

FORMULATION AND EVALUATION OF SUPERABSORBENT HYDROGEL FROM NATURAL POLYMER

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ABSTRACT

Objective: The objective of the present study was to synthesize the hydrogel from natural polymer and evaluation of its physical and chemical properties.

Methods: Hydrogel was synthesized using graft co-polymerization technique from wheat starch, by crosslinking with acrylic acid. The product was purified, dried and micronized. It was then evaluated for water absorption and retention property at varying pH, FTIR, PXRD and Thermal analysis, microscopic, micromeritic and stability studies etc. Furthermore, the effect of NaOH treatment on prepared hydrogel material was studied.

Results: Result of the studies revealed that superabsorbent hydrogel (SAH) product shows good water absorption capacity of 120g/g at neutral pH. Maximum water absorption capacity was at pH 9 which is 146.28g/g. Product shows good thermal stability, less cohesiveness and is amorphous in nature. In hygroscopicity study weight gain by SAH was 6.65% only while for unpurified SAH and NaOH treated SAH, it was 10.5% and 23.42% respectively. NaOH treatment shows a decrease in water absorption capacity by more than 40% also there is change in surface morphology of the product. Additionally, hygroscopicity was more and degradation rate was faster for NaOH treated hydrogel.

Conclusion: Crosslinking with acrylic acid can form superabsorbent hydrogel material from the natural polymer such as wheat starch. The product shows excellent water absorption and retention capacity. pH affects water absorption capacity and shows maximum at pH 9 and at lower and higher pH it decreases to a significant level. There was decline in water absorption capacity and increase in hygroscopicity, when NaOH treatment is given to the SAH powder.

Keywords: Hydrogel, Superabsorbent, Graft co-polymerization, Crosslinking, Natural polymer, Water absorption, Starch

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INTRODUCTION

Hydrogels are the hydrophilic cross-linked polymer matrix that can retain a significant amount of water within their structures and swell without dissolving in water [1]. The hydrogels that can absorb very large amount of water and retain it even under pressure are called superabsorbent hydrogels [2]. After absorbing large amount of water, they become soft, transparent or translucent and mostly remains biocompatible [3]. Natural products like hyaluronic acid, fibrin and collagen, derivatives of natural materials such as chitosan and alginate possess the properties of hydrogel [4]. Likewise, mucilage containing plant products viz. Garden crest seeds, sweet basil seeds and Psyllium husk also show hydrogel like action when come in contact with water [5]. Preparation of this compound in laboratories from different substances is the present area of research in the field of polymer science.

Hydrogels are having applications in agriculture [6–8], drug delivery [9–11], tissue engineering [12–14], contact lenses [15, 16] and water purification [17, 18]. Dry hydrogel material mixed into the soil which in turn absorbs and retains more water that keeps plants hydrated even in time of drought. Hydrogel's biocompatibility and protective nature which causes encapsulation of hydrophilic drug molecule are excellent drug carriers in drug delivery. Contact lenses prepared from silicon hydrogel shows increased oxygen permeability with water content providing optimal comfort and eye health [19]. They are also found useful in early scaffold formation to repair damaged tissues of the body. In water purification, hydrogel was found useful to remove heavy metals like nickel and mercury. Recently, the global issue of oil spillage in the oceans is resolved by preparing hydrogel [20].

Hydrogel is prepared using Physical gel and Chemical gel method. In the former method, the hydrogel is prepared with some external activation technique and is having noncovalent interactions, whereas the latter method is mostly permanent, do not need any

activation technique and formed by cross-linking network formed with covalent bonds. Methods of physical gel include crystallization, stereo complex formation, hydrophobized polysaccharides, ionic interactions and protein interaction whereas chemical hydrogel are formed via crosslinking by radical polymerization, addition and condensation polymerization, gamma and electron beam polymerization. Based on the method of preparation, hydrogels are classified as Homopolymeric hydrogels [21], Copolymeric hydrogels [22] and Multipolymer interpenetrating polymeric hydrogel (IPN) [23]. They may be amorphous, semicrystalline or crystalline. Moreover, on the basis of electric charges imposed on it, they are nonionic, ionic, amphoteric or zwitterionic (like polybetaines).

