Mehran University Research Journal of Engineering and Technology

https://doi.org/10.22581/muet1982.2204.09

2022, 41(4) 87-94

Preparation of a nano-clay-based super absorbent polymer composite for water absorption applications

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Received: 20 May 2021, Accepted: 25 February 2022, Published: 01 October 2022

KEYWORDS

Super Absorbent Polymer Composites (SAPCs)

Nano-Clay

Water Absorbency

Filler

Crosslinking

ABSTRACT

Incorporation of naturally occurring fillers like clay and other mineral improves mechanical, chemical, and physical properties. Herein, we report for synthesized nano-clay based superabsorbent polymer by graft copolymerization of acrylic acid and acrylamide This study focusses the effects of filler (nano-clay), crosslinking agent and initiator concentration on the absorption of the copolymer. Six specimens with varying percentages i.e., 30, 32, 34, 36, 38 and 40 wt. % were supplemented with nano-clay in the matrix of the polymer. Time based immersion was carried out in tap, de-ionized and brine for 2 to 24 h the superabsorbent polymer with 36 wt.% of nano-clay absorbed highest percentage i.e., up to 18 folds after submerging in tap water for 2 hours. Detrimental trend of absorption was observed in specimens with 38 and 40 wt. % that could be due to surge in formation of physical and chemical cross-linking networks in the polymer matrix. However, suspension of superabsorbent polymer composite in brine resulted in decreased absorbency. Such course was also observed for specimens containing 36 wt. % of nano-clay after submerging for 4, 6 and 24 hours. Thermal stability analysis also revealed that the polymer composite supplemented with 38 wt. % of nano-clay exhibited ameliorated stability to 350 °C.

1. Introduction

Super absorbent polymers have gained interest due to their excellent absorption to water, hence making them diverse for food, packaging, sanitary, concrete technology, wastewater treatment, mining and medical applications [1-4]. SAPs tend to absorb and preserve substantial proportion of liquids and water due to its elevated affinity with water in their precise geometry [5-7]. These cross-linked polymeric composites also work in three dimensions making them suitable for absorbing and storing huge amounts of aqueous volume [8].

In literature the Sap has been substituted with hydrogels. However, these both terms have great distinction. Usually, SAPs have possessed dominant attribute of water absorption capacity (1000–100000 %, 10–1000 g/g) than ordinary conventional hydrogels (100 %, 1 g/g) during last two decades' biocompatibility, biodegradability, non-toxicity, abundance, and economic cost suitability has signified the importance and role in synthesizing natural based hydrogels. It has been investigated that by incorporation of micro and nano fillers in the matrix of polymers proved to be viable method in improving the properties of synthesized hydrogel composites.

