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REVIEW ARTICLE

Recent developments in the synthesis of superabsorbent polymer from natural food sources: A review

V. Yamuna and P. Kandhavadivu

Abstract

Superabsorbent polymer is a water-absorbing polymer that absorbs and holds large amounts of water, which it is widely used in agriculture and incontinent pads. In earlier days, superabsorbent polymer was synthesized purely from synthetic materials, which are now being replaced with a combination of natural materials due to the awareness of eco-friendliness and biodegradability. Many research works have been carried out in the preparation of natural-based SAPs. Hence this review focus on the general process of SAP preparation, its mechanism and the types of natural products which is used in food products in recent days for the synthesis of SAP. Test methods to be carried out for SAP and the future scope of SAP preparation without any synthetic products have to be analyzed to meet the environmental challenges for biodegradability.

Keywords: Biodegradability, Incontinent pads, Food products, Mechanism, SAP test.

Introduction

Superabsorbent polymers (SAPs) are three-dimensional hydrophilic network structures that can absorb much water and become hydrogel. Its application plays an important role in the use of incontinent pads which are concerned with health and hygienic aspects used by most of the women's and children's in the world. The type of incontinent pads includes baby and adult diapers, sanitary napkins, nursing pads, etc. The important property required for these incontinent pads is the absorbency of liquids. This absorbency property can be fulfilled and enhanced by two factors fibers and superabsorbent polymers used in the middle layer of pads.

As the fibers used in these pads is also responsible for the thickness of pads, nowadays, the manufacturers concentrate on the development of pads with the usage of minimum fibre content and maximum amount of superabsorbent polymers (SAP), so that ultra-thin pads can be developed.

The majority of SAPs are synthetic or petrochemical in nature. Acrylic acid, its salts and acryl amide are the most common monomers used for SAP preparation. The groups responsible for the water-holding capacity arise mainly due to the presence of amino, carboxyl and hydroxyl groups in the polymer chain (Ahmed, 2015)[1].

SAP Preparation Process

Superabsorbent polymer is prepared by the reaction of the monomer (containing water-attracting groups) with the initiator, crosslinking agent and sodium hydroxide. The monomer is then polymerized with the further reaction of the monomer and forms the sodium salt of the polymer. It is then crosslinked using a crosslinking agent to form the hydrogel which can be dried and powdered to form the superabsorbent polymer. An example of sap synthesis for a synthetic polymer is given below in Figure 1 (Jafari *et al.*, 2021).

A good example of a super-absorbing polymer is the sodium polyacrylate used in most incontinent pads. It is a sodium-containing crosslinked (network) polymer and osmosis is the process by which it absorbs the water.

Swelling Mechanism of SAP

The mechanism by which the polymer absorbs and holds the water to become a hydrogel happens when a sodium-containing polymer comes into contact with water, the

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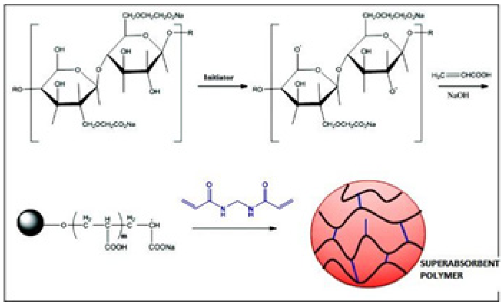


Figure 1: SAP preparation process

sodium tends to spread evenly between the network and the water. This indicates that some sodium atoms desire to leave the network and migrate to the water. These sodium atoms are replaced by water molecules when they leave. To keep the sodium concentration between the polymer and the water balanced, water swells the polymer network. The crosslinks between the chains keep them from dissolving or breaking apart in the water. Swelling mechanism for hydrogel formation is shown in below Figure 2 (Ma & Wen, 2020).

Method of Sap Synthesis

Superabsorbent polymers are made using a variety of synthetic processes. Among them, two main processes used to synthesize SAP are chemical and physical synthesis methods. The chemical synthesis process includes Bulk, Solutions/crosslinking, Suspension and Radiation polymerization. The physical synthesis process includes freeze/thaw cycle technology and hydrogen bond crosslinking. Other technologies being practiced in recent years include interface contact technology and in situ polymerization techniques (Rinaudo, 2008).