Recently, hydrogels are researched in order to improve its properties like water absorbency and its applicability in drug delivery. Starch used is available in abundance and cost effective also the method of graft copolymerization does not need any costly instrumentation or reactants. Since all chemicals used are having excellent water solubility, it is easy to remove any unused reactants by simply soaking product in excess water quantity. Hence in the present study, hydrogel was prepared from natural polymer wheat starch by crosslinking with acrylic acid. Evaluation of prepared hydrogels for various parameters like gel fraction, water absorption capacity at various pH conditions, hygroscopicity, surface morphology and micromeritic properties was undertaken.

MATERIALS AND METHODS

Chemicals

The starch was sourced from homemade wheat powder. Other chemicals and their respective manufacturers are: Sodium hydroxide (Research Lab Fine Chem. Industries Mumbai), urea and ammonium persulphate (Powder Pack Chem, Mumbai), N,N-methylenebisacrylamide and acrylic acid (Loba Chemie Pvt. Ltd.). Distilled water was used as a solvent. All the chemicals used were of AR grade.

Preparation of superabsorbent hydrogel (SAH)

The SAH was prepared by the method described previously by Ting Guo Liu *et al.* [24]. In brief, 3g of starch was added to 55 ml water. It was then gelatinized by slow addition of 36 ml NaOH solution (25% w/v). After vigorous shaking, the previous solution was added with 24 ml urea solution (25% w/v) and 12 ml ammonium persulphate solution (2.5% w/v). After half an hour continuous stirring, 12 ml N,N-methylenbisacrylamide solution (0.5% w/v) was added followed by addition of mixture containing 40 ml acrylic acid in 70 ml of water. The reaction mixture was stirred well and sealed with aluminum foil and was kept at 68 °C for 5 h. The sticky gelatinous mass i.e. modified starch obtained was then cut into smaller pieces and purified by immersing overnight in ethanol (75% v/v). After purification it was dried in hot air oven at 65 °C for 72 h. After 24 h of drying, it was again cut into smaller pieces. The dried SAH was then micronized and evaluated for various parameters.

Residual solvent and reactants were removed from prepared SAH by allowing it to fully swell in water and washed with excess amount of water. This was further dried in hot air oven at 65 °C for 72 h and micronized again.

Evaluation of prepared hydrogel

The following tests was undertaken for evaluation of SAH.

Determination of gel fraction

Gel fraction was measured and evaluated to study the effect of concentration of acrylic acid on gel fraction (n=3). For this, acrylic acid in different concentration viz. 10, 20, 30, 40, 50, 60 ml was added to the reaction mixture and the gel fraction of the product of each was determined by using method described by Md. Murshed Bhuyan *et al.* [25]. In brief, excess water was added to 2g SAH and allowed to swell for 24 h. The gel was filtered on the pre-weighed container (ASTM Sieve no. 120) and dried to constant weight in an oven for 72 h. This removed unreacted fraction of reactants from SAH. The gel fraction was then calculated by using the formula:

$$\text{Gel Fraction(\%)} = \frac{W_t}{W_0} \times 100$$

Determination of swelling properties by Tea-bag method

To determine the swelling capacity, 1g of the purified SAH was added to pre-weighed tea bags [26]. This was then immersed in 1 L capacity glass beaker filled with water and allowed to swell for 24 h. After 24 h, the bags were removed from the beaker and excess water was drained by hanging it for 2 h. After 2 h, the swollen tea bags were weighed repeatedly after an interval of 15 min till constant weight was achieved. The water absorption was calculated by the following formula:

$$\text{Water Absorption (\%)} = \frac{W_t - W_1}{W_1} \times 100$$

Effect of NaOH treatment on water absorption capacity

Micronized SAH (10g) was slowly added with constant stirring to 500 ml of 0.45 N NaOH and allowed to swell for 24 h at room temperature. Excess NaOH was removed by repeated washings with sufficient volume of water till neutral pH. The washed SAH was then dried at 65 °C for 72 h. The changes in the various properties were tested by performing water absorption and water retention study, DSC, PXRD and SEM. The results were compared with the purified SAH.

