Microwave-assisted polymerization: Inert addition and surface coating of superabsorbent polymer for improved physical properties

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ABSTRACT: Two different polymerization techniques, microwave-assisted polymerization and free radical solution polymerization, were utilized in the syntheses of superabsorbent polymers with varying amounts of acrylic acid (31–50%). Degrees of neutralization were in the range of 68–80 mol %, and clay level was varied between 0 and 5%. The base polymer produced with microwave-assisted polymerization had higher absorbency under low load (0.3 psi) than those with the free radical solution polymerization. To improve its absorbency under higher loads (0.6 and 0.9 psi), the surface coating step was implemented by using ethylene glycol diglycidyl ether as a surface crosslinking agent. Properties such as capacity, permeability, and absorbency under different loads were tested in 0.9% sodium chloride solution for the base and the surface-coated polymers. In addition, extractables and residual acrylic acid were measured to determine the reaction's efficiency. In conclusion, surface coating improved polymer properties, and the incorporation of clay imparted permeability to the polymer. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43990.

INTRODUCTION

Superabsorbent polymer, which absorbs and retains large quantity of liquid under moderate to high pressure,[1] is most commonly produced with lightly crosslinked and partially neutralized acrylate or methacrylate monomers.[2] Any monomer with a polymerizable double bond[2] could be used in the making of superabsorbent polymer. Acrylic acid[2] has gained popularity because of its affordability and in combination with free radical solution polymerization is the preferred process in industrial settings.

Compared with a surface-coated superabsorbent polymer, a lightly crosslinked superabsorbent *base* polymer has higher centrifuge retention capacity (CRC) but lacks measureable gel strength. Centrifuge retention capacity is based on the amount of aqueous liquid that a polymer can absorb and bind chemically. It is influenced by the amount of crosslinker in the monomer solution which directly affect capacity. Furthermore, since polyacrylic acid is a water-soluble polymer, a crosslinker is needed to make it water insoluble. Superabsorbent polymer absorbs hundreds of times of its own weight in deionized water,[2,3] its capacity is in the range of 30–50 g/g for 0.9% sodium chloride (used to simulate urine in laboratories).[2] The first generation of superabsorbent polymer in the hygiene industries were *base* polymers. While this was an upgrade to the existing diaper, lower gel strength prevented the utilization of its total capacity due to gel blocking, a phenomenon which prevents liquid movement in the diaper core due to the constriction and eventual blocking of liquid transport channels as the gel swells. Since superabsorbent polymer has found application in different industries,[2,4–31] higher absorbency under load (AUL)[2] is necessary to withstand the applied pressure in these types of applications. AUL improvement is accomplished with a surface crosslinking step that crosslinks the exterior of the superabsorbent polymer particles/granules.

In general, "surface crosslinking," as it is referred to, accounts for a fair portion of the total cost of superabsorbent polymer production, due to the long and complicated surface coating step.[3] This step has been used to improve polymers' absorbency under load,[2] among other properties. It strengthens the *base* polymer by creating a shell around the core, but the trade-off is its decreased absorption capacity.[2,3] Introduction of a photo-induced surface crosslinking agent by S. Jackusch *et al.*[3] is a step in the right direction, as it improves polymer's permeability and shortens the time it takes to perform the surface crosslinking step. However, the presented data is only for the polymers with lower CRC (CRC < 30 g/g), and the combined properties (CRC, AUL, and Permeability) of these
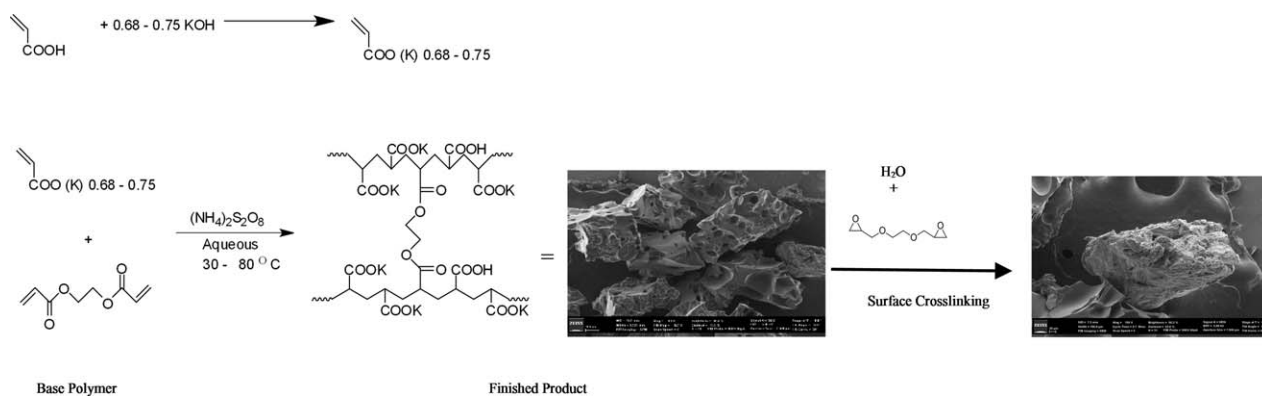
Scheme 1. General polymerization of acrylic acid (microwave-assisted or free radical solution polymerization) and surface coating of the polymer.

polymers are still lower than the one produced with a thermally reactive coating.[3]

Base polymer produced with free radical solution polymerization has lower AUL (AUL < 10 g/g) and higher CRC than that produced via microwave-assisted polymerization, and a long surface crosslinking step has to be implemented to strengthen the polymer. An alternative approach for improving the properties could be done by employing a *base* polymer with higher AUL, and therefore shortening the time that it takes to do the surface crosslinking step. Microwave-assisted polymerization is capable of producing such base polymers (AUL > 14 g/g). Giachi *et al.*[32] described the advantage of microwave-assisted polymerization in producing a homogeneous polymer with far superior properties which could play a central role in optimizing the production processes. In addition, the time it takes to do the heating is substantially less.

