). The new sharp peak observed at 1663cm^{-1} indicated the formation of amide linkage (Mishra *et al.*, 2008; Bakravi *et al.*, 2017). The peak at 1437 cm^{-1} and 1386 cm^{-1} could be as a result of CH_2 and OH bending vibration respectively. The peak at 1061 cm^{-1} is assigned to C-O-C stretching. Therefore, the FTIR Spectrum shown in figure 4, clearly indicates that crosslinking has taken place between carboxymethylcellulose and ethylenediamine, due to the formation amide linkage.

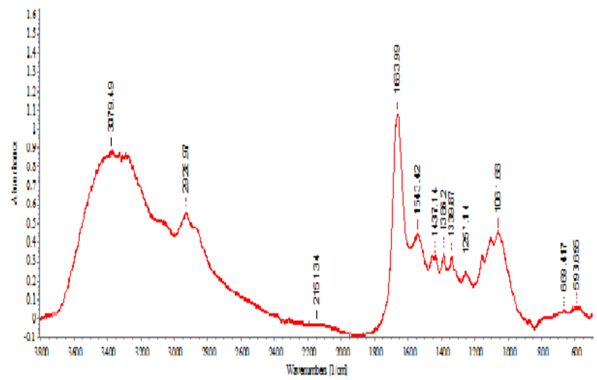

Figure 4: FTIR of cellulose based hydrogel crosslinked with ethylenediamine.

3.4. X-ray Diffraction

The superabsorbent hydrogel and carboxymethylcellulose were characterized by the x-ray diffraction (XRD) pattern. The x-ray diffraction pattern indicates that the superabsorbent hydrogel shows broad peak implying amorphous morphology, it simply attributes to the network of the polymer (Varapsad *et al.*, 2009). They have a main characteristic peak at 2θ=22 and 22 respectively, it is observed that the peak intensity of the superabsorbent hydrogel is higher than the peak of carboxymethylcellulose at 22°. It is clear that carboxymethylcellulose is not completely amorphous, but have a relatively higher degree of crystallinity. The decrease in crystallinity plays a vital role in hydrogel degradability, water uptake and swelling ratio (Costa-Junior *et al.*, 2009).

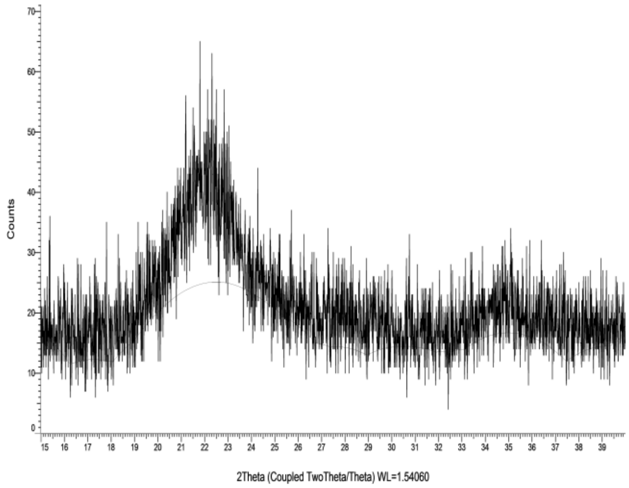

Figure 5: X-ray diffraction of carboxymethylcellulose

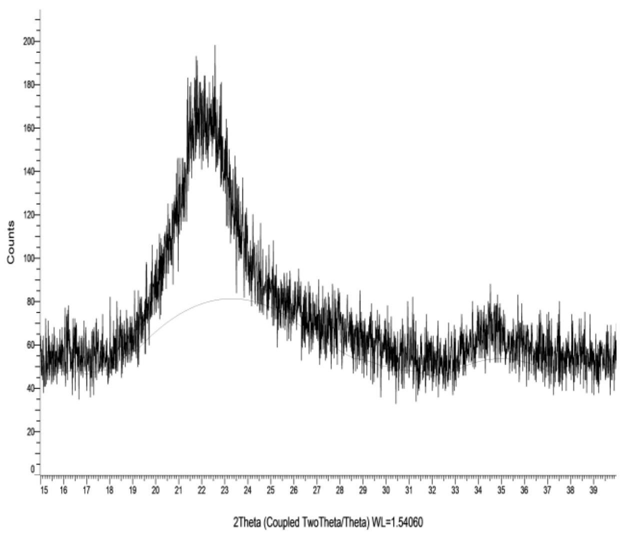

Figure 6: X-ray diffraction of superabsorbent hydrogel crosslinked with ethylenediamine

3.5. Optimizing synthesis conditions for super-absorbent hydrogels

The percentage swelling of the superabsorbent hydrogels depends on the nature of the polymer network, the reaction condition and concentration of the crosslinking agents. It is also a prominent factor that determines the properties and application of the hydrogel. The Optimum conditions of power output, time, CMC: linker mass ratio and solvent required for the production of most desirable, stable and high water absorptivity were investigated.

