



# **A Novel Method to Produce Cost Effective, Environment Friendly Superabsorbent from *Eichhornia crassipes* for Improved Water Retention in Soil**

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## **Authors' contributions**

This work was carried out in collaboration among all authors. Author JGSS designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author CANF overall supervision and mentoring. Author SNTDS Supervision of the project. All authors read and approved the final manuscript.

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## **ABSTRACT**

*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. Enhancement of agricultural production to cater for increased food demand is a global challenge which is aggravated by climate change and scarcity of water. Superabsorbent materials have been developed to improve water retention in soil but majority of superabsorbent are synthetic based and non-biodegradable. Therefore, this study is aimed at developing an eco-friendly superabsorbent from *E. crassipes* in a cost-effective manner and assess its efficacy on improved water retention in soil. *E. crassipes* was collected from tank nearby Faculty of Applied Sciences, Wayamba University of Sri Lanka and petiole was treated with potassium hydroxide

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(KOH) followed by microwave irradiation in which reaction conditions were optimized to obtain maximum water absorption and swelling capacity. Prepared superabsorbent was characterized using Fourier Transform Infrared Spectroscopy (FTIR), XRD and scanning electron microscopy (SEM) techniques. Efficacy of the superabsorbent on improved water retention was assessed using normal loam soil. 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 carboxymethyl cellulose created during microwave irradiation. Water absorption capacity of the superabsorbent 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 superabsorbent from *E. crassipes* in a cost-effective manner excluding toxic chemical reagents which can be used for improved water retention in soil effectively for climate change resilient sustainable agriculture.

**Keywords:** Superabsorbent; water hyacinth; biodegradable; microwave; chemical activation; agriculture.

## 1. INTRODUCTION

Superabsorbent (SA) materials are three dimensional highly hydrophilic networks which imbibe and retain significant amount of water compared to its original dry weight. Ultrahigh superabsorbent materials are capable of absorbing water even greater than 1,000% [1]. The high rate of water absorbency of these materials is attributed to presence of hydrophilic functional groups such as carboxyl, amino, amide, hydroxyl and sulfonic [2]. Hydrophilicity of these functional groups are dependent on pH and ionic strength of the medium which ultimately govern the water absorbent behavior of superabsorbent materials [3]. Superabsorbent materials have applications in diverse range of fields from personal hygiene products to waste treatment in industry. Common superabsorbent polymers are generally hygroscopic materials, which are mainly used in disposable diapers and industrial applications. Mechanism of water absorption by hygroscopic materials can be explained in terms of chemical and physical absorption. Chemical absorption involves water absorption through chemical reaction while physical absorption of water is accomplished by several mechanisms namely reversible changes of its crystal structure (e.g., silica gel and anhydrous inorganic salts), physical entrapment of water within nano- and meso-porous structure via capillary effect and hydration of functional groups. Water absorption can also be accomplished through combination of these mechanisms.

Superabsorbent are divided into two categories based on originality of raw material namely synthetic (petrochemical-based) and natural. Hsu-Feng et al. [4] classifies natural based polymers into three categories in terms of source of origin namely polysaccharides which are complex carbohydrates that are made up of repeating monomer units of monosaccharides widely available in nature and are of either plant, animal, or microbial origins, polypeptides which are mostly protein in nature and bacterial polyesters which are bio-macromolecules produced in nature by bacterial fermentation reactions of sugar or lipids. Majority of the commercially available superabsorbent products are manufactured based on synthetic materials and have poor performance in terms of biodegradability and environment friendliness. Synthetic material based disposable superabsorbent products (baby diapers, sanitary napkins etc.) have created environmental concerns due to non-biodegradability and toxicity. Conventional superabsorbent uses acrylic acid or acrylamide or derivatives such as N,N dimethylacrylamide which pose risk to human health. Acrylic acid is a strong irritant to the skin, eyes, and mucous membranes in humans. Animal studies involving acute exposure have demonstrated that acrylic acid can cause moderate acute toxicity by inhalation or ingestion [5]. Cost of petroleum based synthetic polymers has also been increased over the time. A review on superabsorbent polymer materials carried out by Mohammad et al. [6] highlights necessity of developing natural based

