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Superabsorbent derived from cassava waste pulp

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Abstract

Background: Cassava waste pulp from the tapioca industry is abundant in Indonesia. However, there have been few Indonesian or international papers describing research on the possible use of cassava waste pulp as a superabsorbent after modification. The goal of this work was to increase the added value of cassava waste pulp by converting it into a superabsorbent. This conversion was carried out by a graft copolymerization of cassava waste pulp using acrylamide, ammonium persulfate, and N,N'-methylene-bisacrylamide as a monomer, an initiator, and a crosslinker, respectively. The copolymerization was conducted at 70°C for 3 h and saponified with 1 M NaOH for 2 h.

Results: The superabsorbent had a maximum water absorption capacity of 1,014 g/g at pH 7.3; the absorbency was affected by the salinity of the medium. The rate parameter for absorption in distilled water was 5.4 min. The formation of a superabsorbent was confirmed by FTIR spectroscopy, as the spectra exhibited all characteristic bands of both cassava waste pulp and acrylamide.

Conclusion: Cassava waste pulp has a great potential to be used as a superabsorbent, which could give added value to cassava.

Keywords: Cassava waste pulp, Acrylamide, Copolymerization, Superabsorbent, Water absorption capacity

Introduction

Cassava is one of the most widely grown crops in Indonesia. The acreage devoted to cassava in 2011 was more than 1.2 million ha, with a productivity of 20.3 ton/ha and production close to 24 million tons (Badan Pusat Statistik (BPS) (2012)). After cassava root is processed into flour, the residual pulp is called cassava waste pulp (CWP). CWP constitutes approximately 11.4% of the fresh cassava, meaning that in 2011, the production of CWP was approximately 2.6 million tons. However, until recently, there have been few Indonesian or international papers describing research on the possible use of cassava waste pulp as a superabsorbent.

CWP has relatively high carbohydrate content. The starch content is approximately 50% to 60% w/w (Ubalua 2007) and 61.84% to 69.90% (Srinorakutara et al. 2006), while other constituents 10.61-14.35% is crude fiber (Srinorakutara et al. 2006). Due to the hydroxyl content of starch and cellulose, a great deal of work has been done to

modify those two constituents for other uses, one of which is as a superabsorbent. A superabsorbent polymer (SAP) has hydrophilic groups that are able to absorb and retain fluids and to release the fluids later under certain conditions (Zhang et al. 2006). A polymer is categorized as a superabsorbent if its ability to absorb water is more than 100 times its original weight (Zhang et al. 2007). Most SAPs available on the market have low biodegradability making them environmentally unfriendly in the long run. Therefore, extensive studies have been conducted to use natural base polymers, i.e., starch and cellulose, for biodegradable SAPs (Nakason et al. 2010).

In general, SAP is synthesized by grafting or grafting-crosslinking copolymerization. The monomers used in grafting copolymerization include acrylic acid and acrylamide (Li et al. 2007; Teli and Waghmare 2009), while N,N'-methylene-bisacrylamide (MBA), trimethyl propane triacrylate, and 1,4-butadienol dimethacrylate are used as crosslinkers (Swantomio et al. 2008). Commonly used initiators are persulfate salts, hydrogen peroxide (Moad and Solomon 2006), and cerium salts (Al et al. 2008).

Grafting copolymerization has been carried out to modify cassava starch into a SAP (Khalil and Farag 1998; Lanthong et al. 2006). In addition to starch, lignocellulose and its derivatives are also used as substrates for

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graft copolymerization. Mixtures of acrylamide (AAm) and acrylic acid (AA) monomers were grafted on germinated and gelatinized wheat starch using potassium persulfate as an initiator to yield a superabsorbent (Teli and Waghmare 2009). The SAP produced by the grafting-crosslinking process is expected to have high water absorption capacity and to release its absorbed water slowly.

The aims of this study were to synthesize a SAP by grafting-crosslinking CWP with acrylamide as a monomer, ammonium persulfate (APS) as an initiator, and MBA as a crosslinker. The effects of acrylamide, APS, and MBA concentrations were studied by characterizing the reaction products. The characterization included water absorption capacities, infrared spectra, and grafting efficiencies. Swelling kinetics in distilled water and swelling behavior at various NaCl concentrations as well as at various pH values were also investigated.