Hydrogels can be synthesized by either polymers or monomers known as building blocks through formation of cross-linked networks. A homopolymer results in polymerization of any one form of monomer, however a copolymer is formed when one or more distinct monomers attach with each other during copolymerization [9]. Repeating units of copolymers arrange themselves in random, alternating, gradient, block, or graft positions. Polymeric structure develops majority of functional groups that later contributes to important structures and characteristics. Diverse characteristics may be achieved by incorporation of one or more than one distinct monomer or polymer that create ubiquity role in water and energy applications. Cross linking tunes the molecular structure and topography of hydrogels which are desirable for energy and water applications. Generally cross linking of hydrogels is classified into physical (non-covalent) and chemical bonding (covalent bonding). Cross linking may be combined to synthesize functional hydrogels. Such combinations may provide feasible interactions for designing specific target-based applications. Cross linkers and functional additives are the only deciding parameters for the evaluation of swelling behavior of hydrogels. Hydrophilic groups like hydroxylic (−OH), carboxylic (−COOH), amidic (−CONH−), primary amidic (−CONH2), and sulfonic (−SO3H) have greater affinity with the molecules of water and optimize the water content and water absorption. Chemical stimuli are known as pH changes or solvent exchanges. Since hydrogels are composed of acidic or basic functional derivatives their behavior relies on to changes in pH by protonation and deprotonation. Hence the pH sensitive polymers may be charged (swallowed state) and uncharged (yielding hydrophobic or collapsed state due to the environment of pH. Superabsorbent polymers are based on polyacrylic acid (PA), typically synthesized with acrylic acid (AA) monomers. They are inexpensive and highly reactive with different hydrophilic functional groups which deliver sites for cross-linking and making this pitch-perfect choice for absorbent materials [10]. Major concern for SAPCs is their absorption with fluids containing high concentration of electrolytes. Somehow this problem is countered on expense of the cost of composite [11]. Naturally occurring resources are added to improve the properties of super absorbent polymer composites. Clay is amongst those viable replacements to augment the absorbance of the polymer. It has been reported that radical graft polymerization with vinyl monomer and a crosslinker can be employed to synthesize the superabsorbent polymer composite by supplementing naturally present materials like cellulose, starch, and chitosan to extend the absorbency and liquid retention attributes of the polymeric composites [12-14]. Polymer-clay based superabsorbent composites are widely recommended due to their excellent performance for absorbing liquids specifically in agriculture for uninterrupted water supply to the crops. Clays are always preferred as a filler ingredient for polymer composites because of substantially improved properties and low production expenses. Several studies suggest that mechanical, thermal, flame retardancy and wettability of polymer composites show substantial boost with small percentage of clay incorporation. (1~10). The structure of polymer is intercalated with exfoiled chains of polymer composite, this characterized structure depends on additives and opted method for grafting. Performance of polymer composite is improved by appropriate structure of the composite by taking characteristics and nature of clay into consideration. Elaheh Motamedi et al., 2020 studied the water holding and retentions of prepared hydrogels (nano composites) of soil with different as prepared hydrogels. Results exhibited by adding 1w% of hydrogels NCNPs/hydrogel had highest water holding capacity of 53.9% [15]. Foremost major property that superabsorbent need is to hold large amount of water. This is determined by many variables. Because of the high concentration within the material relative to the atmospheric solution, osmotic pressure injects water into the polymer matrix. The combination of charged groups and extra polar groups, such as hydroxyl, carbonyl or amine, absorbs water and creates hydrogen bonds. Swelling capacity is greatly depends on the amount of ionic or polar groups present. Superabsorbent polymers tend to have greater swelling when introduced into a solution with low ionic concentration. Cross link density provides flexibility, superabsorbent with less dense network makes it flexible and yields higher water absorption. However, low degree of cross-linking can result in premature dissolution with significant fraction in the aqueous solution. High temperature promotes detrimental effect on the swelling equilibrium. Although high temperature can also result in high or low swelling conditional to the thermionic response. As appraised before SAPs mostly consist ionic constituents. These ionic charges are responsible for acid base balance due to the variation of pH of the ionic solution de-protonation of acidic and basic groups occur. Acidic (basic) monomer becomes deprotonated below (above) its pKa hence resulting in declined swelling capacity. Subsequently when the pH is higher (lower) than pKa acids become negatively charged and base becomes positively charged resulting in higher swelling rates due to increased affinity with water [16]. Additional repulsion of the charges results in an increased free volume in which water can accrue. When any mechanical force is applied onto a swollen SAP, resulting force shall expel some volume of water out of the swollen SAP, this is also referred to as the absorbency under load. The absorption capacity is also influenced by the surface to volume ratio swell more rapidly than larger particles which is helpful for osmosis desalination. This also concludes that swelling capacity depends on variety of parameters.

Table 1

Comparative study with other works

Substance	Time	Temperature	Water Retention	Reference
hydroxyethyl cellulose-g-P (AAco-AM)	14 d at RM	-	25%	[17]
CMC-g-PAA/GO	80 min	100 ◦C	60%	[18]
St-g-AA-AMPS	10 h	60 ◦C	70%	[19]
Starch-g-P (AA-co-AM)/NCNPs	6 d	-	66%	[20]
P(AA-co-AM)/PACS	72 h	25◦C	>60%	[21]
Amino ethyl chitosan-g-AA	24 h	-	71%	[22]
PAM/Alg	4 d	-	63%	[23]
KGM-g-P(AA-co-AM)	7 d	-	20%	[24]
LW-g-P (AA-co-DMDAAC)	13 h	30 ◦C	90%	[25]
This work (nano-clay)	24 h	-	40%	-