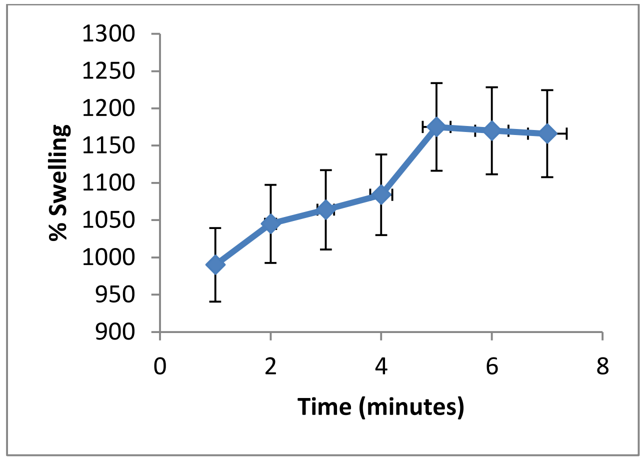

Figure 7: Variation of percentage swelling with reaction time (at Power output 4 equivalent to 280 watts, 10 mL of ethane-1,2-diamine, 5 minutes, 2 g of CMC)

The effect of time on percentage swelling was studied from 1 – 5 minutes. Figure 7 represents the result. The percentage swelling was about 990% at 1 minute, it further increase to 1045, 1064, 1084 and 1175% at 2, 3, 4 and 5 minutes respectively. The increase in percentage swelling increases with longer time, this can be as a result of increases in H^+ and OH^- ions which binds to the hydrophilic groups in the superabsorbent hydrogel. ($CONH_2$ being hydrophilic can easily absorbed high amount of water). Due to the presence of $CONH_2$ in the polymer network, it creates a distance between the matrix, which allow the hydrogel to absorb a lots of water, and there are free available sites where water molecules can be attached to the superabsorbent hydrogel.

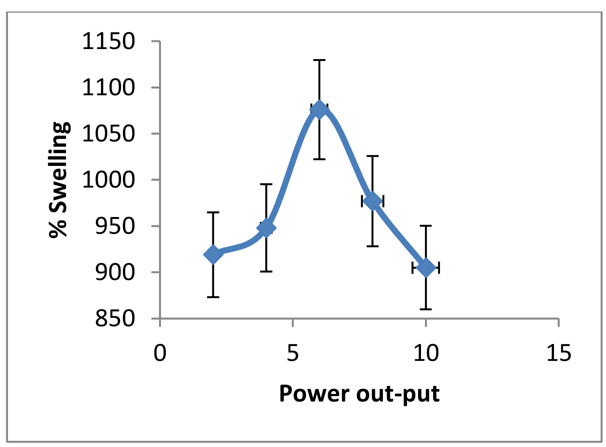

Figure 8: Variation of percentage swelling with power out-put (at 5 minutes, 10mL of ethane-1,2 diamine, 2 g of CMC

The percentage swelling was carried out by varying the power out-put of microwave (from 2 - 10) as illustrated in the figure 8. The percentage swelling increases from 919 – 1076%, when the power out-put was increase from 2 - 6, this is equivalent to 140 - 420 watt respectively. An increase in the power output from 6 – 10, which corresponds to 420 – 700 watts, was accompanied by a decrease in percentage swelling from 1076 - 905. The highest percentage swelling of 1076 occurred at power output of 6 (420 watt) and recorded as optimum. The increase is due to elasticity nature of the matrix and interaction of the polymer network. Gupta and shivakumar 2012 reported that there is increase in mobility chain when temperature increases, it therefore lead to the network expansion and percentage swelling. The decrease in the percentage swelling might be as a result of inadequate entropy and internal energy, leading to low rate of diffusion of water molecules into the hydrogel.

