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A Novel Method to Produce Cost Effective, Environment Friendly Superabsorbent from Water Hyacinth for Sustainability-Driven Medical Applications

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Abstract

Introduction: Superabsorbent materials (SAMs) are the most iconic class of soft materials, and have attracted in a plethora of applications. In recent years, SAMs have been applied extensively in in the field of medicine where super absorbent wound dressings have gained paramount importance in the wound care. The majority of conventional SAMs available in the market are produced based on synthetic materials which pose risks to the health and environment safety. Therefore, we herein devoted to develop a green strategy for the synthesis of an eco-friendly, super absorbent based on Eichhornia crassipes (Water Hyacinth) in a cost- effective manner. Eichhornia crassipes is considered the worst aquatic weed in the world as it has become a serious threat to the environment and biodiversity hence eco-friendly utilization of this hydrophyte is needed and important. Nevertheless, this hydrophyte has potential to harness for development of superabsorbent materials due to relatively high content of cellulose.

Method: *Eichhornia crassipes* was collected from the tank nearby Faculty of Applied Sciences, Wayamba University of Sri Lanka and petiole was treated with potassium hydroxide (KOH) followed by microwave irradiation in which reaction conditions were optimized to obtain maximum water absorption and swelling capacity. Prepared super absorbent was characterized using Fourier Transform Infrared Spectroscopy (FTIR), XRD and scanning electron microscopy (SEM) techniques. The efficacy of the superabsorbent on improved water retention was assessed using normal loam soil.

Result and Discussion: The superabsorbent showed a maximum swelling index of 1276 % at KOH concentration of 0.1 moles/l which is attributed to highly porous structure, presence of hydrophilic functional groups in cellulose and hemicellulose, increased number of surface hydrophilic functional groups during the KOH activation process and carboxy methyl cellulose created during microwave irradiation. Water absorption capacity of the super absorbent is greatly influenced by KOH concentration, reaction time, microwave power and exposure time. Water retention studies in soil showed that superabsorbent has capacity to retain water for 27 days with a slow rate of water evaporation whereas soil samples without superabsorbent showed a high rate of water evaporation retaining water only for 15 days. Findings of this study disclose an innovative method for development of an eco-friendly, super absorbent from *Eichhornia crassipes* in a cost-effective manner excluding toxic chemical reagents which can be used in agricultural and medical applications.

Keywords: Super-absordent; Water hyacinth; Biodegradable, Microwave; Chemical activation; Medical

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INTRODUCTION

Superabsorbent polymers (SAMs) are a class of hydrophilic materials and three-dimensional network systems which can absorb and retain large quantities of water and other aqueous solutions without dissolving [1, 2]. Excellent water retention and absorbency of superabsorbent polymers have been leveraged for product development in the medical sector, particularly in wound management. Efficient exudate absorption, and faster healing process are key attributes of medical super absorbent polymers that have driven their popularity. SAMs have already proven their use in a plethora of applications, namely agriculture, hygiene products, sealing materials, artificial snow, drilling fluid additive, fine coal dewatering, and wastewater treatment, medicine for drug-delivery systems, construction engineering, and communication cables.

In recent years, SAMs have been applied extensively in in the field of medicine namely medical test tablets to retain the examined liquids, exterior drug paste with high water content, bandages that can absorb blood and secretion from surgery or trauma, and artificial antibacterial skin which permit penetration of water and drug whilst retaining animalcule without permeation. In addition, SAMs have also exhibited potential applications as carriers in control and target drug-release and contact lenses. Using SAMs as drug carriers, the drug-release rate can be controlled through modulating the structure and swelling ratio of SAMs in various mediums. The soft contact lens fabricated using SAMs has better oxygen-permeating properties than the others, comfortable to the wearer, and extended shelf life.

Superabsorbent wound dressings have gained paramount importance in the wound care on the coattails of its extremely absorbent and unique core for exudate management. These dressings are deemed highly effective in soaking up wound exudate from venous ulcers, burns, surgical incisions, and chronic wounds. Chronic wound prevails as a chronic issue, despite billions of spending by health care systems in wound care. However, routine clinical practice and research in wound management have meant that advancements in science are imminent for better understanding and addressing chronic wounds. 2-acrylamido-propane sulphonic acid (AMPS) and Polyaspartic acid (PASP) - based superabsorbent polymers, which are sensitive to pH and glucose conditions, are being exploited for chronic wound dressing.