Herein, nano-kaolin that is indigenously available in abundance making it cost effective was used in superabsorbent polymer composite. SAPs usually possess low strength and are prone to disintegrate when exposed to certain environments. From the previous studies it is evident that microfillers did not improve the strength and absorbability due to smaller surface area and non-homogenous distribution of the filler. Nano-fillers fulfill all the mentioned drawbacks of micro-fillers. Hence, this work worth for improving the overall performance of the superabsorbent composite.

2. Materials and Methods

2.1 Materials

Acrylamide purity 98.5% was purchased through Sigma Aldrich. Analytical grade Acrylic acid was purchased from Merck Industrial and Lab Chemicals. Potassium persulfate, and Sodium-Sulfate anhydrous 99% were supplied by Imperial Chemical Industries England. N-N.-Methylene-Bis-Acrylamide ($C_7H_{10}N_2O_2$) & Nano-clay was purchased from Lotte Chemical Industries Republic of South Korea. Chemicals were used as received by the manufacturer.

2.2 Synthesis of Super Absorbent Polymer

Nano-clay based superabsorbent composite was synthesized in a three necked flask. Prescribed percentage of nano-clay was disseminated in distilled water for fifteen minutes at a constant temperature (45°C). Monomer (acrylic acid) was reacted to neutralize the solution by sodium hydroxide with subsequent addition of acrylamide. Furthermore, cross linker and initiators were also incorporated by wt. % in the prepared solution prepared in three necked flasks at 200 RPM for further fifteen minutes at 60°C. After the reaction was terminated, prepared slurry was poured in petri-dish to expel moisture.

2.3 Characterization

FT-IR spectra of the SAPCs samples were obtained with a Thermo Scientific™ Nicolet™ FT-IR spectrometer. The SAPCs-KBr mixtures (1.5:300) were ground, then desorbed at room temperature and pressed to obtain IR-transparent pellets. The thermal stability parameters of SAPCs are analyzed with Perkin Elmer Diamond TG/DTA Thermogravimetric/Differential an average of 10 mg of each sample was heated from room temperature to 600°C under a flow of 100 mL/min of high purity nitrogen. Microstructural characteristics and elemental composition was investigated on Scanning Electron Microscope (JEOL USA JSM-7610F Plus). The water-absorbance process of rewetted super-absorbent polymer composites was evaluated by submerging specimens in various water mediums for a specified time, furthermore the difference between initial and final weight was computed.

3. Result and discussion

3.1 Grafting and Thermal Properties of SAPCs

FTIR spectroscopy was used to investigate the superabsorbent composites supplemented with 36 wt. % of nano-clay as depicted in Fig. 1. The bands at 3690 cm^{-1} and 344 cm^{-1} respectively are because of the extension of –OH group in the absorbent composite. Due to the presence of silicon and oxygen and its out plane stretching at 1117 cm^{-1} can be seen. In plane stretching of

Si-O at 924 cm^{-1} is due to the presence of silicate sheets present in nano-clay. Confirmation of OH and Al-O-Si in superabsorbent composites is depicted at 869 cm^{-1} and 529 cm^{-1} respectively. This FTIR confirms the graft co-polymerization between –OH groups of nano-clay and monomers. Results are in good agreement with recently reported research [26-27].

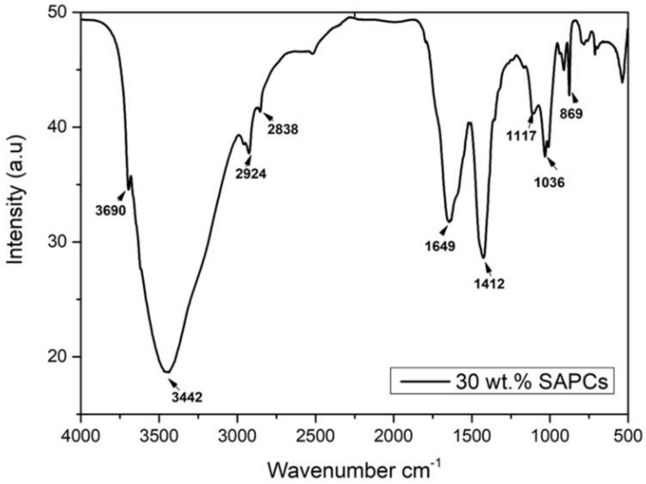

Fig. 1. FTIR- Spectra of superabsorbent polymer composite (SAPCs)