Environmental Concern of Synthetic Sap

Despite the overall convenience offered by pads that uses SAP, one major drawback is that they end up in landfills. Majorities of sanitary napkins are thrown away and buried in the soil. It is a catastrophic environmental concern because these pads take 500-800 years to biodegrade. It is harmful to the environment, and the substance used to make the incontinence pad can create skin allergies if it is not used carefully.

Nowadays, more research has been carried out on developing and using eco-friendly and biodegradable products. Due to the increase in awareness of biodegradability, natural products have been used along with synthetic materials for the preparation of SAP. Thus 100% usage of synthetic material as a superabsorbent polymer can be avoided in hygienic pads. So in consideration with the environmental and health effects, if a replacement for SAP using natural materials that cope up with the properties of synthetic SAP is found, more scope among the users can be gained and the environment can also be subjected to less harm.

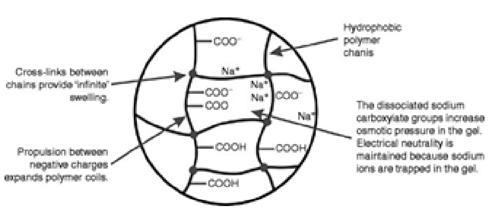


Figure 2: Swelling mechanism of SAP

Natural Source for SAP Preparation

SAPs can be prepared originally based on two main sources i.e., i) synthetic - petrochemical origin and ii) natural - polysaccharide and protein origin. Natural sources containing proteins and polysaccharides used for SAP preparation contain functional groups responsible for water absorption, as shown in Figure 3.

Polysaccharides are available in ample amount as it can be obtained from natural sources like plants and animals. The advantage of using polysaccharide in SAP preparation is it is less expensive and an organic renewable material. Alginate, chitosan, carrageenan, cellulose, agarose, starch, pectin, and certain gums are polysaccharides most often utilized (Martău *et al.*, 2019).

In the current scenario, few bacteria are also used to produce polysaccharides such as bacterial hyaluronan, gellan or xanthan, has also been reported (Bashari *et al.*, 2018). Classification of polysaccharides based on varying sources is given below in Figure 4(Yadav & Karthikeyan, 2019).

Literature Review

Synthesis of Polysaccharide Based Superabsorbent Polymer

Generally, the procedure used to make polysaccharide-based superabsorbent in most cases is graft copolymerization, where a suitable vinyl monomer on polysaccharide reacts

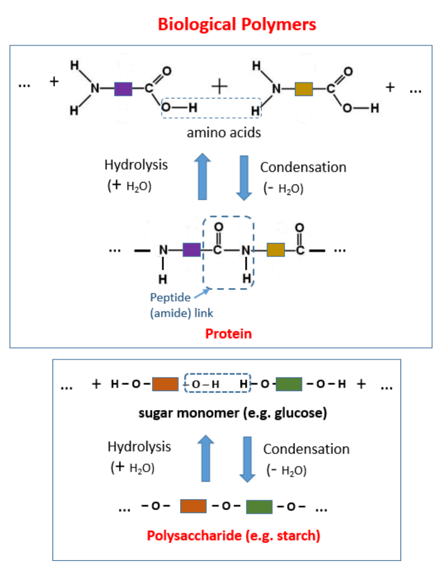


Figure 3: Functional groups of proteins and polysaccharides

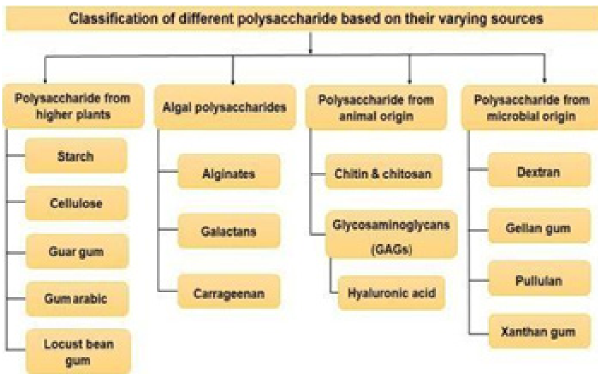


Figure 4: Classification of polysaccharides

with an initiator. The functional groups in the initiator interact to generate redox pair-based complexes. Then the homogenous cleavage of C-C bond produces carbon radicals on the polysaccharide substrate. These free radicals cause the vinyl monomers and crosslinking agent to graft polymerize on the substrate.

Sap from food Resource

Several food products act as thickening agents possess the property of high water absorbency is crosslinked with chemical agents to produce hydrogel for various end uses is described below.