Several injuries to the skin caused by accidents, burns, trauma, chronic wounds and diseases can pose a major healthcare problem. For instance, the incidence of chronic nonhealing wounds, such as diabetic ulcers, continues to increase, have become a serious problem in the clinic because approximately 20% of diabetics are likely to develop chronic nonhealing foot wounds [3,4]. Moreover, according to the World Health Organization, more than 300,000 deaths occur annually as a result of fire-induced burns, with additional deaths attributed to other forms of burns (e.g., electricity, chemicals, radiation). Exuding wound care entails the need for dressing that quickly absorbs exudates, and exert effective moisture management during the healing of wounds. Currently available commercial wound dressings have confined performance and effectiveness due to limitations such as low-absorbency and high evaporation, non-occlusive nature and the requirement for secondary dressing for retaining a moist environment on the surface of the wound.

Market penetration of superabsorbent polymers has increased in the recent past with an emphasis on the medical sector. Centers for Disease Control and Prevention (CDC) forecast that that road mishap and accidents will lead to severe wounds and injuries result in key demand determinant for superabsorbent polymers. Road traffic injuries will become the 7th leading cause of mortality worldwide by 2030. Fact. MR, a US based market research firm forecast that superabsorbent polymer market will depict an impressive rise in the period from 2018 to 2027 with an annual compound growth rate of 7.3 % where US$ 8,300 Mn worth of medical superabsorbent polymers will be sold to the medical industry worldwide by 2027 [5]. A significant portion of revenues from medical superabsorbent polymers would be attributed to applications in advanced wound care. Faster healing process and recovery time together with a high liquid absorption capacity of medical superabsorbent polymers bode well for enhanced therapeutic efficiency requirement in advanced wound care. Safe collection, consolidation, and quarantine and disposal of infected secretions has become vital during the coronavirus (COVID-19) pandemic and demand for superabsorbent polymers is anticipated to witness steady growth in medical, personal care, and packaging applications.

Despite desirable properties of superabsorbent materials and demand growth, sustainability concerns have questioned high negative footprint of SAMs produced based on non-renewable resources [6]. The conventional superabsorbent materials are based on fully synthetic petroleum-based polymers where chemical crosslinkers (e.g., formaldehyde, glutaraldehyde, epichlorohydrin) which being used in the SAM preparation process poses risks and intrinsic cytotoxicity [7]. Synthetic based SAMs also cause generating a significant amount of waste due to non-biodegradability and non-recyclability.

Therefore, use of bio-based materials instead of those based on synthetic polymers is seen as a promising way to reduce pollution [8]. New challenges in this field are devoted to the research of new technologies which would allow the development of renewable materials for the existing requirements. The upward trend of sustainability and green products are now driving efforts for re- invention. Scientists across the globe are therefore working on the production of new eco-friendly alternatives. Due to health and environmental safety concerns and production cost of SAMs preparation [9], recently, there have been a great interest in preparing SAMs from the naturally available raw materials such as polysaccharides [10] including: starch [11], cellulose and cellulose derivatives [12], Chitosan [13,14], guar gum [15] etc. Among several alternatives of natural polymers, cellulose and its derivatives have been broadly used as a natural source for producing of SAMs and composites in many fields. They usually present the benefits of biocompatibility, biodegradability, water solubility, renewable, non-toxic, abundance, low cost, and environmental friendliness [16].

Cellulose being the most abundant natural polysaccharide with excellent biodegradability and biocompatibility has great potential to be harnessed for developing natural based superabsorbent. Carboxymethyl cellulose (CMC), a derivative of cellulose has been identified as one of the organic materials with super absorbent characteristic. A study conducted by Ugya et al. [17] has shown that *Eichhornia crassipes* can be used as low-cost, effective absorbent for the biosorption of Nickel (Ni2+) from refinery wastewater. Synthesis of environment friendly, super absorbent polymer material using *Eichhornia crassipes* (Water Hyacinth) was reported by Pitaloka et al. [18]. Nevertheless, extraction of cellulose from *Eichhornia crassipes* carboxymethylation and purification processes requires solvents and many other chemical reagents which are time consuming and costly process. Use of many chemical reagents and solvents is also not sound in terms of environmental sustainability. Therefore, the extraction process is yet to be improved in terms of minimal use of chemical reagents, use of the environment friendly solvents with enhanced process efficiency. The cost of carbon source is one of the significant factors affecting the cost of cellulose production. Hence utilization of low cost substrates like *Eichhornia crassipes* seems promising [19]. Development of superabsorbent from *Eichhornia crassipes* with minimum involvement of solvents and chemical reagents is timely in line with the recent development of new strategies for making use of low cost, easily available biological materials. Recent research and development have focused on the production of value added products from water hyacinth. *Eichhornia crassipes* has a relatively high cellulose content.