Thermogravimetric analysis was done to analyze thermal stability of the composites with respect to different supplementation of clay into the matrix of superabsorbent polymer composites. Fig. 2 exhibits the degradation phenomenon of superabsorbent composites with varying proportions of clay. After the inclusion of nano-clay, the transfer of thermal decomposition spectra to a higher temperature region shows that the thermal stability of composite is enhanced. In all the clay integrated composites, three-stage decomposition is observed. During the first stage water is expelled which usually occurs in the range of 70-180°C. Second stage dissipates the elimination of amide groups of acrylic acid and acrylamide along with cross-linker from the polymeric chains. At length third stage starts from the range of 680-750°C, due to the thermal deterioration of inorganic clay and polyacrylamide from the skeleton of the polymer. These results are also in agreement with earlier reported work [28-29].

3.2 Surface Morphology of SAPCs

Homogenous filler dispersion plays a crucial role in achieving desired propertied in the polymer composites Fig.3 illustrates the surface topography of the superabsorbent composite, which decipher distinct microstructure of un-grafted polymer composite with respect to the grafted polymer composite. Superabsorbent polymer composite synthesized without grafting exhibits rough surface, non-homogeneous and undispersed filler as shown in Fig. 3(a-b). The SEM images of SAPC incorporated with 34 wt. % of nano-clay is illustrated in Fig. 3(c) exhibits greater homogeneous and permeable structure that is the only reason of water retention in the polymer matrix. Surface morphology of specimen containing 36 wt. % of nano-clay in the superabsorbent polymer composites is shown in Fig. 3(d). The structure is distinguished with finer and improved interstitial spaces that comprehensively enhanced water absorption. EDX analysis of specimen with 36 wt. % of nano-clay in the Fig. 4 manifest the spectra of aluminum and silicon confirming the existence of clay in the synthesized polymer composite. Surface morphology further confirms that acrylamide is successfully grafted and is reason for promoting improved water absorbency in SAPCs. This is consistent with previous reported work [30].