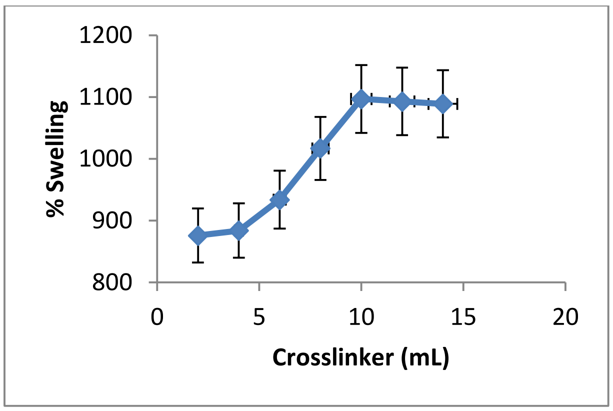

Figure 9: Variation of percentage swelling with amount of cross-linker (at 5 minutes, power output of 4 equivalent to 280 watts, 2 g of CMC

The impact of the amount of crosslinker (ethylenediamine) on percentage swelling was investigated by varying the amount from 2 – 14 mL. The percentage swelling was about 876% at 2 mL, there was an increase gradually to 884, 934, 1017 and 1097% at 4, 6, 8 and 10 mL respectively. Increase in crosslinking agent leads to increase in density of crosslinking and proper interaction of functional groups in the polymer. By using 2 mL, it can be characterized by a low amount of crosslinking agent, therefore it is inadequate to obtained a stable hydrogel, capable of absorbing and retaining high amount of water molecules in the polymer network. As the amount of crosslinker increases the percentage swelling also increases. The amount of water absorbed solemnly depends on the amount of crosslinking agent used in the polymer network, at equilibrium it decreases. However, hydrogel synthesizes in presence of high amount of crosslinker are expected to have low water absorption. (Bennour and Louzri, 2014).

3.6 Effect of superabsorbent hydrogels on growth parameter of maize

Superabsorbent hydrogels improves crop growth by enhancing the water retaining ability in the soil. When the superabsorbent hydrogels are mixed with soil, the retained high amount of water and nutrients are released as required by the crop, hence sustains crop growth.

3.6.1 Plant height (cm)

The effect superabsorbent hydrogels on plant height was investigated. The SAH obtained using crosslinker (ethane-1,2-diamine) was varied from control, 1, 2, 3, 4 and 5 grams, at week 3, 6, 9 and 12 weeks after planting. The results are displayed in figure 10 below.

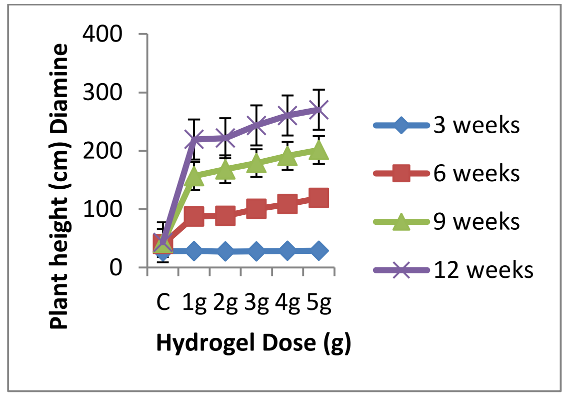

Figure 10: Effect of superabsorbent hydrogel (crosslinked with ethane-1,2-diamine) on plant height.

The effect of superabsorbent hydrogel (crosslinked with ethane-1,2-diamine) on plant height was studied in triplicate by varying the SAH dose from control to 5 grams. The plant height at week 3 ranged from 27.69 to 28.97cm for SAH crosslinked ethane-1,2-diamine while that of week 6 ranged from 40.06 to 119.00cm for SAH crosslinked with ethane-1,2-diamine, between control to 5 grams. Similarly, the plant height at week 9 ranged from 42.21 to 201.44cm for SAH crosslinked with ethane-1,2-diamine, while that of week 12 varied from 43.24 to 270.23cm for SAH crosslinked with ethane-1,2-diamine, between control to 5 grams.

The superabsorbent hydrogel at different doses significantly affect the plant height of maize grown in green house. There is increase in the height of plant from all levels when SAH dose was varied from control to 5 grams at 6, 9 and 12 weeks. As hydrogel dose increased in week 6, 9 and 12, the plant height also increased which is statistically significant. This shows that the superabsorbent hydrogels have positive effect on plant height. The increase in the amount of superabsorbent hydrogels led to the increased in plant height and spread. The increase of plant height could be due to water and nutrients provided by the superabsorbent hydrogels, which have been expected to enhanced the activities of division, expansion and elongation of plant cell. Similar results were observed by Sendur *et al.*, 2001 in tomatoes and Al-Harbi *et al.*, 1999 in cucumber.

At 3 weeks, there is no significant different in the plant height at different hydrogel dose for all the superabsorbent hydrogels, this is because the hydrogel have not being applied to the plants surrounding, which led to draught stress, and significantly affect the plant height. Inadequate amount of water caused decreased in elongation of plant cell. The control pot (no hydrogel) revealed a significant difference in height of plant as compared to the pots with superabsorbent hydrogels at all weeks (3, 6, 9 and 12 weeks). The highest values were recorded at 5 grams for all the superabsorbent hydrogels. A similar result was observed by Yang *et al.,* 2006 that draught stress significantly affect plant height. A similar plant height was observed by Hossain, 2009; Niazuddin *et al.*, 2002. There was control of weed, in other to avoid plant height reduction as observed by Begner *et al.*, 2001.