Chia Seeds and Mimosa Pudica

Chia seeds are also called as *Salvia hispanica L*. is an oval-shaped seed of about 1mm in diameter. The seed has the color of brown and white. In both the color seeds, protein content varies from 16 to 17% and fibre content varies from 32 to 33% (Knez Hrnčič *et al*., 2020). It has many nutritional benefits and is widely used in food preparations. Chia seed can form gel when soaked in water due to the presence of mucilage fibre in the inner layer of seed coat. Since it has a high content of polysaccharides of about 71.22 percent, chia seed could be used to prepare superabsorbent polymer to replace synthetic SAPs.

In 2014, research was carried out to analyze the gelling properties of chia seeds and chia flour and compared the test results with other natural products like guar gum and gelatin, commonly used in food industries as thickening agents. The results showed the possibility of gel formation from chia seeds and its performance against water and oil holding capacity were similar to that of guar gum and gelatin (Coorey *et al*., 2014).

Mimosa pudica is an annual flowering plant called touch me not- "thottal chinnungi" in tamil. The parts of the plants are used for medicinal purposes to treat wounds, ulcers, piles, etc. It has the contents of polysaccharide glucuronoxylan, which can be extracted from the mimosa plant seeds and thus supports hydrogel preparation. It has the highest swelling capacity at alkaline pH and low swelling at acidic pH.

A study has been conducted recently on the analysis of physical and microbial properties of superabsorbent polymer synthesized from the food products viz. chia seeds *(Salvia hispanica L.)*, chia flour and Mimosa pudica hydrogel (MPH) in order to use it in the middle layer of sanitary napkins.

The research on these natural products was carried out using chia seeds, flour extracted from chia seeds after grinding and drying and the hydrogel extracted from mimosa seed. Hydrogel was prepared from mimosa seed by soaking it in water for 12 hours and then heating the gel at 50°c for 30 minutes in a shaker. Gel prepared after the treatment was separated from the seeds by filtering it using a cotton cloth. The gel extracted was dried and powdered thus serves as the superabsorbent polymer. All the three products were analyzed for its physical properties and their performance is shown below in Figure 5 (Peng *et al*., 2021).

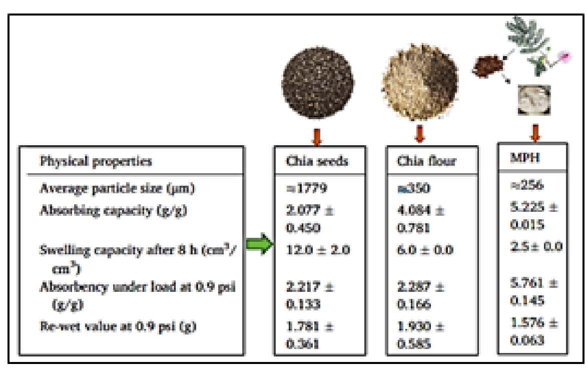
Antibacterial properties of all the natural products were analyzed by a bacterial test carried out against *S. aureus* and *E.coli*. The biodegradability of the products were also analyzed by carrying out soil burial test.

The results proved that *Mimosa pudica* gel shows better performance while analyzing the physical, antibacterial properties and biodegradability test compared to the other two products. It can be considered the most suitable material for SAP preparation.

Orange and Avocado

Cell walls of orange peel and avocado contain insoluble polysaccharides, pectins, cellulose and hemicellulose which can be used for the preparation of hydrogel. Various studies focused on the extraction of polysaccharides from orange peel.

Kiara Nirghin utilized oranges and avocados for the research work to synthesize hydrogel from the fruit peel as a way to combat drought in the fruit industry. For 63% of polysaccharides content is found in orange peels and 54% in natural oil of avocado skin. The process undergone here for SAP preparation is emulsion polymerization using avocado skin oil with orange peel solution. These combinations were then sun-dried and used for plantation testing. When



Physical properties	Chia seeds	Chia flour	MPH
Average particle size (μm)	≈1779	≈350	≈256
Absorbing capacity (g/g)	2.077 ± 0.450	4.084 ± 0.781	5.225 ± 0.015
Swelling capacity after 8 h (cm^3/cm^3)	12.0 ± 2.0	6.0 ± 0.0	2.5± 0.0
Absorbency under load at 0.9 psi (g/g)	2.217 ± 0.133	2.287 ± 0.166	5.761 ± 0.145
Re-wet value at 0.9 psi (g)	1.781 ± 0.361	1.930 ± 0.585	1.576 ± 0.063

Figure 5: Physical properties of natural products

comparing the water absorbency of acrylic SAP (74 percent), the orange peel mixture shows 76% of water absorbency, revealing the best performance (Rose *et al.*, 2005).