Effect of pH on the water absorption

To study the effect of pH on the water absorption capacity of hydrogel, solutions of various pH viz. pH 1, 3, 5, 7, 9, 11 and 12 were prepared. SAH (1g) was placed in pre-weighed tea bags and these bags were immersed in various pH solutions mentioned above. The SAH was allowed to swell for 24 h followed by draining of excess water on teabags. Water absorbed by SAH was determined by taking the final weight at different pH and observations were noted.

Hygrosopicity studies

It is necessary to find out the nature of hydrogels in terms of moisture absorption capacity as it influences formulation

parameters and storage conditions. The study was conducted on all types of SAH; unpurified, purified and NaOH treated. Weighed quantity of 5g of SAH samples (n=3) was transferred to pre-weighed Petri plates placed in a desiccator containing a saturated solution of ammonium sulphate (section 5.11 of the European Pharmacopoeia 6th edition). The saturated solution of ammonium sulphate produces relative humidity of 80±2 %. The amount of moisture absorbed was determined by weight gain of samples after 24 h.

FTIR, PXRD and SEM analysis of hydrogel

FTIR gives idea about the presence or absence of functional groups in individual reactants before reaction and in product after reaction. The FTIR spectra of SAH, NaOH treated SAH and starch was obtained from Fourier transform infrared spectrophotometer in the range 400 to 4000 cm⁻¹ (Shimadzu IR Affinity-1). The crystallinity of the purified SAH was tested by powder X-ray diffraction study (Bruker AXS D8 Advance). The study was performed using Cu radiation generated at 25 mA intensity and 35 kV voltage and at wavelength 1.5406 Å. The XRD pattern was obtained between 3 ° to 90 ° (2θ). The scanning electron microscopic photograph of the SAH and NaOH treated SAH was analyzed for the surface morphology using SEM microscope (Carl Zeiss, Supra 55, Germany) at 20 kV.

Differential scanning calorimetry and Thermogravimetric analysis (DSC and TGA)

DSC and TGA patterns were obtained by using heat flow method [27]. DSC pattern shows exothermic and endothermic crests which are helpful in determining polymorphic nature, melting point, co-crystal formation, heat of fusion, glass transition temperature and endothermic and exothermic behavior of a compound. TGA shows changes in weight of the compound with a gradual increase in temperature and is useful in getting information about exact drying temperature, solvate/hydrate form, volatile components, sublimation, compatibility and thermal stability of the compound [28]. The thermograms were obtained for starch, SAH and NaOH treated SAH by using differential scanning calorimeter (TA Instruments SDT Q600 V20.9 Build 20) with increasing the temperature at the rate 10 °C per minute upto 350 °C. The sample weight taken was approximately 5 mg. The inert environment was maintained by purging of nitrogen throughout analysis.

Particle size analysis and micromeritic characteristics

Particle size analysis (Polydispersity Index-PDI and mean particle diameter) of the SAH, purified hydrogel and NaOH treated hydrogel was performed by using a particle size analyzer (Litesizer 500, Anton Paar) using acetone as a solvent. The flow properties of the SAH were determined by the fixed funnel free standing cone method. Consolidation properties of the SAH and NaOH treated SAH were determined by bulk density testing apparatus. The weight of the sample and consolidation after tappings was recorded and bulk density, tapped density, Hausner's ratio and Carr's compressibility index was recorded.

Kawakita analysis

Kawakita plot gives an idea about cohesion and compactibility of the powders [29]. The Kawakita Equation tells about what happens when a powder in the column is allowed to consolidate freely by application of pressure [30]. In this, comparison of pressure-volume reduction relationship was evaluated with tappings on bulk density testing apparatus. The reduction in volume was noted after every 5 tappings till constant volume. The degree of cohesion and compactibility was calculated from the following Kawakita Equation:

$$\frac{N}{C} = \frac{N}{a} + 1/ab$$

Stability studies

Stability of unpurified SAH, purified SAH and NaOH treated SAH was performed for a duration of three months, in accordance with ICH norms. Stability study gives us information about the changes taking place in compound over time period and varying environmental conditions like temperature and humidity. The study was done by keeping 5g. of samples in a Petri plate at 25 °C±2 °C (60% RH) and

45 °C±2 °C (75%RH) [31]. After three months, the samples were observed and analyzed for any visible changes, change in weight and swelling characteristics.