Although many studies have been conducted on the use of *Eichhornia crassipes* as an adsorption material for removal of dyestuffs and heavy metals in waste water treatment [20-23] subject to chemical and structural modifications in order to enhance the adsorption efficiency, its utilization as a superabsorbent is yet to be explored. Superabsorbent have potential applications in agriculture due to their capacity to influence on soil permeability, density, texture and infiltration rates of water through the soils. With global attention on climate change and food security, great interest has been created on application of superabsorbent as soil additives to increase the water retention of soils as a substitute for traditional moisture retention aids such as peat.

Eichhornia crassipes is a free floating, fresh water perennial aquatic plant grown in tropical and subtropical countries. This plant is capable of growing at a faster rate competing against nutrients and oxygen available in water bodies which affect adversely on the growth of other plants and animals in the ecosystem. Dense mats of plants also act as mosquito breeding grounds leading to increase in mosquito population [24]. *Eichhornia crassipes* is considered the worst aquatic weed in the world. For example, as explored by

Food and Agriculture Organization of the United Nations [25] the main problems arising from the growth of *Eichhornia crassipes* are (a) an enormous water loss through evapotranspiration, that alters the water balance of entire regions; (b) the impediment to water flow, that increases sedimentation, causing flooding and soil erosion; (c) the obstruction of navigation; (d) hampering fishing and dramatically reducing the catch and the source of food and income for local populations; (e) a drastic change in the physical and chemical properties of water and in the environment in the water bodies invaded, with detrimental effects on plants and animals; (f) the reduction of the activity of electrical power stations, jeopardizing the power supply of the country; and (g) a serious threaten to agricultural production, following the blockage of irrigation canals and drainage systems.

The economy of the countries concerned was therefore seriously affected in many aspects. Although *Eichhornia crassipes* is considered as problematic weed, findings from various studies have shown that this hydrophyte can be converted into a diverse range of value added products. Even this plant has potential to provide phytosterols to the pharmaceutical industry [26]. Efforts made to control and eradicate this weed have not been successful due to high costs and labour requirement and yet to find a sustainable solution. Therefore, alternative strategies for converting this weed into value added products will not only solve environmental problems but also helps sustain development. Utilization of *Eichhornia crassipes* as a renewable resource will contribute to the solution of socioeconomic problems associated with this aquatic weed [18]. A typical biomass from terrestrial plants is composed of 30-50 % cellulose, 20-40 % hemicellulose and 15-30 % lignin [27]. Therefore, it has the potential to be used as a source of cellulose for various applications. The high hemicellulose and cellulose content of the *Eichhornia crassipes* can be utilized for the production of various value added products [18]. Since carboxyl methyl cellulose has super absorbent characteristic, *Eichhornia crassipes* has potential to be utilized for development of superabsorbent by transforming its cellulose into carboxy methyl cellulose. *Eichhornia crassipes* is also being used for making compost fertilizer. Therefore, the advantage of using *Eichhornia crassipes* based superabsorbent will not only enable improved water retention in the soil, but also be composted to soil eventually without creating any negative impact to the environment.

Materials and Methods

Preparation of the precursor

Eichhornia crassipes was collected from tank near the Faculty of Applied Sciences, Wayamba University and harvested the petiole (figure 1a & 1b). Petiole was washed with tap water first and then with distilled water to remove adhering dirt particles from the surface. *Eichhornia crassipes* samples were cut into 2 cm pieces (figure 1.c) and dried at 105°C for 24hrs. Dried *Eichhornia crassipes* were treated with Potassium Hydroxide (KOH) solution with different concentrations ranging from 0.05 mole/l to 1 moles/l for a period of two hours. During the KOH treatment, samples were sonicated for a period of 30 minutes. Thereafter, the samples were dried at 105°C for 6 hrs.