Guar Gum

Guar gum is a galactomannan polysaccharide extracted from guar beans which has thickening and stabilizing properties thus used in the food industry, feed and agriculture etc. (Mudgil *et al.*, 2014). Few research works have been carried out in hydrogel formation from gum of guar to use in soil where it can increase the soil moisture to help the farmers save their crops in case of water scarcity.

Guar gum has been treated with a monomer Methylmethaacrylate in the presence of a crosslinking agent Polyethylene glycol. The hydrogel synthesized was tested against the development of sugarcane crop in the field and the study was carried out for 20 days to observe soil moisture content. The observation shows 18% higher moisture content of the hydrogel applied field than the without hydrogel (Songara & Patel, 2021).

Novel technique adopted in analyzing the biodegradability of hydrogel produced by the reaction of guar gum (GG), acrylic acid (AA) and ethylene glycol di methacrylic acid (EGDM) as crosslinker. Soil burial test was carried out for 77 days and soil moisture content and porosity were also observed. Applying guar gum-based hydrogel increases the water holding capacity of soil to 54% and porosity to 9%, which is a wealth for agriculture (Thombare *et al.*, 2018).

Research has also been carried out to prepare hydrogel using guar gum by reacting it with ammonium persulphate as initiator, cobalt III chloride and acrylic acid monomer. All these compounds' concentrations varied to optimize the proportion and analyze the water-holding capacity (Behera *et al.*, 2018).

Cabbage

Cabbage (*Brassica oleracea*) comes under the cruciferous family is rich in vitamins and fibers. Cabbage is an edible vegetable with many nutritional benefits and used in the medical field in powder form or as extracts. It has high water holding capacity and can be used to prepare SAP.

A study on wound dressing for trauma treatment includes cabbage extract of 1 to 10% as a pH indicator and chemicals for preparing hydrogel patch. The cabbage extract was observed to act as color change indicator during inflammation and bleeding and hence avoid bacterial infection (KR101539675B1). Waste cabbage is used to synthesize superabsorbent polymer in a research work carried out using monomers 2- acrylamide-2-methyl-1-propane sulfonic acid and acrylic acid. This work helps reduce pollution due to the increase of biowaste in the environment. Potassium persulfate acts as initiator and N, N′-methylene-bisacrylamide was used as crosslinking agents. The findings revealed that the gel produced from the polymer shows good water absorbency under pure water as 1914g/g, distilled water as 1726g/g, tap water as 306g/g and 0.9% NaCl solution as 114 g/g (Zhang *et al.*, 2021).

Potato

Potatoes *(Solanum tuberosum)* are starch rich edible food. Potato starch contains polysaccharides, namely amylose and amylopectins in the range of 15 to 25% which can be genetically modified in order to get more yield percentage of about 70 to 90% (Tomasik, 2009). As the potato is rich in metal salts, the starch can be used widely in gel preparation. Potato starch can be combined with proteins due to the presence of negative charges in electrolytes and produces complex compounds used for various applications.

Research work focused on the preparation of superabsorbent polymer from normal potato starch and sweet corn potato starch by reacting it with a monomer acrylic acid, crosslinking agent glycerol and an initiator $K_2S_2O_2$. NaOH was used to gelatinize the starch and neutralize the acetic acid. Ratios and concentrations of the reagents were varied to optimize the polymer preparation to obtain maximum water and gel strength uptake. Potato starch shows 600 times more water uptake than sweet potato starch and is hence chosen for SAP preparation. Concentrating extracted solution was synthesized by varying the concentration of the chemicals in order to avoid the loss of protein and polysaccharide to less than 10%. The optimized concentrated solution was tested against 0.9% saline solution, cordyceps extract, sweet potato extract and bovine serum albumin, shows better results (Liu *et al.*, 2011).

As the protein can be used as an alternative source for polysaccharide in preparing natural superabsorbent polymer, potato protein concentrate (PPC) was procured in a study and acylated with five different agents like Succinic anhydride, 1,2,3,4-butanetetracarboxylic acid, ethylene diamine tetra acetic dianhydride, citric acid and ethylene diamine tetra acetic acid in order to evaluate its water holding capacity. The results showed higher absorption in water and saline solution for the polymer treated with succinic anhydride(SA). A modified version of SA can also increase the water holding capacity (Capezza *et al.*, 2019).