Methods

Materials

Cassava waste pulp was obtained from a cassava home factory at Bogor, Indonesia. Nitrogen gas (HP grade) was purchased from BOC gases vendor, Jakarta, Indonesia. Acrylamide, ammonium persulfate, methanol, ethanol (95%), acetone, KBr for IR analysis, and NaOH were all of analytical grade and manufactured by Merck (Darmstadt, Germany). MBA was manufactured by Fluka (Buchs, Switzerland).

Preparation and proximate analysis of raw material

Cassava waste pulp was washed, dried, and ground to 100-mesh size. Proximate analyses including moisture (gravimetric method), ash (gravimetric method), fat (Soxhlet extraction method), and protein (Kjeldahl method) contents in accordance with the standard methods (Association of

Official Analytical Chemist AOAC 2006), while carbohydrate contents were analyzed using the phenol-H₂SO₄ method (Dubois et al. 1956).

Grafting-crosslinking of acrylamide onto CWP

The grafting-crosslinking of acrylamide onto CWP was performed by following the method of Liang et al. (2009) with minor modification. CWP was weighed and placed in a three-neck flask that was equipped with a condenser, a flow of nitrogen gas, thermometer, and stirrer. Distilled water was added to the CWP to form a slurry. The slurry was heated to a temperature of 95°C while the flask was purged with nitrogen gas. The temperature was held at 95°C for 30 min and then it was reduced to 60°C to 65°C. APS was added and the mixture was stirred for 15 min. Known weights of acrylamide and MBA were then added to the flask, and the temperature was gradually increased to 70°C over 3 h.

The product was then precipitated with methanol and ethanol. The mixture was then refluxed with acetone for 1 h. The resulting solid was dried at 60°C until its weight was constant. The dried product was ground and sieved to a particle size of 80 to 100 mesh. The optimization of the grafting-crosslinking process was performed using a fractional factorial design with three factors and two levels. The factors were acrylamide concentration (ranging from 60% to 90%), ratio of the APS initiator concentration to monomer concentration (0.74% to 1.67% w/w), and ratio of the MBA crosslinker concentration to monomer concentration (0.08% to 0.13% w/w) (Table 1).

Saponification

Saponification was carried out according to the method of Nakason et al. (2010). Forty grams of graft copolymer was added to a mixture of 100 mL of 1 M NaOH and 100 mL of

Table 1 Fractional factorial design for SAP optimization

Number	Copolymer code	CWP (%)	Acrylamide (%)	MBA-monomer ratio (%)	APS-monomer ratio (%)
1	C1	40	60	0.08	1.67
2	C1	40	60	0.08	1.67
3	C2	40	60	0.19	1.11
4	C2	40	60	0.19	1.11
5	C3	25	75	0.11	1.11
6	C3	25	75	0.11	1.11
7	C3	25	75	0.11	1.11
8	C3	25	75	0.11	1.11
9	C4	10	90	0.06	0.74
10	C4	10	90	0.06	0.74
11	C5	10	90	0.13	1.11
12	C5	10	90	0.13	1.11

distilled water. The mixture was heated to 90°C. After 2 h the mixture was neutralized with 1M HCl, and then precipitated sequentially with methanol and ethanol. The saponified copolymer was dried at 60°C and was ground and sieved to 80 to 100 mesh.

Characterization

SAP characterization included determining the water absorption capacity, grafting efficiency, IR spectra, swelling kinetics, pH sensitivity, and salt sensitivity.

Water absorption capacity

Samples of the dry polymer (with and without saponification) weighing 0.1 g were soaked in 200 mL of water and allowed to stand for 24 h at room temperature to reach equilibrium. The swollen samples were then recovered with 100-mesh sieve. The water absorbency (Q_{eq}) was calculated by Equation 1 as follows:

$$Q_{eq} = \frac{(m_2 - m_1)}{m_1}, \quad (1)$$

where m_1 is the weight of dried SAP (g), and m_2 is the weight of swollen polymer (g).

Grafting efficiency

Grafting efficiency is calculated based on nitrogen content according to Equation 2:

$$\begin{aligned} \text{Grafting efficiency (GR, \%)} \\ = (N_{exp}/N_{th}) \times 100, \end{aligned} \quad (2)$$

where N_{exp} and N_{th} are experimental and theoretical percentages of nitrogen, respectively.