3.6.2 Number of leaves

The impact superabsorbent hydrogels on number of leaves was also investigated. The SAH obtained using ethane-1,2-diamine crosslinker was varied from control, 1, 2, 3, 4 and 5 grams, at week 3, 6, 9 and 12 weeks after planting. The results are displayed in figure 3 and 4 respectively.

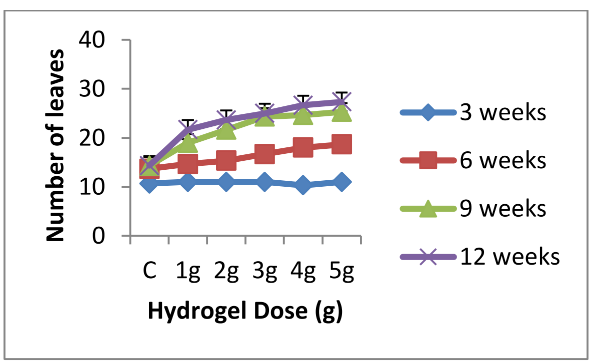

Figure 11: Effect of superabsorbent hydrogel (crosslinked with ethane-1,2-diamine) on number of leaves per pot.

The mean number of leaves at week 3 ranged from 8.00 to 11.00 for the superabsorbent hydrogels, at this stage, the superabsorbent hydrogels have not been applied to the plants. The mean number of leaves increased from 13.66 to 18.66 for SAH crosslinked with ethane-1, 2-diamine. At week 9, the mean number of leaves increased from 14.33 to 25.33 for SAH crosslinked with ethane-1,2-diamine while at week 12, the mean number of leaves increased from 14.33 to 27.33 for SAH crosslinked with ethane-1,2-diamine respectively.

At week 6, there is quite significant difference between the number of leaves in the pots, despite the variability in the amount of crosslinkers used. However, at week 9 and 12, the number of leaves for all the superabsorbent hydrogels varied significantly, which can be due to the dosage of the superabsorbent hydrogel. Higher number of leaves was observed when 3 to 5 grams of superabsorbent hydrogels are used which was statistically significant for all the superabsorbent hydrogels, irrespective of the crosslinker.

Superabsorbent hydrogels have significant effect of number of leaves. Increase in water deficit and low amount of superabsorbent hydrogels diminished the number of leaves which can led to dryness and leaf death of plants. Incorporation of superabsorbent hydrogels to soil around the root, revealed that water stress significantly lowered the number of leaves. The increase in amount of superabsorbent hydrogels (hydrophillic), significantly increased the number of leaves, this implied that application of superabsorbent hydrogel compensate the negative impact drought at high amount. Similar results were observed by Sendur *et al.*, 2001 and Al-harbi *et al.*, 1999.

3.6.3 Leaf area

The effect superabsorbent hydrogels on leaf area was also investigated. The SAH obtained using crosslinker ethane-1,2-diamine was varied from control, 1, 2, 3, 4 and 5 grams, at week 3, 6, 9 and 12 weeks after planting. The results are displayed in figure 12 below.

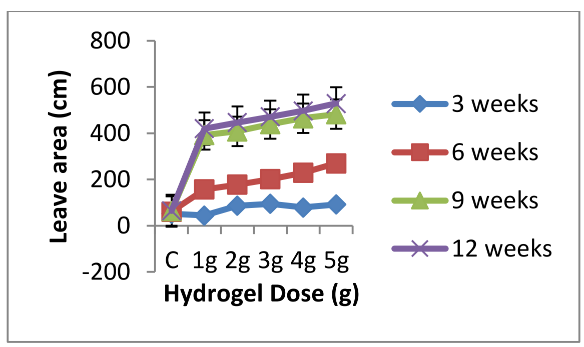

Figure 12: Effect of superabsorbent hydrogel (crosslinked with ethane-1, 2-diamine) on leaf area per pot.

The mean leaf area at week 3 ranged from 44.33 to 92.33 for SAH crosslinked ethane-1,2-diamine, while at week 6, there is increase in mean of leaf area from 60.93 to 296.75 for SAH crosslinked with ethane-1,2-diamine, respectively.