Since AUL is an indicator of the amount of liquid that a polymer can absorb (under specific amounts of pressure) and CRC determines its retention capacity, permeability of a swollen bed of superabsorbent polymer translates over to a polymer's ability to distribute the absorbed liquid in a diaper core. Therefore, the liquid distribution in a diaper core is as critical as is its CRC or AUL.[3] For a polymer with higher CRC (CRC > 30 g/g), the surface coating process improves its absorbency under load but has very little effect on the permeability of a swollen gel bed. Polymer without permeability of its gel bed

cannot wick the liquid away from the point of "insult"[2] in a hygiene article (e.g., diaper) and therefore underutilizes the full potential[2] of the superabsorbent polymer in the intended application. Addition of clay to the polymer is one way of improving the permeability. In free radical solution polymerization, clay addition to the monomer is riddled with pitfalls. Since the initiation process is relatively slow, clay has a tendency to separate from the monomer solution and sinks to the bottom. To overcome this problem, it could be added to the extruded gel, but again, the heterogeneity of the clay inherent to this application method becomes an issue. In microwave-assisted polymerization, due to the fast nature of the initiation step, dispersion of the clay in the monomer solution is uniform and once again avails the possibility for the use of clay in the polymerization step.

In the experiments discussed in this manuscript, microwave-assisted polymerization was employed to produce a *base* polymer with higher gel strength for higher CRC-polymer, and the results are compared with those produced with free radical solution polymerization. Acid content, degree of neutralization (DN), and percentage of clay were varied to produce these polymers. Ethylene glycol diglycidyl ether (EGDGE) was used as a surface crosslinking agent, and the surface crosslinking times were measured and compared for polymers with high and low gel strengths. Properties such as Pressure Absorbency Index (PAI), the sum of four AULs at 0.01 psi (0.7 g/cm^2), 0.3 psi (21 g/cm^2), 0.6 psi (42 g/cm^2), and 0.9 psi (63 g/cm^2), were used to monitor the efficiency of the crosslinking step. Energy-dispersive X-ray spectroscopy (EDX) was used to confirm the presence of the clay, and permeability was measured to monitor its uniform distribution.

EXPERIMENTAL

Materials

Purchased chemicals: Glacial acrylic acid from BASF, potassium hydroxide, sodium chloride, hydrogen peroxide (H_2O_2), ascorbic acid ($C_6H_8O_6$), hydrochloric acid, 85% O-phosphoric acid, high performance liquid chromatography (HPLC)-grade methanol, ultrapure water, ethylene glycol diglycidyl ether, and ammonium persulfate [$(NH_4)_2S_2O_4$] from Aldrich, Laptonite clay (Synthetic Hectorite-like clay, diameter = 25 nm and thickness = 1 nm) from Southern clay, and ethoxylated
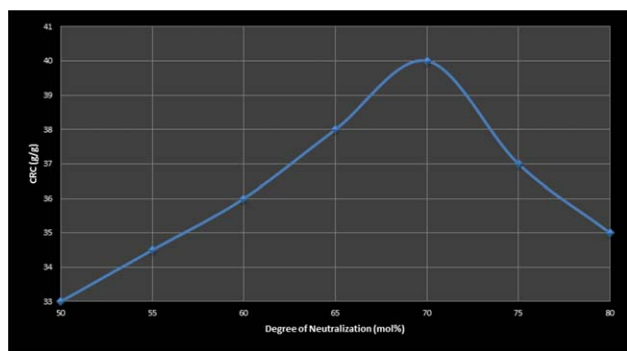

Figure 1. Degree of neutralization vs. capacity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
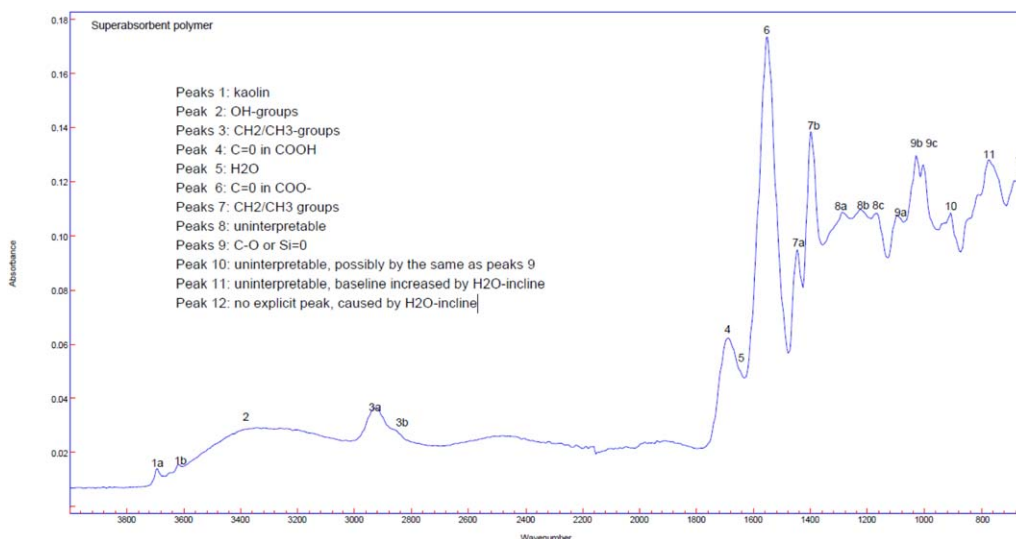
Figure 2. FTIR spectra (surface-coated polymer). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