RESULTS AND DISCUSSION

Formulation of SAH and mechanism of reaction

Graft co-polymerization is a technique by which natural polymers can be converted into superabsorbent material. Here, natural polymers undergo crosslinking with suitable agent to form ultra-high molecular weight polymers with crosslinked three-dimensional network structure. Starch is having a good number of hydrophilic groups and is biodegradable, inexpensive and easily available polymer. Sodium hydroxide gelatinizes starch and also aids in neutralization of excess acrylic acid. Neutralization causes increase in hydrophilic groups-COONa so as to increase the osmotic driving force causing more water absorption. Ammonium persulphate acts as initiator and N, N-methylenebisacrylamide as a crosslinker [32].

Evaluation of prepared hydrogel

Results of various evaluation tests performed and discussion is as follows:

Determination of gel fraction

In the formulation of hydrogel, concentration of acrylic acid is found to have an impact on the gel formation and hence effect of amount of acrylic acid added on the gel fraction was determined. At lower concentration, SAH yield was less and yellowish white in color. The color intensity increases towards brown as concentration of acrylic acid increases. The results show that at lower concentration of acrylic acid, gel fraction is considerably low while maximum gel fraction is observed with 40 ml for 3g of starch (table 1).

Determination of swelling properties by tea-bag method

Purified SAH was found to absorb water to the tune of 120.002±3.3299 g. The rate of swelling found is dependent on the particle size and it also slows down if hydrogel forms lumps. This

implies that rate of swelling is directly proportional to surface area exposed to water.

Table 1: Amount of acrylic acid and respective gel fraction in SAH

S. No.	Amount of acrylic acid (ml)	Gel fraction
1	10	39.67±2.2228
2	20	63.98±3.4403
3	30	75.03±6.0178
4	40	94.50±1.3229
5	50	91.48±4.0051
6	60	90.63±5.6636

(n=3, all the data are in mean±SD)

Effect of NaOH treatment on water absorption capacity

In a previous study of starch acrylic acid SAH was synthesized using gamma radiation, wherein swelling behavior of hydrogel improved when treated with 0.45 M NaOH [23]. However, in this study, it is noted that the water absorption capacity reduced to a greater extent after NaOH treatment. The NaOH treated hydrogel could absorb 88.778±0.8864 g water, 40% less than SAH. This significant difference in water absorption may be ascribed to hydrolysis of SAH by alkali. The result was not showing resemblance with the preceding study.

Effect of pH on the water absorption

The water absorption studies performed at different pH on the purified SAH reveals (fig. 1) that highest water absorption and ultimately swelling of the hydrogel results at pH 9 (146.277±3.503 g per gram of SAH), followed at pH 7 (113.352±1.824 g of water per gram of SAH). Water absorption decreases gradually towards extremes of pH range-pH 1 (3.457±0.395 g water per gram of SAH) and pH 12 (34.059±0.276 g water per gram of SAH). Change in swelling capacity of hydrogel is attributed to protonation or ionization of functional group of hydrogel [33].

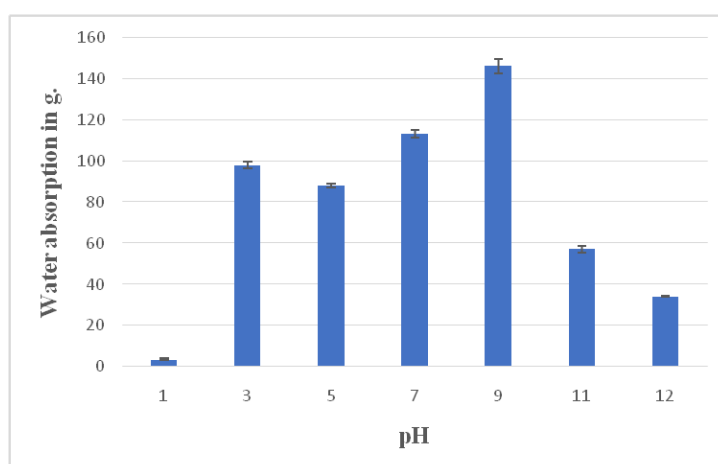


Fig. 1: Amount of water absorbed at different pH (n=3)