superabsorbent for the future since synthetic based superabsorbent incur high cost and pose health and environmental risks. Therefore, there is a growing interest in development of environment friendly superabsorbent based on natural polymers such as starch, cellulose, chitosan, agarose and alginate etc. Naturally originated precursors (polysaccharides and polypeptides) of superabsorbent have gained great attention of researchers due to their desirable properties- biodegradable, biocompatible, renewable, non-toxic and relatively low cost. Naturally derived superabsorbent hydrogels are made by crosslinking of these natural polymers in which major backbone is formed with natural polysaccharides. Moreover, polymer backbone can chemically be modified to improve desirable physicochemical properties. There are basically two approaches for the preparation of bio-based superabsorbent hydrogels namely graft polymerization and crosslinking reaction [7].

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 superabsorbent characteristic. A study conducted by Ugya et al. [8] has shown that *Eichhornia crassipes* can be used as low-cost, effective absorbent for the biosorption of Nickel ( $\text{Ni}^{2+}$ ) from refinery wastewater. Synthesis of environment friendly superabsorbent polymer material using *E. crassipes* (Water Hyacinth) was reported by Pitaloka et al. [9]. Nevertheless, extraction of cellulose from *E. crassipes*, carboxymethylation and purification processes requires solvents and many other chemical reagents which is time consuming and costly process. Use of many chemical reagents and solvents is also not sound in terms of environmental sustainability. Therefore, extraction process is yet to be improved in terms of minimal use of chemical reagents, use of environment friendly solvents with enhanced process efficiency. Cost of carbon source is one of the significant factors affecting the cost of cellulase production. Hence utilization of low cost substrates like *E. crassipes* seems promising [10]. Development of superabsorbent from *E. crassipes* with minimum involvement of solvents and chemical reagents is timely in line with recent development of new strategies for making use of low cost, easily available

biological materials. Recent research and development have focused on production of value added products from water hyacinth. *E. crassipes* has a relatively high cellulose content. Although many studies have been conducted on use of *E. crassipes* as an adsorption material for removal of dyestuffs and heavy metals in waste water treatment [11-14] 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.

*E. crassipes* is a free floating, freshwater 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 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 [15]. *E. crassipes* is considered the worst aquatic weed in the world. For example, as explored by Food and Agriculture Organization of the United Nations [16] the main problems arising from the growth of *E. 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 *E. crassipes* is considered as problematic weed, findings from various studies have shown that this hydrophyte can be converted into diverse range of value

added products. Even this plant has potential to provide phytosterols to the pharmaceutical industry [17]. 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 sustainable development. Utilization of *E. crassipes* as a renewable resource will contribute to the solution of socioeconomic problems associated with this aquatic weed [9]. A typical biomass from terrestrial plants is composed of 30–50% cellulose, 20–40% hemicellulose and 15–30% lignin [18]. Therefore, it has the potential to be used as a source of cellulose for various applications. The high hemicellulose and cellulose content of the *E. crassipes* can be utilized for the production of various value added products [9]. Since carboxy methyl cellulose has superabsorbent characteristic, *E. crassipes* has potential to be utilized for development of superabsorbent by transforming its cellulose into carboxy methyl cellulose. It is also being used for making compost fertilizer. Therefore, the advantage of using *E. crassipes* based superabsorbent will not only enable improved water retention in soil but also be composted to soil eventually without creating any negative impact to the environment.

## 2. MATERIALS AND METHODS

### 2.1 Preparation of the Precursor

*Eichhornia crassipes* was collected from tank near the Faculty of Applied Sciences, Wayamba University and harvested the petiole (Fig. 1a & 1b). Petiole was washed with tap water first and then with distilled water to remove adhering dirt particles from the surface. *E. crassipes* samples were cut into 2 cm pieces (Fig. 1c) and dried at 105°C for 24 hrs. Dried *E. crassipes* were treated with potassium hydroxide (KOH) solution with different concentrations ranging from 0.05 moles/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.

### 2.2 Microwave Irradiation

Microwave irradiation was conducted using a laboratory scale microwave oven. KOH treated and dried *E. 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 was varied from 1 to 6 minutes. The resultant activated *E. crassipes* was washed repeatedly with distilled water until pH of the water is reached 6-7 to remove the excess KOH and the sample was dried at 105°C for 6 hrs (Fig. 1d).

### 2.3 Characterization of Microwave Irradiated *E. crassipes*

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 activation process.