trimethylolpropane triacrylate (ETMPTA) from Sartomer. All of the chemicals were used as received.

Equipment

The following equipment were used for polymerization and characterization: an HPLC instrument from Water with UV detector, a Nucleosil column (C8, 120 A°, 5 mm, 250 × 4.6 mm, with a mobile phase of 0.2 mL 85% O-phosphoric acid, 5.0 mL of HPLC-grade methanol, and 0.9948 L of ultrapure water) for residual acrylic acid analysis, a Retsch ZM1000 for milling, a RO-TAP model RX-29 equipped with USA Standard Test Sieve for sieving, a Heraeus Instrument Labofuge 400 for centrifuge retention capacity (CRC), a Thermoscientific Lindberg Blue M lab oven for the drying of the polymer, a Brinkman 816 titration system for extractables, and a Microwave from CEM for polymerization.

Monomer Solution

Monomer solutions were prepared as reported in our previous work.[33]

Microwave-assisted Polymerization

Polymerizations were performed as reported in our previous work.[33]

Free-radical Solution Polymerization

Polymerizations were performed as reported in our previous work.[33]

Surface Crosslinking Procedure

Weigh out 100 g of superabsorbent base polymer and spray it with 3.3 g of 0.1% EDGE solution in a Kitchen Aid bowl mixer while the mixer is on medium speed. Take the polymer out of the bowl and dry it at 155 °C for 20 to 45 min. Sieve it to 106–850 μ.

Water Content (WC) Measurements

The water content of the superabsorbent polymer particles is measured by the European Disposables and Nonwovens Association (EDANA) recommended test method No. 430.2-02 "Moisture content."

Table I. Base Polymers: Measured Properties at Different Acrylic Acid Content (Free Radical Solution Polymerization)

	Sample	CRC (g/g)	0.3 AUL (g/g)	RAA (ppm)	1 hr extractables (%)
1	31% AA-1	37.2	8.1	1251	12.1
2	31% AA-2	39.1	8.4	1232	11.9
3	31% AA-3	41.3	7.9	1165	12.9
4	35% AA-1	39.5	8.4	1181	13.5
5	35% AA-2	41.2	7.7	1111	14.1
6	35% AA-3	41.9	7.5	1089	14.8
7	50% AA-1	42.1	7.9	985	19.9
8	50% AA-2	43.2	7.2	812	18.9
9	50% AA-3	44.4	7.4	865	17.5

AA-1: $H_2O_2 = 150$ ppm, $C_6H_8O_6 = 150$ ppm, $(NH_4)_2S_2O_8 = 75$ ppm.
AA-2: $H_2O_2 = 175$ ppm, $C_6H_8O_6 = 175$ ppm, $(NH_4)_2S_2O_8 = 100$ ppm.
AA-3: $H_2O_2 = 225$ ppm, $C_6H_8O_6 = 225$ ppm, $(NH_4)_2S_2O_8 = 125$ ppm.
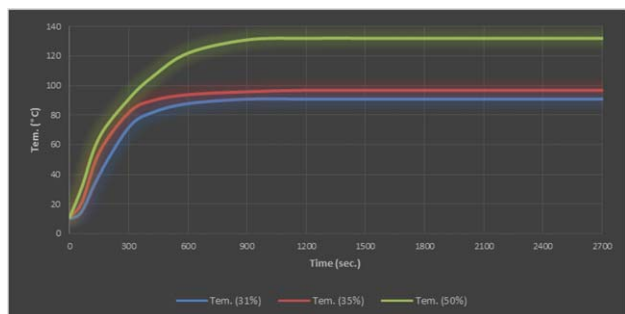AA, acrylic acid.

Figure 3. Temperature profile (free radical solution polymerization). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Centrifuge Retention Capacity Measurements

The centrifuge retention capacity of the superabsorbent polymer particles is measured by the EDANA recommended test method No. 441.2-02 "Centrifuge Retention Capacity."

Absorbency under Load Measurements

The absorbency under load of the superabsorbent polymer particles is measured by the EDANA recommended test method No. 442.2-02 "Absorption Under Pressure," using a weight of 0.7 psi (49 g/cm^2) instead of a weight of 0.3 psi (21 g/cm^2), 0.9 psi (63 g/cm^2), 0.6 psi (42 g/cm^2), and 0.01 psi (0.7 g/cm^2).

Extractables Measurements

The percent extractables of the superabsorbent polymer particles is measured by the EDANA recommended test method No. 470.2-02 "Extractable."

Residual Acrylic Acid (RAA) Measurements

The residual monomers content in the superabsorbent polymer particles is measured according to EDANA recommended test method No. 410.2-02 "Residual Monomers."