Sugarcane Bagasse

Sugarcane bagasse (*Saccharum officinarum*) is a dry pulp material obtained after crushing and extracting juice from sugarcane stalks. The waste pulp material can be converted into many useful products. This waste is utilized in various chemical industries, construction industries, biofuel manufacturing, and absorbents (Ajala *et al.*, 2021). Untreated raw sugarcane bagasse contains around 40 to 50% of cellulose, and various chemical treatments can increase cellulose content yield from 50 to 90% (Mahmud & Anannya, 2021).

The sugarcane bagasse is a heterogeneous polysaccharide containing the chemical compounds xylose, glucose, galactose, and arabinose. As a polysaccharide compound, it can be used for the preparation of superabsorbent polymer using different types of crosslinking agents.

A research work concentrated on the synthesis of biodegradable superabsorbent polymer using the monomer sugarcane bagasse cellulose (SCB) which was treated with NaOH and milled to reduce its size before the polymerization process and acrylic acid (AA) with the crosslinker N,N-methylene-bisacrylamide and the initiator ammonium persulfate/mixture of Ammonium persulfate and sodium sulfite. Acrylic acid was neutralized by using NaOH in the solution and the proportion of monomers were varied in order to optimize the solution. The reaction process takes place in a rotating twin-screw extruder using radical graft polymerization (Figure 6). The results showed that superabsorbent polymer produced using ammonium persulfate/sodium sulfite as a pair initiator had higher swelling ability (Neamjan *et al.*, 2019).

Sugarcane baggase was treated with monomers acrylamide and methyl methacrylate in a research work with the crosslinking agent N,N-methylene-bisacrylamide and potassium persulfate as initiator. High absorption and swelling capacity of hydrogel was achieved by varying the temperature, pH and monomer concentration (Mondal *et al.*, 2022).

Sap Characterisation

Sap characterization and performance evaluation can be done using various instruments and test methods. Pores size of hydrogel can be identified by scanning electron microscope(SEM), particle size and shape of dried powder obtained from hydrogel can be identified by transmission electron microscope (TEM). Presence of functional groups in the polymer can be analyzed from the results of fourier transform infrared spectroscopy (FTIR) and thermal resistivity can be observed by Thermo gravimetric analysis(TGA). To analyze the performance of water absorption, holding and retaining capacity of polymer produced from the hydrogel and its performance against bacteria and degradability, various test methods has to be carried out as follows: Free-absorbency Capacity, Tea-bag method, centrifuge method, sieve method, absorbency under load (AUL), wicking capacity, swelling rate, swollen gel strength, soluble fraction, residual monomer, ionic sensitivity, soil burial test for biodegradability, Antimicrobial and Toxicity test.

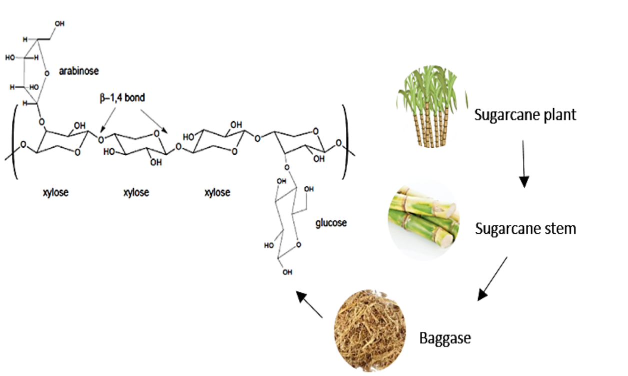


Figure 6: Process cycle of sugarcane to bagasse and its compounds

Conclusion

Based on the literature review, it can be concluded that most of the research works have been carried out in the area of natural superabsorbent polymers in order to have biodegradable products. Since the SAP is mostly used in hygienic pads, eco-friendliness is a major concern. Hence future research works can be focused on using natural food products as monomers for SAP preparation with natural crosslinking agents like Genipin, Vanillin, tannic acid, caffeic acid, procyanidin, proanthocyanidin, epigallocatechin gallate and citric acid etc. so that the synthetic material usage can be avoided totally to achieve an inexpensive and non-toxic Superabsorbent polymer preparation (Klein & Poverenov, 2020).

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