### 2.4 Swelling Index Studies

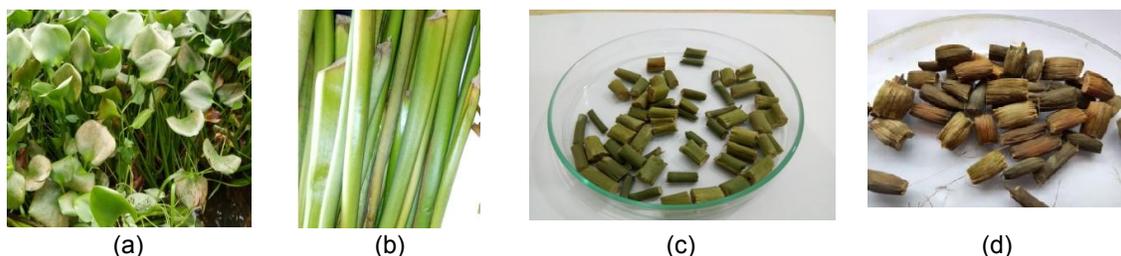
Initial dry weight ( $W_i$ ) of microwave treated *E. crassipes* was measured and then placed in a beaker filled with distilled water. It was allowed to reach equilibrium for a period of 6 – 7 hrs. Thereafter, the *E. crassipes* was taken out from the beaker, blotted with a tissue paper to remove the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

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

### 2.5 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 *E. 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 *E. crassipes*. The sample containers were kept under 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.

$$W\% = ((W_i - W_f)/40) 100$$



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

### 3. 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 is 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 graphene layers and also the breaking of fibers) result in generating the porosity. Similar mechanism can be expected pertaining to action of KOH on cellulosic fibers in *E. 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 crystallite 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 swelling rate is to increase micro, meso- and nanoporosity of the superabsorbent material while

enhanced number of hydrophilic functional groups. A SEM image of surface of *E. crassipes* petiole just after KOH treatment and before microwave irradiation is given in Fig. 2a. High porosity and surface area in *E. crassipes* after microwave irradiation is clearly evident from Fig. 2 (b) and 2 (c).

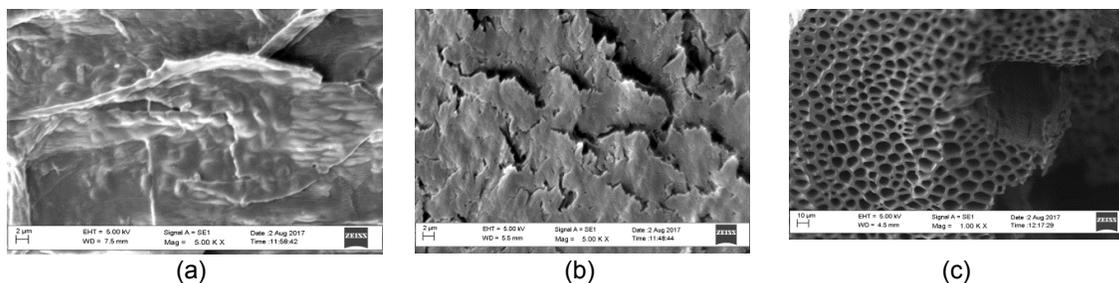
While KOH involve in development of porosity and improvement in specific surface area, it also creates hydroxyl (OH) functional groups on the carbon surface. The 'OK' groups formed on the carbon surface during the activation process gets transformed to hydroxyl (-OH) groups on washing with water by ion 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 with the pronounced band at  $3500\text{ cm}^{-1}$  in the FTIR spectrum (Fig. 4). High porosity and hydrophilicity both contribute to enhanced capacity of water absorption. As per Fig. 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 collapse of structural integrity. Therefore, water absorption capacity and swelling index are reduced at higher KOH concentrations.