The EDANA test methods are obtainable from the European Disposables and Nonwovens Association, Avenue Eugène Plasky 157, B-1030 Brussels, Belgium.

Permeability Index (PI) [using Gel Bed Permeability (GBP) Test Measurements]

The method for Free Swell Gel Bed Permeability is described in US patent application no. US 2005/0256757 A1, paragraphs 61 through 75.

RESULTS AND DISCUSSION

The monomer solutions were prepared with different concentrations of acrylic acid (31–50%) with both free radical solution polymerization and microwave-assisted polymerization. One of the major problems with free radical solution polymerization is its low acid content, which requires lots of energy and money to get rid of water in the drying stage.[34] Formulation with acid content greater than 35% is impractical and polymerization is uncontrollable. On the contrary, in microwave-assisted polymerization, since the purpose of microwave heating is to heat the monomer solution and not its surroundings, increased acid content did not affect the polymerization step. Acid content of 50% was picked as an extreme case to differentiate microwave-assisted polymerization from free radical solution polymerization.

Potassium hydroxide was used as a neutralization agent to overcome the solubility issue of sodium hydroxide at higher acid content and the degree of neutralization was evaluated at 60–80 mol %. Neutralization of acrylic acid is a way of imparting charge density to the polymer's backbone.[2] Polymers became stickier to the touch as the degree of neutralization moved toward 60 mol %. This could be attributed to the acidic nature of the polymer, which affects both processing of the polymer and its properties in a negative way. The properties were also impacted negatively for the polymers with higher degrees of neutralization (80 mol %), but these were processable (being less sticky). In both microwave-assisted polymerization and free radical solution polymerization, the optimum DN was 70 mol % which gave the highest CRC. According to counter-ion condensation theory, as the degree of neutralization increases, the free ions will increase on the polymer backbone, but those free ions, which contribute to the osmotic pressure, will not increase after a certain value,

Table II. Base Polymers: Measured Properties at Different Acrylic Acid Content (Microwave-assisted Polymerization)

	Sample	CRC (g/g)	0.3 AUL (g/g)	RAA (ppm)	1 hr extractables (%)
1	31% AA-1	38.9	14.4	1050	2.6
2	31% AA-2	38.1	14.6	922	2.5
3	31% AA-3	37.5	15.1	855	2.4
4	35% AA-1	38.4	15.4	985	3.1
5	35% AA-2	37.9	15.2	844	2.4
6	35% AA-3	36.7	15.9	790	2.6
7	50% AA-1	38.1	15.1	899	2.1
8	50% AA-2	37.8	15.8	755	1.8
9	50% AA-3	37.4	15.7	712	2.1

AA-1: $(NH_4)_2S_2O_8$ = 5 ppm.
AA-2: $(NH_4)_2S_2O_8$ = 8.5 ppm.
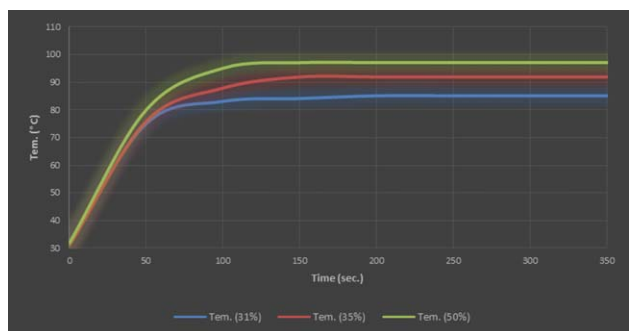AA-3: $(NH_4)_2S_2O_8$ = 10 ppm.
AA, acrylic acid.

Figure 4. Temperature Profile (microwave-assisted polymerization). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and therefore, its CRC will not be as high as one might expect through linear extrapolation (see Figure 1).

CRC of the *base* polymer is determined by the amount of crosslinker in the polymerization step. Any crosslinker with two or more double bonds,[2] which are capable of polymerization, could be used as a core crosslinker and ETMPTA was picked for this study.

In the free radical solution polymerization, *base* polymer was produced by utilizing ascorbic acid and hydrogen peroxide as a redox couple, while ammonium persulfate was used as a thermal initiator.[2] Both types of initiation packages are essential in this type of polymerization; redox coupling promotes polymerization and the thermal initiator is used to reduce residual acrylic acid in the final product. In the microwave-assisted polymerization, only 5–10 ppm of thermal initiator (ammonium persulfate) was needed to do the polymerization. Polymer with no initiator was hard to process (sticky to the touch), but as the amount of initiator increased (>10 ppm), the polymerization became unstable and the reproducibility from one run to the next was challenging. The same was true for the combination of redox coupling and thermal initiator use.

Since *base* polymers are prone to gel blocking (liquid movement is impeded), the surface coating step is used to improve its gel strength. EGDGE forms an ester bond (see Figure 2) in the surface of superabsorbent polymer and strengthens the base polymer[3] by creating a hard shell around its softer core. In addition, the surface coating step provided another opportunity to modify the polymer's properties.[2] Scheme 1 depicts the process of making and then surface coating the superabsorbent polymer.