Microwave irradiation

Microwave irradiation was conducted using a laboratory scale microwave oven. KOH treated and dried Eichhornia crassipes was placed in a cleaned ceramic bowl and placed in the chamber of the microwave oven. Power controller of the oven was set in low- range (270W) and mid-range (450W) for samples separately. Microwave exposure time varied from 1 to 6 minutes. The resultant activated Eichhornia crassipes was washed repeatedly with distilled water until the pH of the water is reaches 6-7 to remove the excess KOH and the sample was dried at 105°C for 6 hours (Figure 1d).

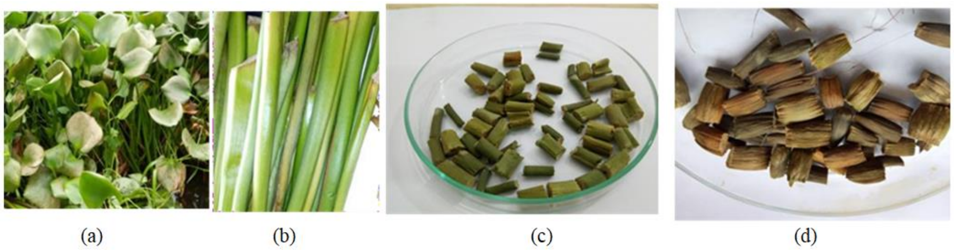
(a) (b) (c) (d)

Figure 1: (a) Densely grown *Eichhornia crassipes*, (b) *Eichhornia crassipes* petiole, (c) Petiole cut into pieces, (d) *Eichhornia crassipes* petiole after microwave irradiation.

Characterization of microwave irradiated *Eichhornia crassipes*

The surface morphology of superabsorbent was examined by Scanning Electron Microscopy (SEM). An X-ray diffractometer (XRD) was used to identify homogeneity and degree of crystallinity. Fourier-transform infrared spectroscopy (FTIR) was used to study the extent of hydrophilic functional groups developed during the activation process.

Swelling Index studies

Initial dry weight (WI) of microwave treated *Eichhornia crassipes* was measured and then placed in a beaker filled with distilled water. It was allowed to reach equilibrium for a period of 6-7 hours. Thereafter, the *Eichhornia crassipes* was taken out from the beaker, blotted with a tissue paper to remove the excess surface water and final weight (Wf) was recorded. The swelling index was calculated by using the following standard formula.

$$\text{Swelling Index (\%)} = ((W_f - W_i)/ W_i)\,100$$

Soil water-retention studies

The water retention studies were conducted with normal loam soil samples collected from the Applied Sciences faculty premises of Wayamba University. Plastic containers were used to ensure that water is lost from the containers only by evaporation. Each container was filled with 30g of soil, 3g of activated *Eichhornia crassipes* and 40 ml of normal water which was gradually added up into the containers and thereafter weight (W_i) of the container was measured. The experiments were also conducted with soil samples without *Eichhornia crassipes*. The sample containers were kept below room temperature and were weighed (W_f) once every three days until a constant weight is obtained. The rate of water evaporation (W%) of soil samples was evaluated by using the following formula.

$$W\% = ((Wi- Wf)/40)\ 100$$

Results and Discussion

The activating agent KOH functions as dehydrating agent that influence pyrolytic decomposition inhibiting the formation of tar and thereby enhancing the yield of carbon. The temperatures built in microwave irradiation are lower than that used in the physical activation process, thus development of the porous structure is better in the chemical activation method. Kim et al. [19] have examined and explained the process of KOH activation and development of porosity in graphitic nanofibers in which KOH triggers breaking of longer fibers to shorter fibers, expansion of the graphene layers by potassium intercalation (widening of pores) and exfoliation (involving a combined effect of separation of grapheme layers and also the breaking of fibers) result in generating the porosity. Similar mechanism can be expected pertaining to actions of KOH on cellulosic fibers in Eichhornia crassipes. The presence of potassium and oxygen bond triggers oxidation of cross-linked carbon atoms in the adjacent lamella during the process of activation. Surface functional groups are created at the edges of the lamella resulting removal of cross linking between adjacent lamella and also the formation of new functional groups on individual lamella. The lamellas of the crystalline are disturbed from their normal form into a slightly wrinkled or folded form. Potassium metal produced during the process of activation also intercalates in to the lamella of the crystallite. After the activation process, when the carbon material is washed with water, the potassium salts present in the carbon particles are removed by leaching. At the same time, the lamella cannot return to their original state, creating interlayer voids causing porosity and yielding high a surface area.