Hygroscopicity studies

Depending on the hygroscopicity, the substances are divided in four categories i.e., deliquescent, very hygroscopic, hygroscopic and slightly hygroscopic. Hygroscopicity studies performed on the unpurified, purified and NaOH treated SAH shows that the purification process reduces the hygroscopicity of SAH while NaOH treatment tremendously increases it. All three samples kept in ammonium sulphate saturated solution gives 6.5±0.251% weight gain in case of SAH, 10.5±0.851% weight gain in case of unpurified SAH while 23.42±0.884% weight gain in case of NaOH treated SAH. Increased surface area because of the expanded structure after

NaOH treatment may be responsible for the increase in hygroscopicity.

FTIR, PXRD and SEM analysis of hydrogel

In graft copolymerization of starch with acrylic acid, formation of ultra-high polymeric structure takes place. This contains multiple-COO-groups which reflects in the FTIR spectra (fig. 2) of all hydrogel materials viz. unpurified SAH, SAH and NaOH treated SAH giving peak at 2357/2358 cm⁻¹. There are significant reductions in the intensity of peak at 2922 in purified SAH and NaOH treated SAH as compared to unpurified SAH which might be due to removal of

unused acrylic acid in the purification process. FTIR spectra of starch gives strong absorption band at 3200–3300 cm^{-1} , a characteristic of OH bond also other bands obtained (2930: C-H stretching, 1650: carbonyl group, 1152 and 1018: -CO stretching) resembles the standard bands of starch [34, 35].

Diffractogram obtained from Powder X ray diffractometry (PXRD) studies of starch, SAH and NaOH treated SAH (fig. 3), shows that there are no sharp peaks in diffractogram of SAH while NaOH treated SAH shows a sharp peak at 2θ of 7.88° and doublet at about 2θ of 30° and 31° . This indicates that amorphous SAH on treatment with NaOH turns crystalline. Diffractogram of wheat starch gives strong diffraction peak at 2θ of 15.28° and 22.91° and a diffused

peak between 2θ of 17° - 18° which indicates crystalline structure of starch and is analogous to the diffractogram of normal starch [36].

Scanning electron microscopic (SEM) photographs of the prepared and purified SAH and NaOH treated SAH are depicted in fig. 4. It can be clearly seen that in NaOH treated SAH there is a significant change in the surface of the SAH particle. Surface of the purified SAH is smooth while surface of NaOH treated SAH is rough and porous. This indicates that NaOH reacts with the SAH and cause chemical changes that are liable to change in swelling properties and hygroscopicity. The altered and expanded structure may be due to cross linking induced repulsion between grafted chains with the larger electrostatic force between -COO- groups.

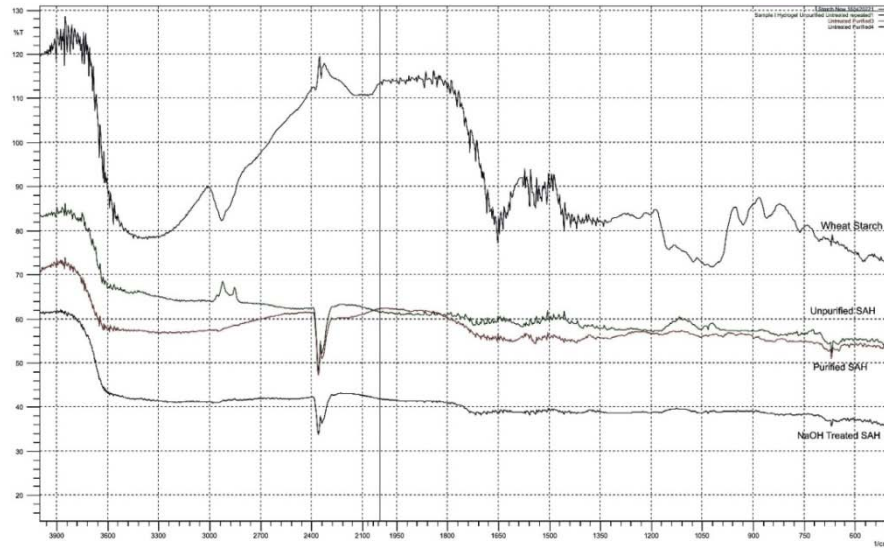


Fig. 2: FTIR spectrum of wheat starch, unpurified SAH, purified SAH and NaOH treated SAH