Base polymers produced by free radical solution polymerization (with acid contents of 31–50%) had CRCs in the range of 38–44 g/g, with low 0.3 AUL (~8 g/g), and relatively high extractables (see Table I). The polymerization at 50% acid content was impossible to control and only a small amount of polymer was collected for characterization purposes. A closer look at the temperature profile (see Figure 3) shows that temperature increased at a drastic rate and rose beyond 100 °C in less than 5 min. This occurs because there is inadequate amount of water to act as a heat sink to dissipate the heat of polymerization, and once above 100 °C, violent, explosive conditions are produced by the vaporization of water trapped in the gel.

Properties of the polymers produced with microwave-assisted polymerization were superior to those with free radical solution polymerization. CRCs stayed in the range of 37–39 g/g, while polymers had higher 0.3 AUL (~15 g/g) and lower extractables (<5%). Even polymers at 50% acid content had comparable properties (see Table II). The temperature profile (see Figure 4) shows polymerization temperature under 100 °C even at 50% acid content. Homogeneity of the polymer in the microwave-assisted polymerization is one reason for getting superior properties. Instead of heating the vessel, the energy is directed toward the monomer solution, and one is able to increase the solid content of the polymer without affecting its properties. Gradient programing helped to control the kinetics in the polymerization step, which resulted in formation of a homogeneous network. Alternating heating
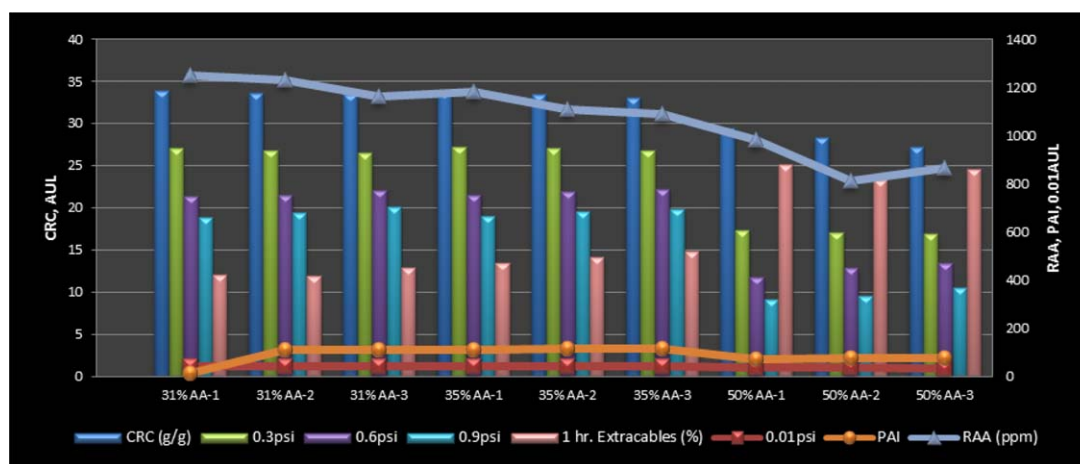

Figure 5. Surface-coated polymer: Measured polymer properties at different acrylic acid content (free radical solution polymerization). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
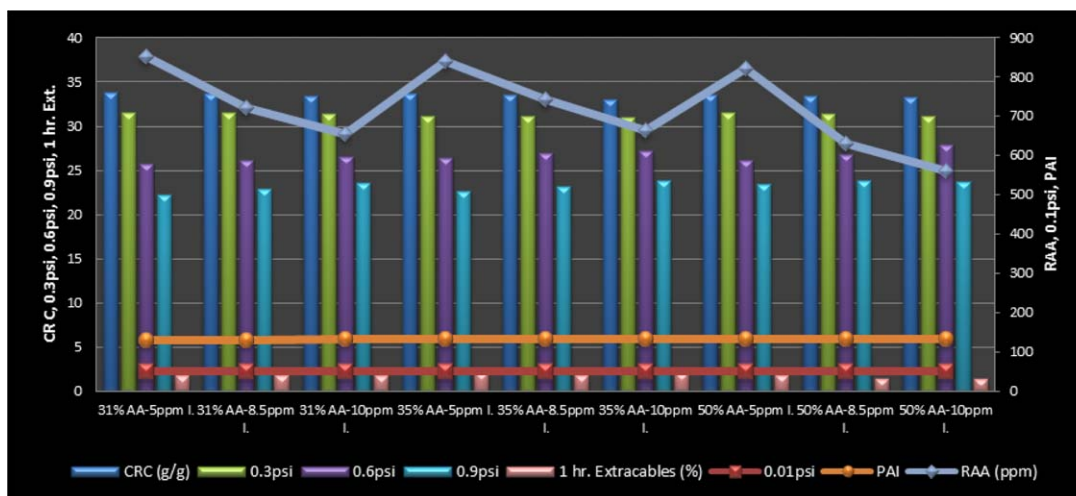
Figure 6. Base polymers with clay-measured polymer properties at different acrylic acid content and clay levels (microwave-assisted polymerization). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and cooling steps helped to keep the uniformity of the network while a condenser provided a relatively controlled environment inside the polymerization vessel. The third factor was optimization of the initiation package.

In microwave-assisted polymerization, a small amount of initiator (5–10 ppm) was used to initiate the polymerization. Low levels of extractables in the polymer could be attributed to the low level of initiator used in the polymerization step. Although initiator is essential in the initiation step, too much of it could be the cause of higher extractables in the superabsorbent polymer. At higher initiator levels, a greater number of smaller polymer chains are produced along with the likelihood for some of these chains not to be crosslinked into the polymer network. These so-called "extractables" are soluble oligomer and polymer chains which can freely migrate from

the crosslinked gel once it begins to swell. They have a thickening or viscosifying effect on the liquid around the polymer, and viscous liquid will have a harder time to penetrate into the polymer network. In accordance with Darcy's Law, speed of penetration of a liquid through a porous medium decreases as its viscosity increases.