One of the key strategies to enhance the swelling rate is to increase micro, meso and nano porosity of the superabsorbent material while an enhanced number of hydrophilic functional groups. An SEM image of the surface of *Eichhornia crassipes* petiole just after KOH treatment and before microwave irradiation is given in figures 2a. High porosity and surface area in *Eichhornia crassipes* after microwave irradiation is clearly evident from the figures 2 (b) and 2 (c).

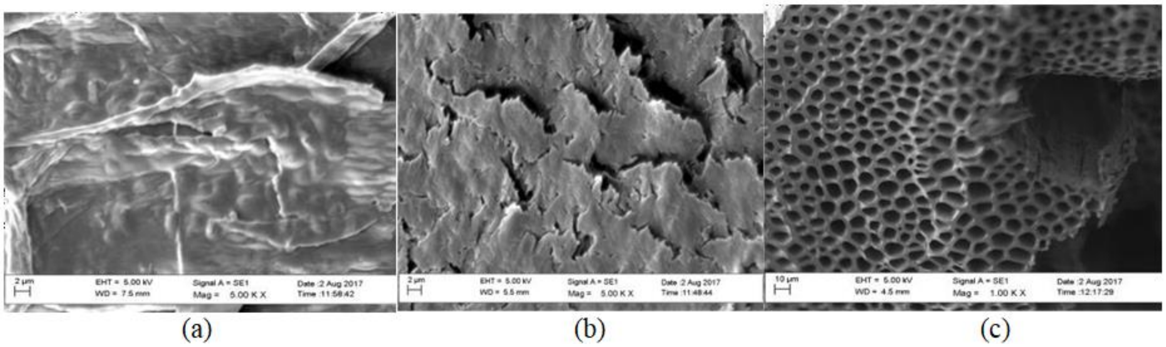

Figure 2: SEM images (a) surface of *Eichhornia crassipes* petiole before activation, (b) surface of *Eichhornia crassipes* petiole after microwave irradiation, (c) a cross section of *Eichhornia crassipes* petiole after microwave irradiation.

While KOH involve in the development of porosity and improve in specific surface area, it also creates hydroxyl (OH) functional groups on the carbon surface. The 'OK' groups formed on the carbon surface during the activation process gets transformed into hydroxyl (-OH) groups on washing with water by ion exchange reaction. KOH activation creates voids by the removal of potassium during washing with water while creating a surface that rich in oxygenated functional groups ultimately result in hydrophilic surface. This increased number of hydroxyl groups is confirmed by the pronounced band at 3500 cm^{-1} in the FTIR spectrum (figure 4). High porosity and hydrophilicity both contribute to enhanced capacity of water absorption. As per figure 3, a maximum swelling index of 1276 % was achieved in the present study at 0.1 M KOH concentration and swelling index decreased upon increased KOH concentration beyond 0.1 M. Higher KOH concentration causes excess carbon loss result in collapse of pore wall and loss of porosity and subsequently reducing the specific surface area. It also causes the collapse of structural integrity. Therefore, water absorption capacity and swelling index are reduced at higher KOH concentrations.

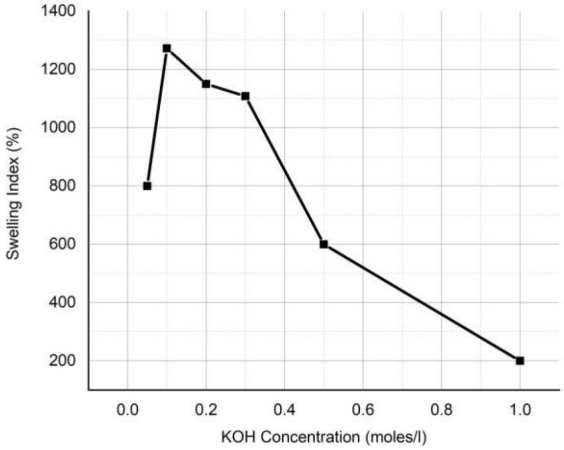

Figure 3: Effect of KOH concentration on swelling index.

FTIR spectrum of prepared superabsorbent is given in figure 4 and the band at 3500 cm^{-1} is attributed to stretching OH groups in cellulose, hemicellulose as well as surface OH groups created during the activation process.