At week 9, the mean of leaf area also increase from 63.76 to 482.57 for SAH crosslinked with ethane-1,2-diamine, while at week 12, the average mean of leaf area increased from 64.59 to 528.78 for SAH crosslinked with ethane-1,2-diamine, respectively.
The result obtained shows the effects of superabsorbent hydrogels leaf area of maize at different doses. At week 3, the leaf area of maize was not static, because the superabsorbent hydrogels have not been applied. At week 6, 9 and 12, there was increase in leaf area as the dosage increased which was statistically significant, for the superabsorbent hydrogels. The mean values of the control pot, show a significant variation in leaf area as compared to rest of the hydrogel doses, therefore the highest leaf area was recorded at 5 grams dose for superabsorbent hydrogel. The growth of crop, productivity and healthy are related to leaf area, being an aerial part of the plant playing vital role in photosynthetic capacity through light absorbing pigments. Due to increase in the amount of superabsorbent hydrogel, a significant increase in leaf area was observed. The superabsorbent hydrogel increase the turgor pressure inside the plant, by providing adequate amount of water and nutrients as required by the plant, this led to increase in leaf area and other growth parameters (Yazdani *et al.,* 2007; Al-Harbi *et al.,* 1999)
Aflakpui *et al.,* reported a decreased in leaf area of maize caused by weeds, the superabasorbent hydrogel crosslinked with ethane-1,2-diamine revealed a significant higher leaf area at all stages. Therefore the water holding capacity of superabsorbent hydrogel can influence the leaf area of maize.

3.6.4 Leave length (cm)

The effect of superabsorbent hydrogel crosslinked with ethane-1,2-diamine on leaf length was studied in triplicate by varying the SAH dose from control to 5 grams, at week 3, 6, 9 and 12 weeks after planting. The results on leaf length of maize as influenced by the application of different levels of superabsorbent hydrogels are presented in figure 13 below.
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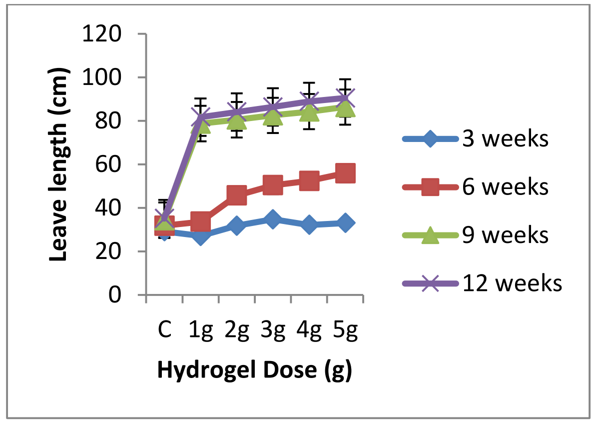

Figure 13: Effect of superabsorbent hydrogel (crosslinked with ethane-1,2-diamine) on leave length

The leaf length ranged from 27.21 to 34.71 cm for SAH crosslinked with ethane-1,2-diamine while at week 6, it ranged from 31.82 to 55.88cm for SAH crosslinked with ethane-1,2-diamine. Similarly, at week 9, the leaf length increased from 34.32 to 86.36cm for SAH crosslinked with ethane-1,2-diamine, while that of week 12, ranged from 35.06 to 90.50cm for SAH crosslinked with ethane-1,2-diamine.
At week 3, there is no significant different in the leaf length of the maize, because the hydrogel have not been applied. The leaf length varied significantly as the dosage of the SAH increased. The control pot showed a significant difference with other hydrogel dose, and 5grams was recorded as highest values. The value of the leaf length was not altered by weed. The application of superabsorbent hydrogels at all doses into the soil, significantly increased the leaves length. The increased could be due to water and nutrients provided by the superabsorbent hydrogel being hydrophilic in nature which increase cell elongation. The differences in doses were not quit significance between the superabsorbent hydrogels.

3.6.5 Leaf width (cm)

The impact of superabsorbent hydrogel crosslinked with ethane-1,2-diamine, on leaf width was studied in triplicate by varying the SAH dose from control to 5 grams, at week 3, 6, 9 and 12 weeks after planting. The results on leaf length of maize as influenced by the application of different levels of superabsorbent hydrogels are presented in figure 14 below.
At week 3, the leaf width ranged from 2.18 to 3.72 cm for SAH crosslinked with ethane-1,2-diamine, while at week 6, it ranged from 2.63 to 6.43cm for SAH crosslinked with ethane-1,2-diamine. Similarly, at week 9, the leaf length increased from 2.70 to 7.44cm for SAH crosslinked with ethane-1,2-diamine, while

that of week 12, ranged from 2.72 to 7.78cm for SAH crosslinked with ethane-1,2-diamine, respectively.
At week 3, the variation in leaf width per pot did not varied significantly, after the application of superabsorbent hydrogel at week 6, 9 and 12 the leaf width of pot which contained SAH crosslinked with ethane-1,2-diamine varied significantly due to increase in dose of hydrogels and the amount of water absorbed. Incorporation of superabsorbent hydrogels to soil significantly increased the leave width as compared to control. The increased can be due to water released by the superabsorbent hydrogels to plants. Anupama *et al.*, 2007 observed the performance of hydrogel, the most prominent growth with leave diameter, plant height, number of leaves as compared to control.