In the absence of initiator, the polymerization did not proceed to completion. While one needs <0 ppm of initiator in microwave-assisted polymerization, the level of initiator needed in free radical solution polymerization is more than 300 ppm.

Also, in contrast to the conventional free radical solution polymerization, purging of oxygen out of the monomer solution was not needed due to the efficiency of the heating. On the contrary, in free radical solution polymerization, nitrogen
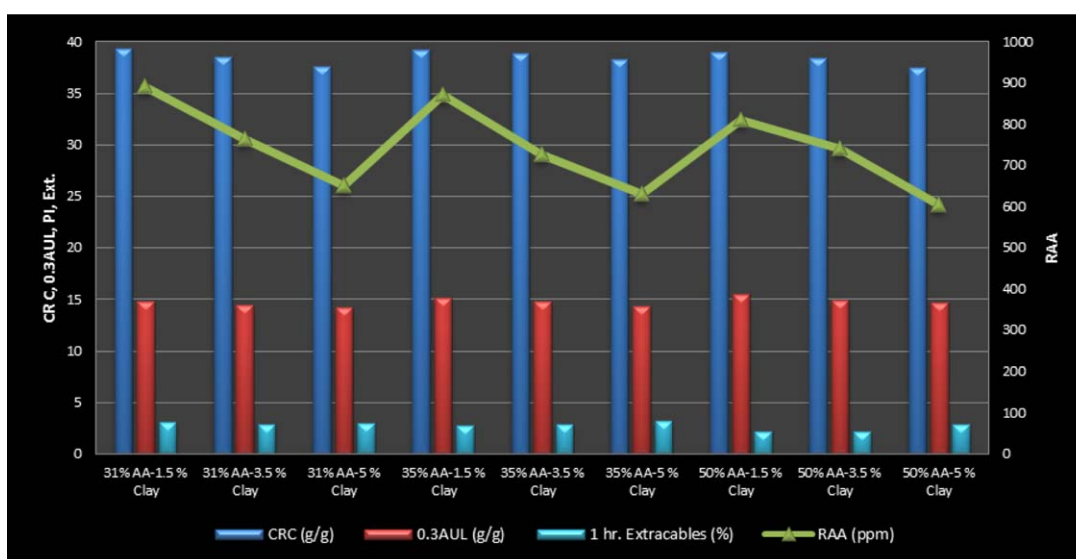

Figure 7. Surface-coated polymer: Measured polymer properties at different acrylic acid content with 8.5 ppm initiator (I.) level (microwave-assisted polymerization). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table III. Surface-coated Polymers: Measured Polymer Properties at Different Acrylic Acid Content (Microwave-assisted Polymerization)

	Samples	CRC (g/g)	0.01 AUL (g/g)	0.3 AUL (g/g)	0.6 AUL (g/g)	0.9 AUL (g/g)	PAI (g/g)
1	31% AA-1.5% Clay	33.5	50.2	31.7	26.1	21.8	129.8
2	31% AA-3.5% Clay	33.7	50.1	31.4	25.8	22	129.3
3	31% AA-5% Clay	33.6	50.2	31.2	26.4	23.1	130.9
4	35% AA-1.5% Clay	34.1	50.9	31.4	26.2	23.1	131.6
5	35% AA-3.5% Clay	33.4	50.3	31.6	26.5	23.4	131.8
6	35% AA-5% Clay	32.9	51.4	32.1	26.1	23.7	133.3
7	50% AA-1.5% Clay	33.6	50.7	31.2	26.5	23.5	131.9
8	50% AA-3.5% Clay	33.1	51.2	31.1	26.7	23.6	132.6
9	50% AA-5% Clay	33	50.1	31	26.4	23.9	131.4

AA, acrylic acid.

purging is necessary to eliminate dissolved oxygen in the monomer solution.[35,36] Due to its electrophilicity, or its affinity for electrons and free radicals, oxygen can prematurely terminate and even inhibit polymerization. Because heating of monomer solution is very efficient with microwave-assisted polymerization, thermal initiation can proceed despite the presence of dissolved oxygen.

To reinforce its gel strength, *base* polymer was coated with 0.1% of EDGE solution and then was cured in a convention lab oven at 155°C for 45 min. Pressure Absorbency Index (PAI) was used as a method for determining the efficiency of this reaction. PAI is the sum of four AULs [0.01 psi (0.7 g/cm^2), 0.3 psi (21 g/cm^2), 0.6 psi (42 g/cm^2), and 0.9 psi (63 g/cm^2)]. This is an important property as one could judge the gel strength of a polymer at different pressure points and choose an appropriate polymer for the intended application.

In free radical solution polymerization, PAIs were in the range of 112–113 g/g for the polymers with the acid contents of 31 and 35%, but it dropped to 74–76 g/g range as the acid level increased to 50%. These polymers (31 and 35%) had CRCs in the range of 33–34 g/g, 0.9 AULs of 18–20 g/g, extractables of 12–14%, and RAA of 1100–1200 ppm (see Figure 4). Again, both CRCs and 0.9 AULs were lower for those polymers made with 50% acid. This clearly shows the destructive effect of uncontrollable polymerization on the polymer's properties.