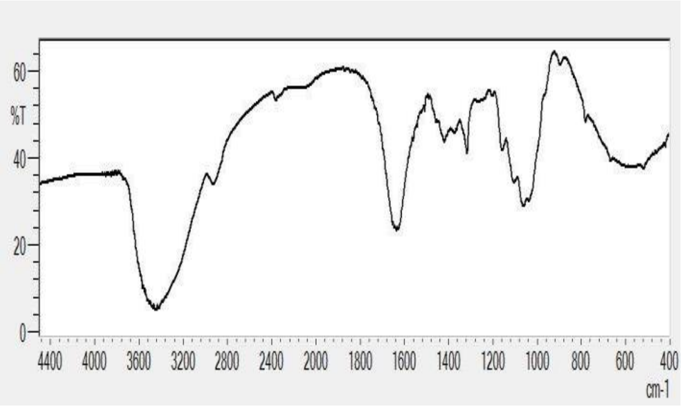

Figure 4: FTIR spectrum of *Eichhornia crassipes* petiole after microwave irradiation.

The presence of peak at 1635 cm^{-1} corresponds to stretching vibration of C=O in lignin. However, a band of a such intensity can't be expected since Eichhornia crassipes has a very small percentage of lignin. Therefore, this band can also represent a carboxyl group (COO^-) of carboxymethyl cellulose resulted from carboxymethylation of cellulose under microwave exposure in the presence of potassium hydroxide. Bands at 3000 cm^{-1} and 1050 cm^{-1} represent CH_3 bending vibrations of lignin and C- O stretch respectively. The FTIR characteristics obtained in this study corroborates with results obtained by Asrofi et al. [20] in their study on FTIR studies of cellulose fibers from *Eichhornia crassipes.*

XRD spectrum of prepared superabsorbent is given in figure 5 where the crystal structure of cellulose is evident from two prominent peaks at $2\theta = 22^0$ and 16^0. Peak broadening suggests amorphous character and non-homogeneity of the sample.

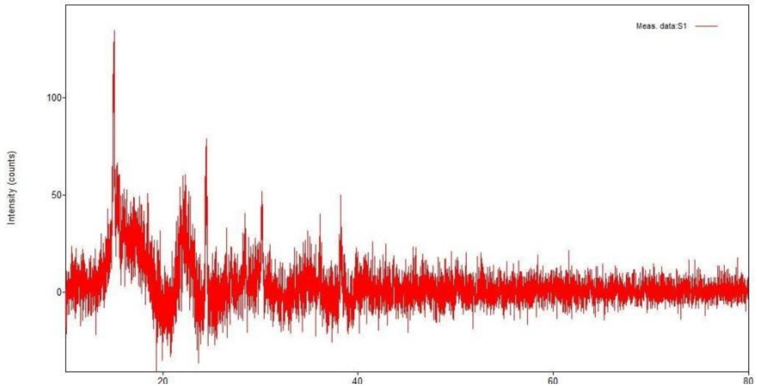

Figure 5: XRD pattern of the superabsorbent prepared based on water hyacinth.

A higher swelling index was achieved by exposure to microwave power of 450W than the power of 270W (figure 6) and optimum exposure time found to be 3 minutes (figure 7). The swelling index was increased with the increased exposure time up to 3 minutes. Swelling index was decreasing beyond 3 minutes exposure time. Thermal energy generated at microwave irradiation is accumulated at longer exposure time duration leading to thermal degradation of cellulose and hemicellulose while collapsing the structural integrity ultimately result in low water absorption capacity. With the mild reaction conditions, lignin present in the *Eichhornia crassipes* is not decomposed during the process thus conserving the structural integrity of the final product enabling absorption of a significant amount of water.

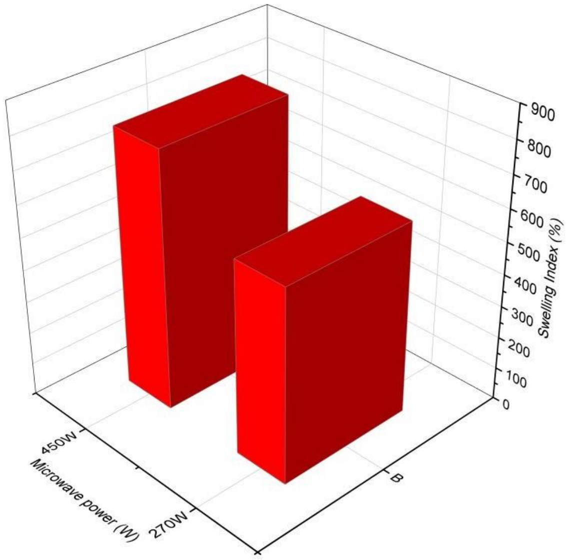

Figure 6: Effect of microwave power on swelling index.