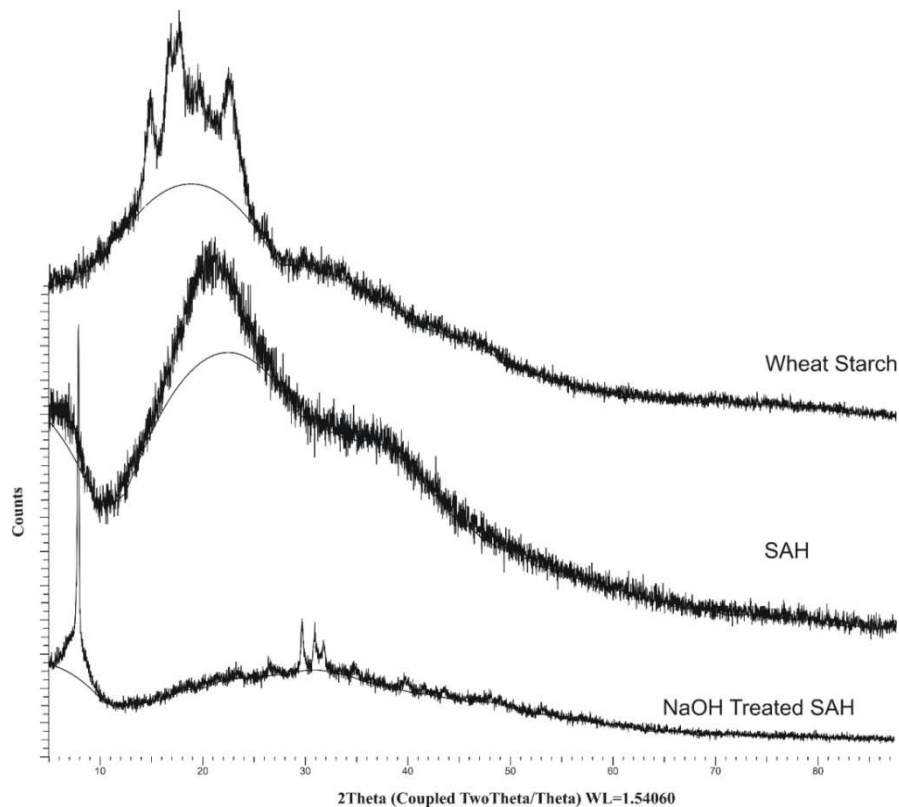


Fig. 3: Powder X ray diffractogram of wheat starch, purified SAH and NaOH treated SAH

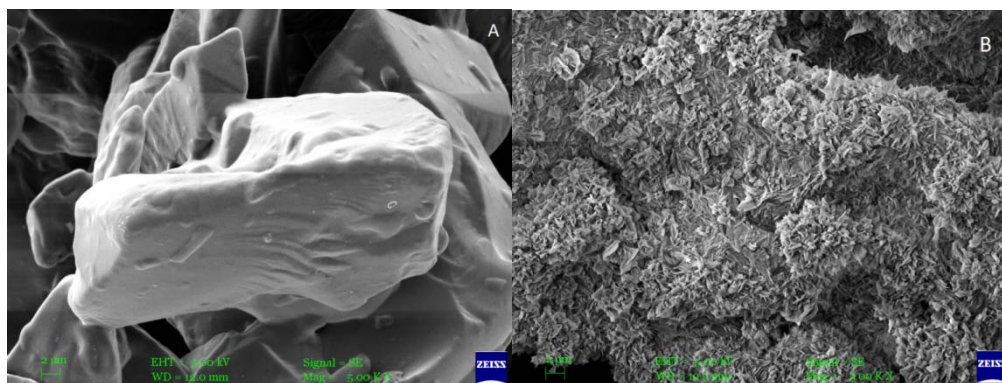


Fig. 4: SEM photographs of A. SAH and B. NaOH treated SAH

Differential scanning calorimetry and thermogravimetric analysis (DSC and TGA)