IR spectral analysis

The functional groups of CWP and unsaponified and saponified SAPs were examined using Prestige 41 Shimadzu Fourier transform infrared FTIR spectroscopy (Shimadzu Co. Ltd., Beijing, China) with a KBr pellet. IR solution 1.20 Interface software was used in the connected data acquisition system (Shimadzu Co. Ltd).

Swelling kinetics

To study the absorbency rate of the SAP, 1.0 ± 0.01 g samples with an average particle size of approximately 100 mesh were poured into weighed tea bags and immersed in 800 mL of distilled water. At consecutive time intervals, the water absorbency of the SAP was then measured according to the aforementioned method.

pH sensitivity

The swelling behavior at different pH values each was determined after 24-h observation. The solutions were

adjusted to the desired pH values (ranging from pH 1 to 14) by the addition of diluted HCl or NaOH.

Saline sensitivity

The swelling capacity of the SAP was measured at different concentrations of NaCl ranging from 0.01 to 1 M.

Results and discussion

Proximate analysis of CWP

The proximate analysis results for the CWP sample are as follows: moisture content (11.30%), ash (0.55%), protein (4.56%), fat (0.21%), and carbohydrate (82.70%). The results indicate that carbohydrate is the main component while protein, fat, and minerals are comparatively insignificant. This insignificance means that treatment to remove protein, fat, and minerals is not required.

Grafting-crosslinking of acrylamide into CWP

CWP is the backbone in the grafting-crosslinking copolymerization, with acrylamide as the monomer, APS as the initiator, and MBA as the crosslinker. Copolymerization was carried out under inert conditions with oxygen removed from the reaction system by a nitrogen gas purge. The purposes of the inert conditions were to minimize peroxide radical formation that could inhibit the copolymerization and to suppress homopolymer formation. The copolymerization temperature was 70°C, as suggested by Lanthong et al. (2006), Li et al. (2007), and Hua and Wang (2009).

The copolymer was precipitated with methanol and then with ethanol to pull water from the SAP. The SAP was then refluxed with acetone to remove homopolymers. The water absorption capacity of saponified SAP was approximately 1,000 times its initial weight; therefore, it was categorized as a superabsorbent. In contrast, the unsaponified SAP had a lower Q_{eq} value than that of the saponified SAP (Figure 1), which is consistent with data obtained by Li et al. (2007) and Nakason et al. (2010) (Table 2).

An increase in the Q_{eq} value of saponified SAP is closely related to the degree of conversion of amides ($-\text{CONH}_2$) into carboxylic groups ($-\text{COOH}$) (Teli and Waghmare 2009), which is in line with the decrease in nitrogen content, as shown by Figure 2. The grafting efficiency increased with decreasing percentage of monomer used in the reaction (Figure 3). This phenomenon may have been caused by the higher availability of monomers in the vicinity of CWP carbohydrate molecules as well as a higher monomer concentration in the polymerization medium. The greater availability of the monomer in the polymerization medium acted in favor of molecular collision (Sikdar et al. 2003; Hebeish et al. 1981), thereby enhancing polymerization in general or homopolymer formation in particular. In addition, the

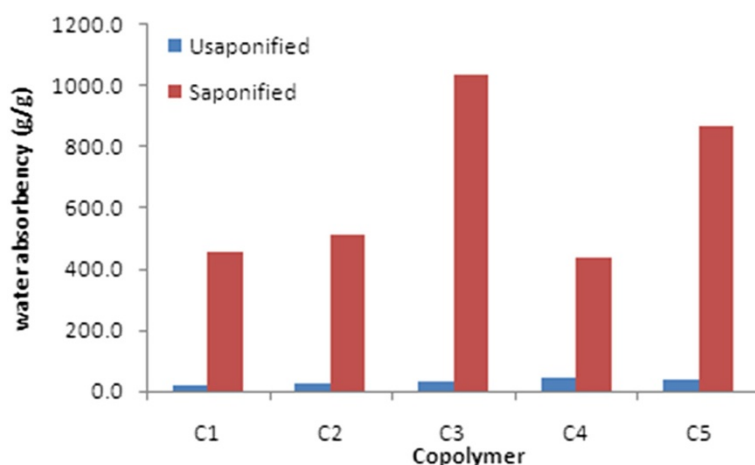


Figure 1 Saponification effect on water absorption capacity of SAP.

increase in grafting efficiency does not affect water absorption because grafting does not influence the spatial structure of the hydrophilic copolymer.