FTIR spectrum of prepared superabsorbent is given in Fig. 4 and the band at  $3500\text{ cm}^{-1}$  is attributed to stretching OH groups in cellulose, hemicellulose as well as surface OH groups created during the activation process. The presence of peak at  $1635\text{ cm}^{-1}$  corresponds to stretching vibration of C=O in lignin. However, a band of a such intensity can't be expected since

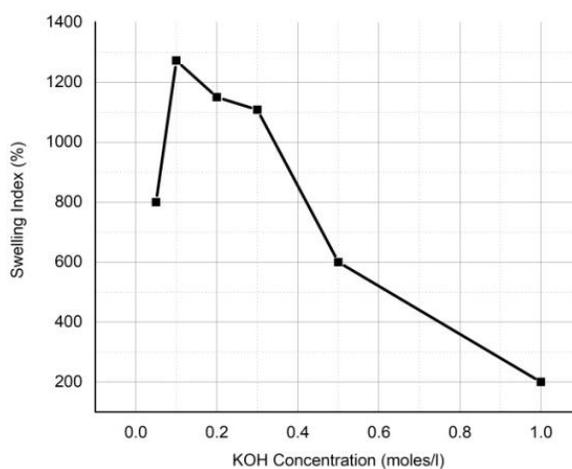
*E. crassipes* has a very small percentage of lignin. Therefore, this band can also represent carboxyl group ( $\text{COO}^-$ ) of carboxymethyl cellulose resulted from carboxymethylation of cellulose under microwave exposure in the presence of potassium hydroxide. Bands at  $3000\text{ cm}^{-1}$  and  $1050\text{ cm}^{-1}$  represent  $\text{CH}_3$  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 *E. crassipes*.

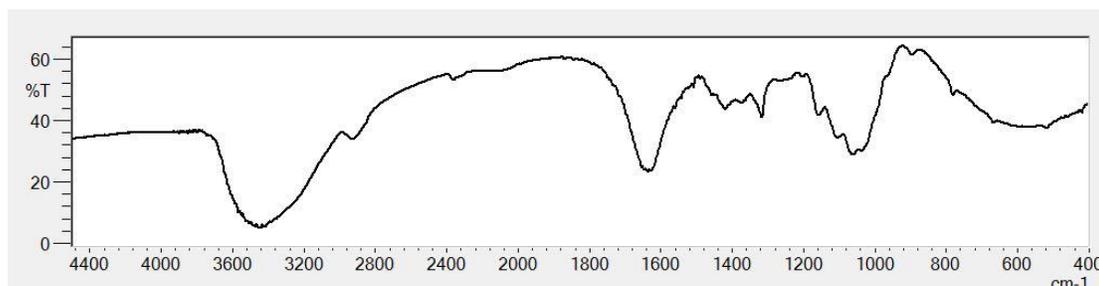
XRD spectrum of prepared superabsorbent is given in Fig. 5 where crystal structure of cellulose is evident from two prominent peaks at  $2\theta = 22^\circ$  and  $16^\circ$ . Peak broadening suggests amorphous character and non-homogeneity of the sample.



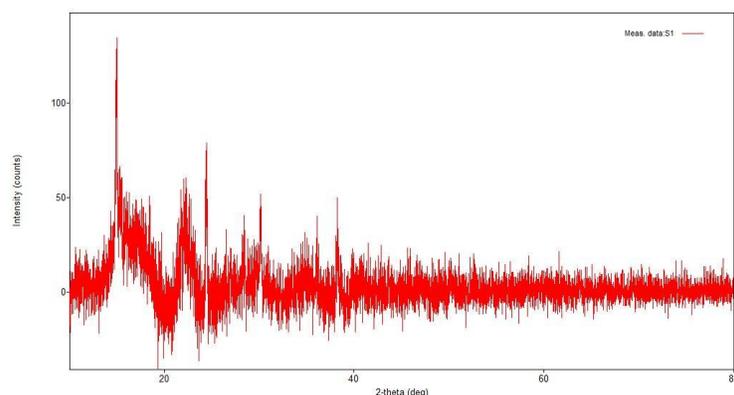
**Fig. 2. SEM images (a) surface of *E. crassipes* petiole before activation, (b) surface of *E. crassipes* petiole after microwave irradiation, (c) a cross section of *E. crassipes* petiole after microwave irradiation**