Thermograms (DSC and TGA) of SAH, NaOH treated SAH and starch is shown in fig. 5. DSC of SAH shows endothermic glass transition temperature at ~ 200 °C while that of NaOH treated SAH gives it at 80 °C. Decomposition exotherm was observed at ~ 275 °C for SAH and that for NaOH treated SAH at 225 °C. TGA studies of starch

shows 8.4 % reduction in weight between 60-110 °C due to dehydration. Starch usually degrades at ~ 300 °C which can be observed by 51% decrease in weight at 270-310 °C. TGA of SAH and NaOH treated SAH reveals good thermostability of both and shows only about 13% reduction in weight up to 200 °C. Both plots exhibit gradual decrease in weight as temperature increases. This indicates that hydrogel and starch are thermally stable to a sufficient range while NaOH treatment causes no effect on thermostability.

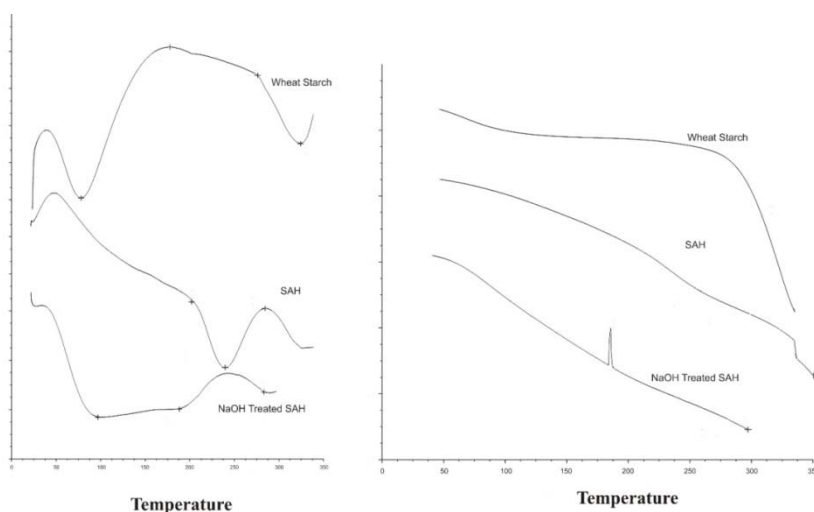


Fig. 5: DSC (Left) and TGA (Right) of wheat starch, SAH and NaOH treated SAH

Particle size analysis and Micromeritic characteristics

Polydispersity index (PDI) is used to measure heterogeneity in particle size means it gives width of particle size distribution. Range of PDI value ranges from 0 to 1. In case of agglomeration, aggregation or wide range of particle size, it increases while in case of homogeneous particles, it tends toward 0. As per ISO, for monodisperse systems PDI < 0.5 is normal while for polydisperse system <0.7 is acceptable [37, 38]. PDI of SAH and NaOH treated hydrogel was 0.179 ± 0.1344 and 0.2635 ± 0.0205 respectively while mean particle diameter was

1.4756 ± 0.4244 and 1.1047 ± 0.5017 (table 2). Thus, SAH show a narrow range of molecular weight distribution. On NaOH treatment though mean particle diameter decreases, PDI increases. This may be due to formation of small particles due to hydrolysis of the hydrogel. Angle of Repose, Hausner's ratio and Carr's compressibility index values of prepared hydrogel (respectively) show good flow properties while there is a decrease in flow properties on NaOH treatment (respectively). The flow properties of treated hydrogel lie in fair category. This may be due to surface erosion by the alkali causing rough texture that increases friction among particles.