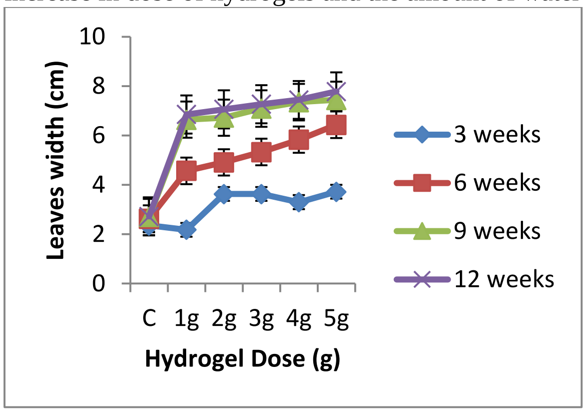

Figure 14: Effect of superabsorbent hydrogel (crosslinked with ethane-1,2-diamine) on leave width.

Table 1: Effect of SAHs crosslinked with ethane-1,2-diamine on pre-harvest studies

Weeks	SAH Dose	(Plant Height) Mean±SD	(Number of leaf) Mean±SD	(Leaf Length) Mean±SD	(Leaf width) Mean±SD	(Leaf area) Mean±SD
3	Control	27.69±1.60^{a}	5.66±1.52^{a}	29.21±2.54^{bc}	2.36±0.15^{b}	52.10±7.57^{b}
	1g	28.21±2.22^{a}	5.00±1.00^{a}	27.09±0.73^{c}	2.18±0.13^{b}	44.33±1.60^{b}
	2g	27.52±1.83^{a}	5.00±1.00^{a}	31.75±1.27^{ab}	3.63±0.15^{a}	86.77±6.78^{a}
	3g	27.88±1.80^{a}	5.00±1.00^{a}	34.71±0.73^{a}	3.63±0.15^{a}	94.75±3.27^{a}
	4g	28.48±2.10^{a}	5.33±0.57^{a}	32.17±1.19^{ab}	3.30±0.67^{a}	79.09±2.83^{a}
	5g	28.97±2.49^{a}	6.00±1.00^{a}	33.02±1.27^{b}	3.72±0.15^{a}	92.33±6.88^{a}
6	Control	40.06±0.97^{e}	5.66±0.57^{d}	31.82±1.42^{e}	2.63±0.05^{e}	60.93±1.2^{e}
	1g	87.46±0.69^{d}	6.66±0.57^{d}	33.60±2.54^{d}	4.57±0.00^{d}	156.80±0.06^{c}
	2g	88.32±1.24^{d}	7.33±0.57^{cd}	45.72±0.00^{c}	4.91±0.29^{cd}	177.71±10.59^{cd}
	3g	100.57±0.71^{c}	8.66±0.57^{bc}	50.37±0.73^{b}	5.33±0.25^{bc}	201.61±12.81^{c}
	4g	108.58±1.07^{b}	9.00±1.00^{ab}	52.91±0.73^{b}	5.83±0.25^{b}	228.93±9.29^{b}
	5g	119.00±3.11^{a}	10.66±0.57^{a}	55.88±1.27^{a}	6.43±0.14^{a}	269.75±11.91^{a}
9	Control	42.21±0.41^{f}	8.33±0.57^{d}	34.32±0.65^{e}	2.70±0.01^{d}	63.76±1.00^{d}
	1g	156.71±1.23^{e}	10.00±1.00^{c}	78.74±2.20^{d}	6.64±0.07^{b}	392.53±13.64^{c}
	2g	168.25±1.15^{d}	12.66±0.57^{b}	80.52±0.66^{cd}	6.72±0.12^{b}	408.22±11.04^{c}
	3g	179.05±1.10^{c}	14.33±0.57^{a}	82.55±1.27^{bc}	7.09±0.02^{b}	439.29±8.37^{b}
	4g	191.40±1.38^{b}	15.66±0.57^{a}	84.24±0.73^{ab}	7.35±0.01^{a}	464.76±2.94^{a}
	5g	201.44±1.05^{a}	27.33±0.57^{a}	86.36±0.00^{a}	7.44±0.15^{a}	482.57±9.50^{a}
12	Control	43.24±0.56^{e}	8.33±0.57^{d}	35.06±0.82^{e}	2.72±0.01^{e}	64.59±0.64^{f}
	1g	219.50±1.32^{d}	12.66±1.15^{c}	81.70±0.73^{d}	6.85±0.00^{d}	420.24±3.77^{e}
	2g	221.74±1.99^{d}	13.66±1.52^{bc}	84.06±0.92^{c}	7.06±0.07^{cd}	446.69±7.39^{d}
	3g	243.39±2.62^{c}	15.00±1.00^{ab}	86.36±0.00^{b}	7.27±0.14^{bc}	471.61±9.50^{c}
	4g	260.41±1.02^{b}	17.66±1.15^{a}	88.90±0.00^{a}	7.44±0.15^{b}	496.77±9.78^{b}
	5g	270.23±2.28^{a}	18.33±0.57^{a}	90.50±0.81^{a}	7.78±0.14^{a}	528.78±13.45^{a}