**Fig. 3. Effect of KOH concentration on swelling index**



**Fig. 4. FTIR spectrum of *E. crassipes* petiole after microwave irradiation**

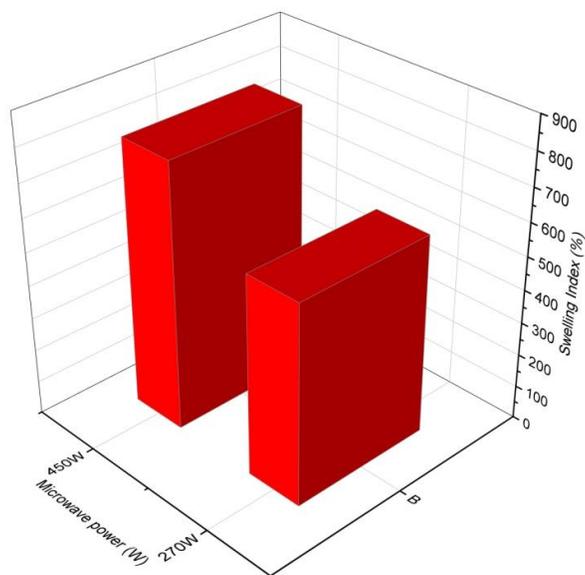


**Fig. 5. XRD pattern of the superabsorbent prepared based on *E. crassipes***

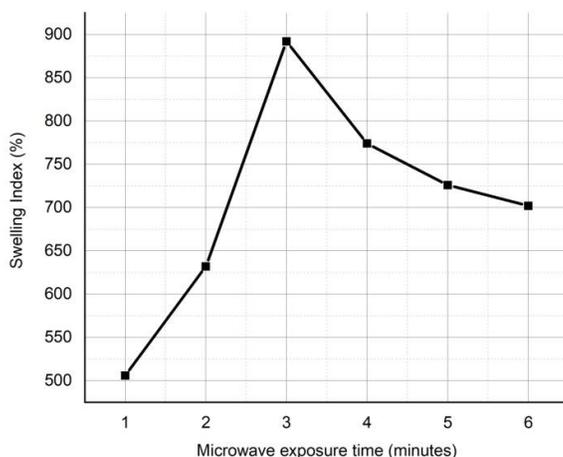
A higher swelling index was achieved with exposure to microwave power of 450W than the power of 270W (Fig. 6) and optimum exposure time found to be 3 minutes (Fig. 7). The swelling index was increased with the increased exposure time up to 3 minutes. Swelling index was decreasing beyond 3 minutes exposure 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 *E. crassipes* is not decomposed during the process thus conserving the structural integrity of the final

product enabling absorption of a significant amount of water.

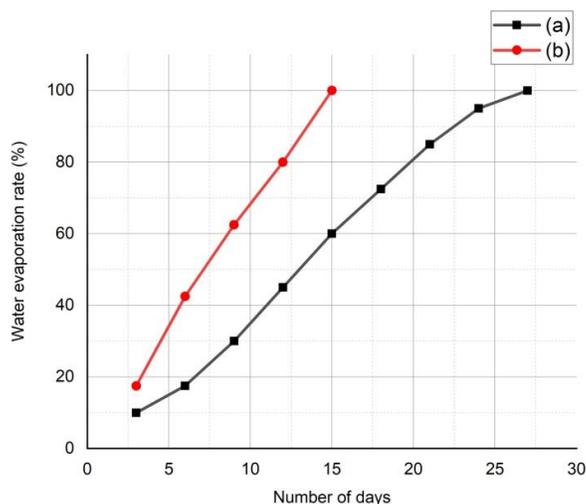
The water evaporation ratio was evaluated and the curves are given in Fig. 8 in which water evaporation ratio in soil samples containing the superabsorbent is lesser than that of control soil sample (without superabsorbent). The rate of reduction in water content is lower in the soil samples with superabsorbent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



**Fig. 6. Effect of microwave power on swelling index**



**Fig. 7. Effect of microwave exposure time on swelling index**



**Fig. 8. Water evaporation rate of (a) soil with superabsorbent, (b) soil without superabsorbent**

#### 4. CONCLUSION

A natural based, environment friendly, biodegradable superabsorbent can be developed from *E. 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 superabsorbent 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 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.

Findings of this study disclose an innovative method for development of an eco-friendly superabsorbent from *E. crassipes* in a cost-

effective manner excluding toxic chemical reagents which can be used for improved water retention in soil effectively. *E. crassipes* based superabsorbent can facilitate not only solutions to global environmental and socio-economic issues associated with this hydrophyte but also climate change resilient sustainable agriculture.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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