Table 2: Particle size analysis and micromeritic characteristics of SAH and NaOH treated SAH

Parameter	SAH	NaOH treated SAH
PDI	0.179 ± 0.1344	0.2635 ± 0.0205
Mean Particle Diameter (μm)	1.4756 ± 0.4244	1.1047 ± 0.5017
Angle of Repose	33.50 ± 1.3541	36.26 ± 0.7814
Bulk Density	0.7229 ± 0.0037	0.6900 ± 0.0116
Tapped Density	0.9597 ± 0.0107	0.9958 ± 0.0331
Hausner's Ratio	1.3276 ± 0.0205	1.4434 ± 0.0472
Carr's Compressibility Index	24.6667 ± 1.1547	30.6667 ± 2.3094

(n=3, all the data are in mean \pm SD)

Kawakita plot

In Kawakita analysis, 'a' value is indicative of total reduction in the value of powder, hence indicates compactibility, while 'b' is indicative of cohesiveness and is inversely proportional to yield strength of particles. Increase in value of 'a' indicates good

compressibility while if 'b' value is greater than 'a' show good compressibility and less yield strength. Prepared hydrogel as well as NaOH treated hydrogel shows less cohesiveness as 'b' value is less than 'a' value. In both the samples value of 'a' is greater than 'b' and hence the samples show good compactibility (table 3).

Table 3: Parameters obtained from Kawakita plots of SAH and NaOH treated SAH

Parameter	Prepared hydrogel	NaOH treated hydrogel
'a' (Compactibility)	0.2735±0.0069	0.2503±0.0225
'b' (Cohesiveness)	0.208±0.0467	0.1715±0.0352
Slope 'm'	3.6572±0.0908	4.0159±0.3639
Coefficient of Correlation 'r'	0.9969±0.0016	0.9925±0.0025
'Y' Intercept	18.26±4.5457	23.77±2.7351

(n=3, all the data are in mean±SD)

Stability studies

Stability studies data reveals that the swelling properties of the unpurified SAH, SAH and NaOH treated SAH remain undisturbed even at higher temperature of 45 °C and at 75% RH. As per result, there is a slight decrease in the swelling properties per gram of sample that might

be due to increase in moisture concentration which proportionally reduces the hydrogel concentration. At both 65% RH and 75% RH, the hydrogels absorb moisture that shows its hygroscopic nature and the results are in accordance with hygroscopicity study. Results of this study revealed that, there is highest moisture absorption by NaOH treated SAH followed by unpurified SAH and least for SAH (table 4).

Table 4: Parameters showing observations and results of stability studies

Compound	Evaluation parameter	Initial observations	Storage condition for 3 mo	
			25 ±2 °C (65±5%RH)	40 ±2 °C (75±5%RH)
Unpurified SAH	Color	Yellowish	Yellowish	Yellowish
	Appearance	Free flowing	Damp Mass	Damp Mass
	Change (Gain) in weight	0	7.86±0.7302	11.9067±0.9729
	Swelling Capacity	111.265±2.5294	108.2892±3.5681	106.2509±2.8524
Purified SAH	Color	Gray	Gray	Gray
	Appearance	Free flowing	Damp Mass	Damp Mass
	Change (Gain) in weight	0	5.46±0.6636	8.6933±0.7821
	Swelling Capacity	120.002±3.3299	118.2561±4.5891	116.0583±4.2083
NaOH treated SAH	Color	Gray	Gray	Gray
	Appearance	Free flowing	Damp Mass	Damp Mass
	Change (Gain) in weight	0	16.66±1.6773	27.5733±2.6941
	Swelling Capacity	88.778±0.8864	84.6058±2.6820	83.5831±2.9524

(n=3, all the data are in mean±SD)

CONCLUSION

From the present study it can be concluded that superabsorbent hydrogel can be satisfactorily synthesized from wheat starch by graft copolymerization technique and by crosslinking with acrylic acid monomer. It shows good water absorption and retention capacity. SAH obtained shows excellent water absorption and retention capacity and maximum water absorption at pH 9. PXRD study reveals its amorphous nature which on NaOH treatment again turns crystalline. The SAH product is thermostable, shows good flow properties and less cohesive in nature. On treatment with NaOH, change in surface texture happens which gives a crystalline product with poor flow properties and a decrease in water absorption capacity. SAH had established its ability on storage at higher temperature and relative humidity. Consequently, SAH formed, can find application in and suitable for biomedical and environmental remediation, biodegradable foam, pollution control, sanitary napkins, medicated absorbable wound dressing and can be investigated for drug delivery also.

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Nil

AUTHORS CONTRIBUTIONS

All the authors have contributed equally.

CONFLICT OF INTERESTS

Declared none

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