Means that do not share a letter are significantly different ($p < 0.05$)

3.7 Post-harvest

The results on yield parameter of maize as influenced by superabsorbent hydrogel crosslinked with ethane-1,2-diamine, at different dosage are presented sub-sections below. The attribute yield of number of cob per pot, cob length, cob width, cob weight, grain weight and number of grains are presented below.

3.7.1 Number of cob per pot

The effect of superabsorbent hydrogels crosslinked with ethane-1,2-diamine on number of cobs was investigated at varied dose ranging from control to 5 grams. The results are presented in table 2. The average mean of the number of cob increased from 0.33 to 3.33 for SAH crosslinked with ethane-1, 2-diamine. The average mean of the number was quite influenced by the superabsorbent hydrogels. The increase in hydrogel dose increased the number of cobs, but not quite significant. (Rohit, 2015) also observed similar results. Therefore, the number of cob was influenced the superabsorbent hydrogels.

The impact of superabsorbent hydrogels crosslinked with ethane-1,2-diamine on cob length was investigated at varied dose ranging from control to 5 grams. The results are displayed in table 2. The average mean of the cob length increases from 2.77 to 18.53cm for SAH crosslinked with ethane-1,2-diamine. The length of the cob was significantly affected by the application of superabsorbent hydrogel to the soil. Increase in dose of the superabsorbent led to increase in cob length. (Rohit, 2015) observed that application of 150% SAH to soil has a significant effect on cob length. The highest value of the cob length was observed with the superabsorbent hydrogel crosslinked with ethane-1,2-diamine. Similar cob length were also reported by Hossain, 2009; Niazuddin *et al.*, 2002.

The effect of superabsorbent hydrogels crosslinked with ethane-1,2-diamine on cob width was investigated at varied dose ranging from control to 5 grams. The average mean of the cob width increases from 0.66 to 3.80cm for SAH crosslinked with ethane-1,2-diamine. The results obtained shows that the cob width is influenced by superabsorbent hydrogels, the cob width increased with increased in hydrogel dose significantly. The highest value of the cob width was observed under superabsorbent hydrogel crosslinked with ethane-1,2-diamine at 5 grams.

The impact of superabsorbent hydrogels crosslinked with ethane-1,2-diamine on cob weight was investigated at varied dose ranging from control to 5 grams. The results are displayed in table 2.

The average mean of the cob width increases from 0.00 to 180.00g for SAH crosslinked with ethane-1,2-diamine. The data obtained on cob weight per plant was significantly influenced by superabsorbent hydrogel in maize. The increase in dose, significantly increase in cob weight. A similar result was observed by Sendure *et al.*, 2001, that increased in fruit weight per tomatoes is due to soil incorporation with superabsorbent hydrogels. The highest value of the cob weight was observed under superabsorbent hydrogel crosslinked with ethane-1,2-diamine at 5 grams.

The effect of superabsorbent hydrogels crosslinked with ethane-1,2-diamine on grain weight was investigated by varying the superabsorbent dose ranging from control to 5 grams. The average mean of the grain weight increases from 0.00 to 158.33g for SAH crosslinked with ethane-1, 2-diamine. The results obtained on grain weight per pot were increased significantly with increase in dose. Nazarli, 2010, reported that application of superabsorbent polymer enhance the 100seed weigh of sunflower compared to control. The value of grain weight of superabsorbent hydrogel crosslinked with ethane-1,2-dimaine was higher at 5 grams.