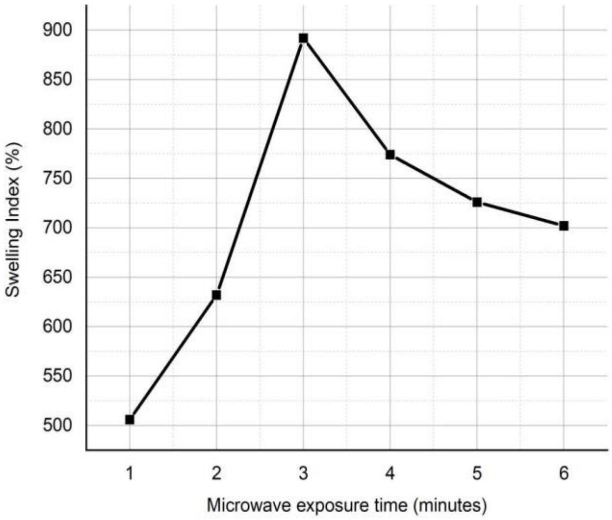

Figure 7: Effect of microwave exposure time on swelling index.

The water evaporation ratio was evaluated and the curves are given in figure 8 in which water evaporation ratio in soil samples containing the superabsorbent is lesser than that of control soil sample (without super absorbent). The rate of reduction in water content is lower in the soil samples with super absorbent while retaining water for 27 days whereas soil samples without super absorbent showed a high rate of water evaporation retaining water only for 15 days (figure 8). The study revealed that water retention was more pronounced in the soil amended with *Eichhornia crassipes* based superabsorbent.

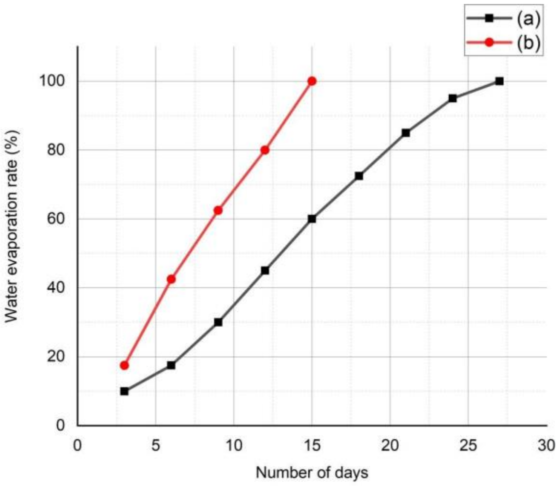

Figure 8: Water evaporation rate of (a) soil with superabsorbent, (b) soil without superabsorbent.

Conclusion

A natural based, environmentally friendly, biodegradable super absorbent can be developed from *Eichhornia crassipes* in a cost- effective manner in place of synthetic based superabsorbent which can be used as an effective soil amendment to improve the water retention in soil. The microwave assisted super absorbent development process can be achieved entirely excluding toxic solvents and chemical reagents while minimizing the time and energy consumption. The water absorption capacity and the swelling index of the developed superabsorbent is greatly influenced by the potassium hydroxide concentration, reaction time, power of microwave and microwave exposure time. Therefore, stringent control of process parameters is required to ensure the development of superabsorbent while conserving its structural integrity to impart a higher water absorption capacity. Higher water absorption capacity is attributed to highly porous structure, presence of hydrophilic functional groups in cellulose and hemicellulose, increased number of surface hydrophilic functional groups during the KOH activation process and carboxy methyl cellulose created during microwave irradiation.

The findings of this study disclose an innovative method for development of an eco-friendly, super absorbent from *Eichhornia crassipes* in a cost- effective manner excluding toxic chemical reagents to be used in agricultural and medical applications. *Eichhornia crassipes* based superabsorbent can facilitate not only solutions to global environmental and socio-economic issues associated with this hydrophyte but also climate change resilient agriculture and sustainable medical applications.

Conflict of Interest

The authors declare no conflict of interest.

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