The optimum conditions for graft-crosslink copolymerization were obtained at the composition of 75% acrylamide, 0.11% MBA, and 1.11% APS with Q_{eq} 1,040.1 g/g. The p value of the curved quadratic model obtained by an analysis of variance (ANOVA) (<0.05) strengthens the above optimization. Visualization of this model is shown in Figure 4. In addition, the ANOVA data show that all three factors significantly affect the Q_{eq} of SAP at the 90% confidence level.

IR spectral characterization

Figure 5 shows the representative FTIR spectra for CWP, unsaponified SAP, and the saponified product. The broad absorption bands at 3,550 to 3,200 cm^{-1} , which represent the O-H stretching vibration, and the medium absorption peak at 2,923 cm^{-1} , which represents of the C-H stretching vibration, are clearly seen. The triplet peaks with a strong absorption at 1,149, 1,080, and 1,026 cm^{-1} indicate the presence of the C-O-C stretching vibration. The IR spectrum of unsaponified SAP exhibits all the absorption bands of CWP, as well as absorption peaks at

3,201, 1,662, and 1,616 cm^{-1} , which are indicative of N-H stretching, C=O stretching, and N-H bending of the amide groups, respectively. Those are the band characteristics of $-CONH_2$ groups of acrylamide. Furthermore, the peak at 1,419 cm^{-1} represents $-C-N$ stretching, and the peak at 766 to 710 cm^{-1} represents the weak band of N-H engaged in out-of-plane bending, all of which are also characteristic bands for amide grafted onto CWP.

After the saponification, the following absorption peaks were observed that confirm the occurrence of PAM: the N-H stretching of the N-H stretching of $O=C-NH$ (amide band); the C=O stretching; the N-H bending at 1,419 cm^{-1} ; for the $-C-N$ stretching, a relatively intense and sharp peak at 2,376 cm^{-1} which is the $-C\equiv N$ (nitrile or cyanyl) peak; and an intermediate peak from the saponification in alkaline solution of the amide group converted to the nitrile or cyanyl group (Nakason et al. 2010). Under saponification conditions, the amide groups were converted to carboxylate groups. In the saponified SAP, the very intense characteristic band at 1,562 cm^{-1} is due to C=O asymmetric stretching in the carboxylate anion.

Swelling kinetics

Figure 6 represents the swelling capacity of saponified and unsaponified SAPs in distilled water at different time intervals. The rate of water uptake is initially very high, and then it begins to level off. Equilibrium swelling was achieved after approximately 60 min. A power law behavior is indicated by Figure 6, and the specific data are well fitted with a Voight-based equation (Equation 3) as follows:

$$S_t = S_e \left(1 - e^{-\frac{t}{\tau}}\right), \quad (3)$$

where S_t (g/g) is swelling at time t , S_e is equilibrium swelling (g/g), t is time (minute) for swelling and τ

Table 2 Maximum water absorbency of three different saponified SAPs

Composition of SAP	Maximum water absorbency (g/g)
Results of this study; 25% CWP/75% acrylamide	1,040.1
Li et al. (2007); 20% starch/80% acrylic acid	1,077
Nakason et al. (2010); 50% starch/50% acrylamide	606