The impact of superabsorbent hydrogels crosslinked with ethane-1,2-diamine on grain weight was investigated by varying the superabsorbent dose ranging from control to 5 grams. The average mean of the grain weight increases from 0.00 to 418.67 for SAH crosslinked with ethane-1, 2-diamine. The results showed that the number of grain was significantly influenced by the superabsorbent hydrogels. The increase in dose significantly affects the number of grain obtained in the maize. The application of superabsorbent hydrogel improve crop yield. Khadem *et al.*, 2010 observed that incorporation of superabsorbent polymer to soil increased the number of grain by 16.2% as compared to control. The highest value of number of grain was observed under superabsorbent hydrogel crosslinked with ethane-1,2-diamine.

Table 2: Effect of SAHs crosslinked with ethane-1,2-diamine on post-harvest studies

SAH Dose	(Number of cobs) Mean±SD	(Cob length) Mean±SD	(Cob width) Mean±SD	(Cob weight) Mean±SD	(Number of grain per cob) Mean±SD	Grain weight Mean±SD
Control	0.33±0.57[d]	2.77±4.79[c]	0.66±1.55[c]	0.00±0.00[f]	0.00±0.00[f]	0.00±0.00[f]
1g	1.00±0.00[cd]	11.06±0.81[b]	2.42±0.58[b]	112.67±4.04[e]	242.67±2.52[e]	93.67±3.51[e]
2g	1.66±0.57[bcd]	13.27±0.83[ab]	2.78±0.15[b]	126.00±1.73[d]	294.67±3.79[d]	105.66±1.52[d]
3g	2.33±0.57[abc]	16.24±0.95[ab]	3.07±0.13[ab]	135.33±3.79[c]	324.33±3.21[c]	116.33±2.52[c]
4g	2.66±0.57[ab]	17.76±0.58[a]	3.20±0.20[ab]	154.67±4.04[b]	380.00±4.58[b]	133.33±1.52[b]
5g	3.33±0.57[a]	18.53±1.33[a]	3.80±0.11[a]	180.00±5.57[a]	418.67±8.08[a]	158.33±2.08[a]

Means that do not share a letter are significantly different ($p < 0.05$)

Table 3: Superabsorbent hydrogels and their pH

SAH	pH
SAH crosslinked with ethane-1,2-diamine	7.98

The result shows that the pH of all the superabsorbent hydrogel ranged between 6.0 – 8.0 and was considered in the application of the synthesized SAH in the green house. The percentage swelling of superabsorbent hydrogel depends on its ionic features (Pereira *et al.*, 2017). Therefore, the ionic feature becomes stable at neutral.

Conclusion

In this study, we have successfully obtained a superabsorbent hydrogel (from rice husk cellulose derivatives through carboxymethylcellulose) and ethylenedimaine as crosslinker. Fourier transform infrared and x-ray diffraction confirms the modification and synthesis. The percentage swelling depends on water diffusion through the hydrogel by absorbing water. The percentage swelling (at 5 minutes, power out-put 6, amount of crosslinker 10mL and carboxymethylcellulose dose 3g) of 1175% was recorded as optimum. As part of the recommendation, this SAH can be applied in arid areas for agricultural practices towards addressing drought which is in progress. The results on application of the superabsorbent hydrogels in a greenhouse showed it ability of water retaining capacity. Incorporation of superabsorbent hydrogel with the soil increases the water holding capacity of the soil, enhanced plant growth, crop yield and creating a conducive environment for root growth in well-drained soil. The plant growth parameters, plant height, number of leaves, leaf area, leave length, leaf width significantly increased with an increase in superabsorbent hydrogel dose. The increase in plant growth parameters can be attributed to water and nutrients released by the superabsorbent hydrogels as required. Being hydrophilic, they enhanced the activities of cell division, expansion and elongation of plant cell. The application of superabsorbent hydrogels significantly reduced irrigation frequency. The post-harvest parameters, number of cobs, cob length, cob width, cob weight, grain weight, number of grain are significantly influenced by superabsorbent hydrogels dose, this could be due to incorporation of superabsorbent hydrogels with soil, the retained water and nutrients are released as required by the maize. It reduces fertilizer lost and sustains crop growth. The highest yield was recorded at 5g of superabsorbent hydrogels. Sequel to results obtained from this study, we can therefore conclude that, superabsorbent hydrogels exhibit superior features for agriculture in arid and semi-arid zones. We recommend for investigating the efficacy of the SAHs obtained from this study, in an open field farm on pilot scale to determine possibility of up scaling and also the efficacy of SAHs on other crops and vegetables in an open field farm and green house.

Declarations

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Consent for publication

All authors have read and approved the final draft of the manuscript.

Availability of data and material

All data generated or analyzed during this study are included in this published article.

Competing interests

The authors declare that they have no competing interests.

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