In the case of microwave-assisted polymerization, base polymers were also surface coated with 0.1% of EGDGE solution, and it took only 20 min to cure the polymer in a conventional lab oven at 155°C. PAIs were in the range of 130–134 g/g for all three acid levels, with CRCs of 33–34 g/g, 0.9 AULs of 22–23 g/g, extractables of 1–2%, and RAA of 600–900 ppm (see Figure 5). These polymers were devoid of any swollen gel permeability.

As was the case with the *base* polymers, properties of the finished products produced with microwave-assisted polymerization were also superior to those produced with free radical solution polymerization. Shorter surface crosslinking time and superior properties are attributed to a stronger *base* polymer which was produced in the polymerization step.
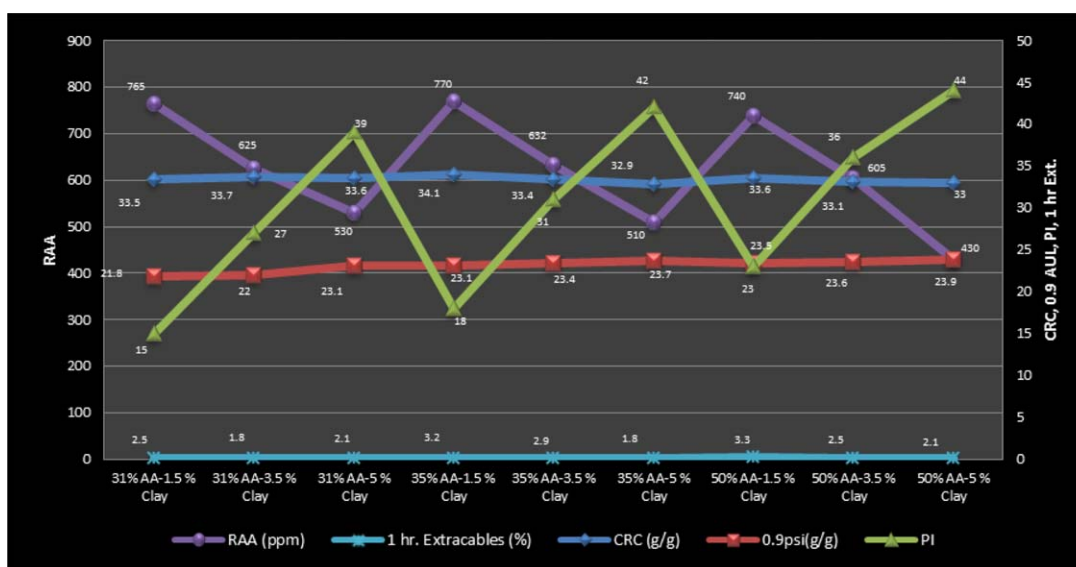

Figure 8. Surface-coated polymers: Measured polymer properties at different acrylic acid content (microwave-assisted polymerization). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
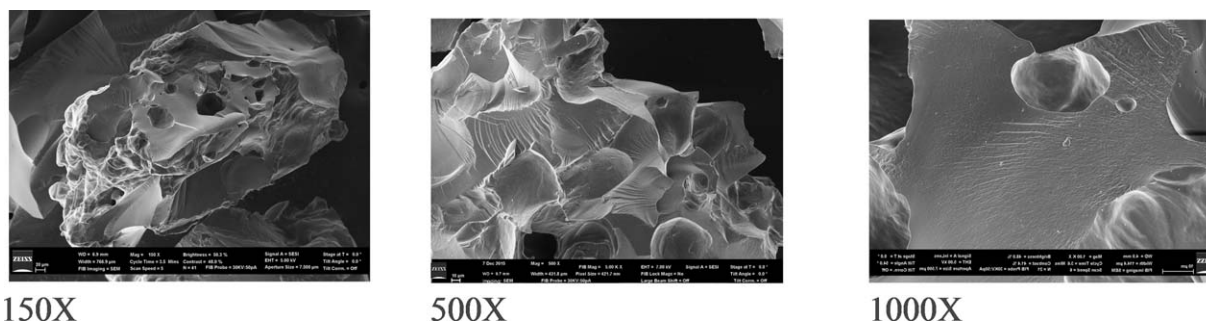
Figure 9. SEM images of the coated polymer particles (without clay) from microwave-assisted polymerization.
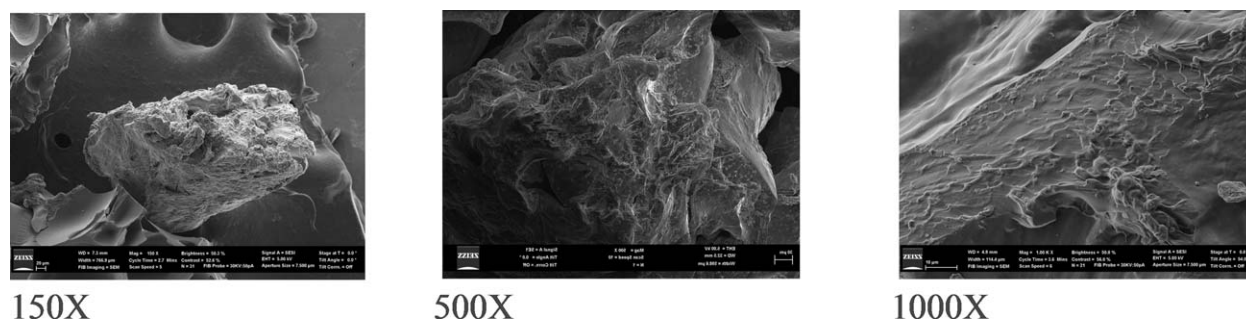

Figure 10. SEM images of the coated polymer particles (with clay) from microwave-assisted polymerization.