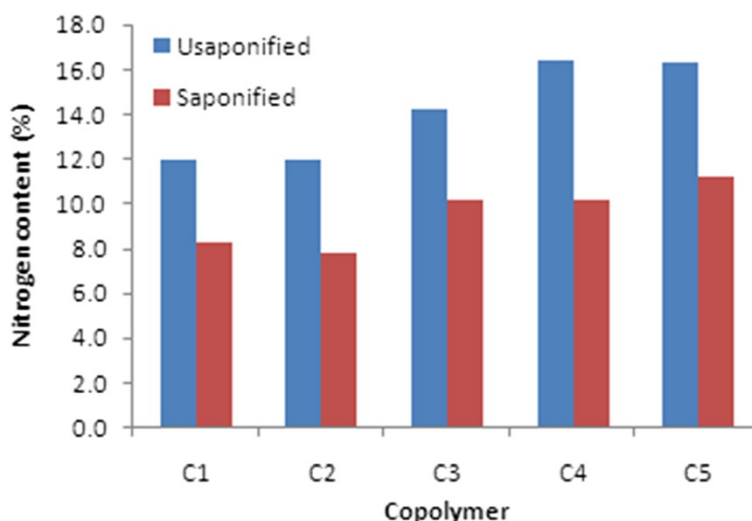


Figure 2 Saponification effect on nitrogen content of unsaponified and saponified SAPs.

(minute) stands for the 'rate parameter.' The swelling pattern of the SAP was similar to that obtained by Pourjavadi et al. (2010). The swelling kinetic equations for the unsaponified (Equation 4) and saponified (Equation 5) SAPs were obtained using Curve Expert 1.4 software (www.curveexpert.net).

$$S_t = 23.30(1 - e^{-0.9136t}); r^2 = 0.963, \quad (4)$$

$$S_t = 1,2841(1 - e^{-0.1845t}), r^2 = 0.973 \quad (5)$$

According to Equations 4 and 5, the rate parameters (τ) for swelling in water were found to be 1.09 and 5.4 min for unsaponified and saponified SAPs, respectively. Pourjavadi and Mahdavinia (2006) obtained the rate parameters (τ) of 2.5 and 1.3 min, respectively, for unsaponified and saponified chitosan-g-PAAm with particle size of 60 to 80 mesh, and Pourjavadi et al. (2010)

reported a τ value of 7.5 min for the saponified (starch-carrageenan)-g-PAN with particle size of 60 to 80 mesh.

pH sensitivity

The pH sensitivity of saponified SAP was studied by observing the equilibrium swelling (ultimate water absorption) at various pH values ranging from 1.0 to 14.0. The pH of the medium was adjusted with diluted NaOH or HCl. No buffer solution was used because the buffer ions could potentially reduce the water absorption capacity of the SAP. As shown in Figure 7, the swelling of the SAP increased as the pH increased from 1 to 7, but it decreased over the pH range of 8 to 12. The effect of pH on swelling follows a Gaussian equation:

$$Q = 1.64 \times 10^3 \exp(-(pH - 7.27)^2 / 8.37), \quad r = 0.945. \quad (6)$$

From this equation, the maximum water absorbency of the SAP was obtained at pH 7.3.

In acidic media, most carboxylate groups are protonated. This causes a decrease in the repulsion of anionic groups, which leads to a decrease in swelling. In addition, carboxylate ion repulsion is screened by H^+ ions, which does not allow the polymer chain to expand and results in a decreased Q_{max} . At pH values between 7 and 9, most carboxylic acid groups are in the ionized form ($-COO^-$), and the electrostatic repulsive force between the charged sites (COO^-) causes an increase in swelling. However, the presence of excess counter ions (Na^+) at pH values of 9 to 13 results in a 'charge screening effect' that shields the carboxylate anions, preventing effective anion-anion repulsion (Jenkins and Hudson 2001) and resulting in a decreased swelling of the SAP.

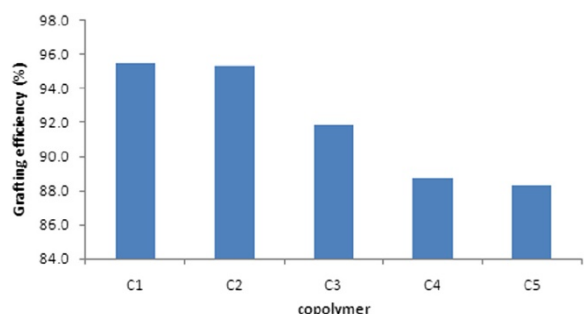


Figure 3 Grafting efficiency of SAP.

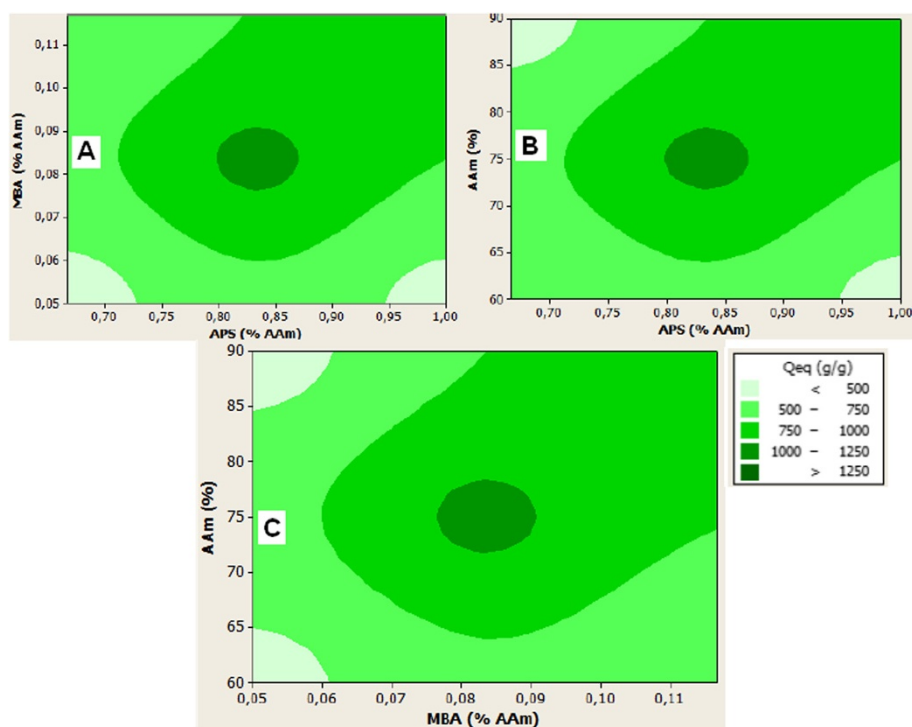


Figure 4 Contour of water absorption capacity of grafted-crosslinked SAP. Contour of water absorption capacity of grafted-crosslinked SAP as functions of (A) MBA and APS, (B) AAm and APS, and (C) AAm and MBA.

In addition, at both high and low pH values, ion concentrations are high, leading to an increase in ionic strength. When the ionic strength of the solution is increased, the difference in osmotic pressure between the SAP and the medium is decreased. Thus, at both low and high pH values, the swelling capacity of the SAP decreased. This type of behavior can also be explained on the basis of osmotic swelling pressure (π_{ion}) (Horkay et al. 2000). For a weakly charged SAP chain, the osmotic swelling pressure is given by Equation 7:

$$\pi_{ion} = RT\Sigma(C_i^g - C_i^s), \quad (7)$$

where C_i^g and C_i^s are the molar concentrations of mobile ions in the swollen gel and external solution, respectively, R is the gas constant, and T is the absolute temperature.

The saponified SAP contains carboxylate groups (COO^-) along the polymer chain. In a neutral medium, the concentration of mobile ions in the external solution (C_i^s) is almost negligible; therefore, π_{ion} becomes very large, leading to a larger Q_{max} . By contrast, in an acidic medium, π_{ion} becomes very small because the carboxylate ions within the swollen gels are protonated, resulting in a low value of C_i^s . On the other hand, in an alkaline solution, dissociation of -COOH groups is almost

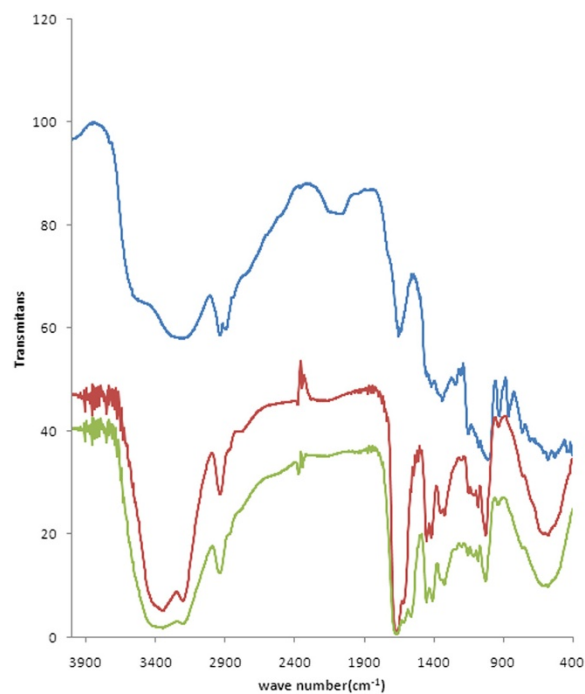


Figure 5 IR spectra of ongkok, unsaponified, and saponified SAPs. Onggok (blue line), unsaponified SAP (red line), and saponified SAP (green line).