As absorbency under load improved after surface coating step, the polymer still lacked permeability. Addition of clay to the monomer solution improved the permeability factor. Depending on the process, introduction of clay to the monomer solution could be challenging. In the free radical solution polymerization, since the initiation reaction is not instantaneous, clay sinks to the bottom and separates from the monomer solution. This produces a heterogeneous polymer with inconsistent properties. On the contrary, due to the fast nature of the reaction, microwave polymerization is much more conducive to the clay addition. Therefore, microwave-assisted polymerization not only could become a platform to improve the polymer's properties, but also to reduce the cost

of producing superabsorbent polymer, both in the polymerization and surface crosslinking steps.

In microwave-assisted polymerization, as clay level increased from 1.5 to 5%, the drop on centrifuge retention capacity of the *base* polymers was in the range of 0.5 to 2 g/g. Since clay has lower capacity than superabsorbent polymer and in addition, it acts as a crosslinker, drop in capacity was predictable. On the other contrary, the increased acid content (from 31 to 50%) had very little effect on the CRC (see Figure 7).

Surface crosslinked polymers had CRCs in the range of 33–34 g/g and 0.9 AULs of 22–24 g/g (see Table III). These are surprisingly close to the properties of those polymers produced
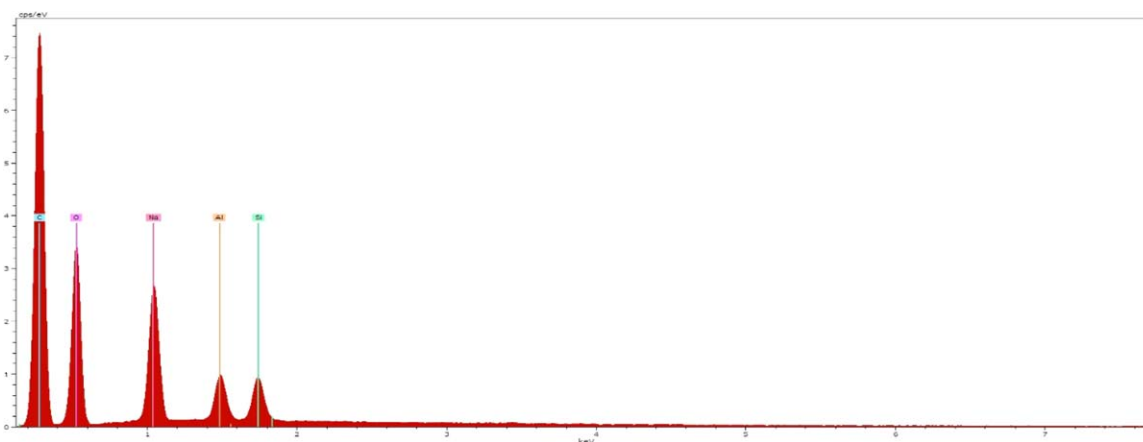

Figure 11. EDX spectra of the clay containing coated polymer particles from microwave-assisted polymerization. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

with no clay in their monomer solutions (see Figure 6). In addition, permeability index was directly proportional to the amount of clay, but the acid level had very little effect on the permeability. The rise in the permeability index (>15 Darcy) with each clay addition in the monomer solution is a good indication that clay is imparting gel permeability to the polymer. In addition, the increased permeability (from 15 to 40 Darcy) was directly proportional to the amount of clay (1.5–5% by weight of the polymer) in the monomer solutions for all three acid levels (see Figure 8). These results indicate that clay is uniformly distributed in the polymer network. Another interesting observation was the lower level of RAA in the clay-containing polymers (about 200 ppm); this could be an indication of an improved polymerization efficiency (see Figures 6 and 8).

SEM images of the clay-containing particles made with the microwave-assisted polymerization have rougher surfaces than those without (see Figures 9 and 10), and EDX spectra show distinct peaks for aluminum and silicon in the clay-containing particles (see Figure 11).

In combination, SEM images, EDX spectra, and permeability data point to the existence and the uniformity of the clay distribution in the superabsorbent polymer.

CONCLUSIONS

Microwave-assisted polymerization not only reduced the polymerization time[37], but also improved properties such as absorbency under load, centrifuge retention capacity, pressure absorbency index, and above all extractables. It also facilitated the addition and uniform distribution of the clay and further imparted improved permeability to the polymer with minimal effect on capacity reduction while improving residual acrylic acid content of the final product. Surface crosslinking time with EGDGE as a crosslinker was reduced to <20 min and polymers properties were better than those produced with free radical solution polymerization. Microwave-assisted polymerization is a major technological improvement over free radical solution polymerization and could be scaled-up without too much difficulty.

In the next phase of this work, co-monomers such as maleic and fumaric acid will be added into the formulation for investigation of their impact on physical properties.

ACKNOWLEDGMENTS

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