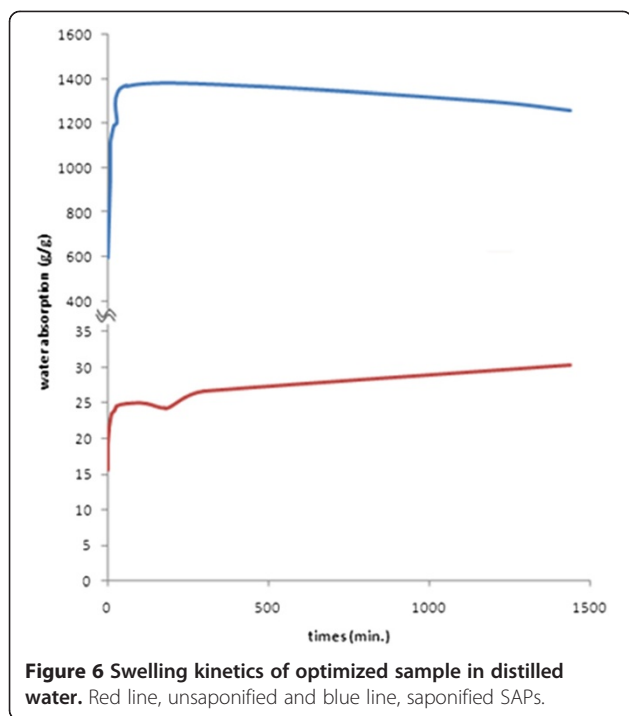


Figure 6 Swelling kinetics of optimized sample in distilled water. Red line, unsaponified and blue line, saponified SAPs.

complete, but very high concentrations of Na^+ and OH^- ions lead to a reduction in π_{ion} and Q_{max} .

Saline sensitivity

The variation in the swelling capacity of SAP in different concentrations of NaCl was investigated (Figure 8). The swelling capacity of ionic SAP in salt solutions was significantly reduced compared to the absorption capacity in distilled water. This known phenomenon is often attributed to a decreased osmotic pressure difference between the SAP network and the external solution due to a low efficiency anion-anion electrostatic repulsion. The ions attached to the polymer network are immobile and

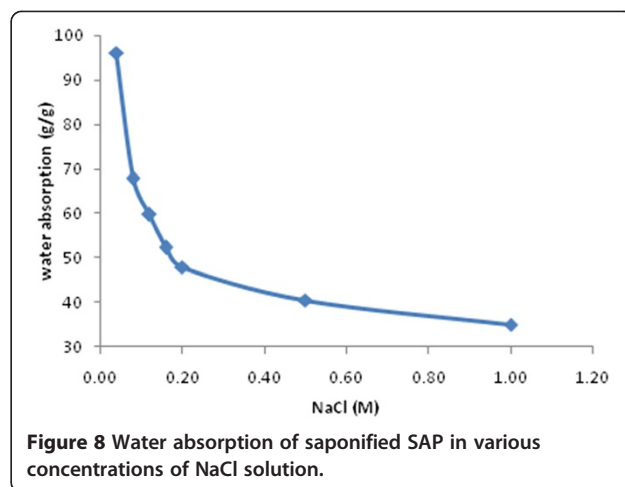


Figure 8 Water absorption of saponified SAP in various concentrations of NaCl solution.

considered to be separated from the external solution by a semi-permeable membrane. When a SAP is placed in water, the maximum osmotic pressure is developed, and the maximum swelling is achieved. When the polymer is in a salt solution (for example, NaCl), the development of osmotic pressure is much lower because the external solution contains Na^+ and Cl^- . Therefore, the swelling is drastically reduced. There is a familiar relationship between the swelling and concentration of salt solution that is stated in Equation 8 (Flory 1953):

$$\text{Swelling} = k[\text{salt}]^{(-n)}, \tag{8}$$

where k and n are constants for an individual superabsorbent. The k value indicates the absorption capacity of SAP at a high salt concentration, and the value of n is a measure of the dependence of swelling on salt concentration. The relationship between the swelling and salt solution concentration for the saponified SAP is shown by Equation 9:

$$\text{Swelling (g/g)} = 29.06 [\text{NaCl}]^{-0.35} \tag{9}$$

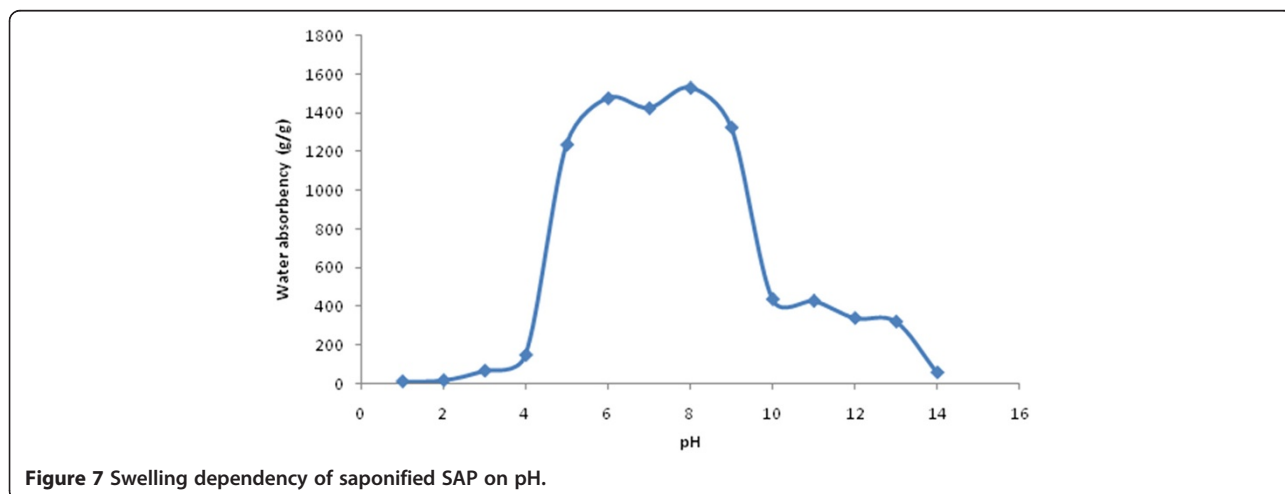


Figure 7 Swelling dependency of saponified SAP on pH.

According to this equation, the k and n constants for SAP are 29.06 and 0.35, respectively. This means that a NaCl concentration greater than 0.35 M has no appreciable effect on water absorption by the superabsorbent. This finding is in line with those obtained by Sadeghi and Koutchakzadeh (2007) for CMC-poly(NaAA-co-AAm), i.e., 8.3 and 0.38, respectively. This indicates that the SAP obtained is intolerant to saline solution and probably is not suitable for soil conditioner in saline soils. However, the SAP will work well on low salinity soil. In addition, other SAP applications are as biomedical bandages, waste solidification, diapers, etc.

Conclusions

Superabsorbent derived from cassava waste pulp has a water absorption capacity of 1,040.1 g/g, or approximately 1,000 times its dry weight. The study of swelling kinetics produced a rate parameter (τ) for the superabsorbent in distilled water of 5.4 min. The maximum water absorption capacity of the superabsorbent was achieved at pH 7.3. The water absorbency of SAP was strongly affected by the salinity of the medium and decreased as the salinity increased. The results showed that cassava waste pulp has a great potential to be used as a superabsorbent, which could increase the value of cassava.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

ZAA designed the overall study and was responsible for the synthesis strategy. MK was responsible for the technical aspect of the synthesis and characterization of the raw materials. MF conducted the FTIR study and helped draft the manuscript. MA characterized the swelling behavior of the superabsorbent. AN conceived the study, participated in its design, interpretation, and coordination, and helped draft the manuscript. All authors read and approved the final manuscript.

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