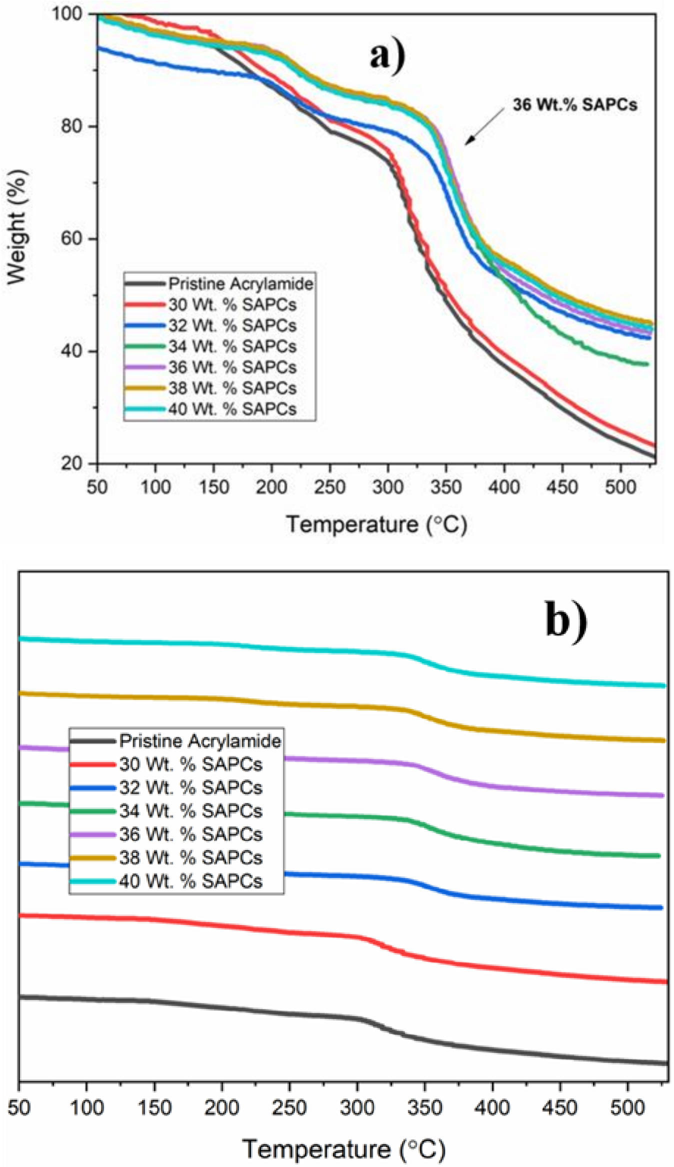

Fig. 2. (a) Weight loss spectra of composite (SAPCs) with different wt. %, (b) Stacked of (a)

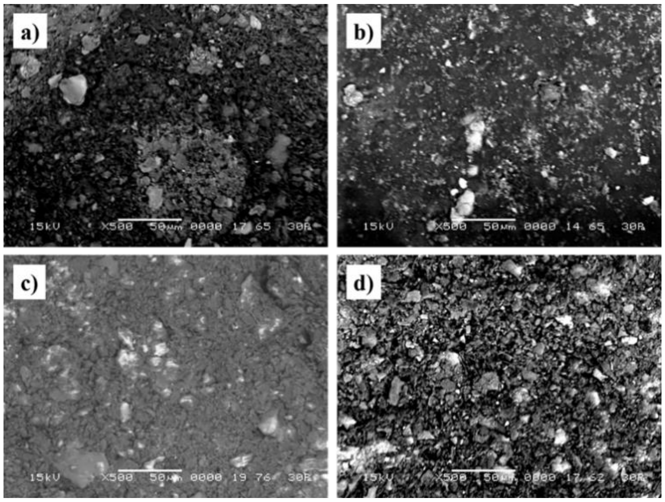

Fig. 3. SEM micrographs of 36. wt. % of super absorbent composites; (a-b) neat-clay (c-d) grafted-clay

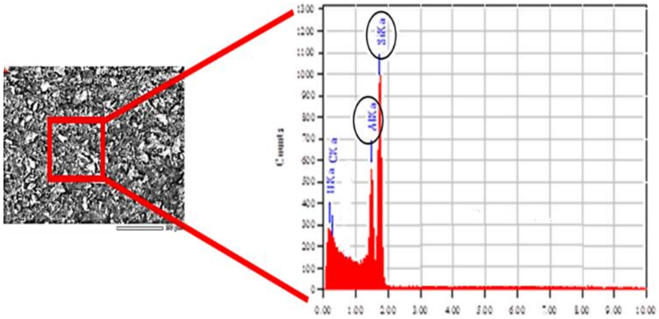

Fig. 4. EDX- Spectrum of 36 wt. % composites (SAPCs)

3.3 Water and Saline Absorbency of SAPCs

SAPCs were immersed in tap, de-ionized and brine mediums to evaluate swelling behavior with respect to time as depicted in Fig. 5. The water absorption of SAPCs with 30 and 32 wt. % showed incremental rise in absorption reaching to ~138% and ~134% respectively as depicted in Fig. 5(a). Substantial rise in water absorbency was measured in SAPC with 36 wt. % of nano-clay after swamping in tap water. However further incorporation of inorganic filler i.e., 38 and 40 wt. % has shown declined water absorption. This is due to the development of physical and chemical cross-links as interaction of clay is increased with acrylic acid. The structure of polymer also becomes conjugated and less elastic, and additional formation of chemical bonds further reduces water absorption [31-32]. Highest water absorbency in tap water is observed in specimens with 36 wt. % of nano-clay reaching at ~184%. Similar patterns have also been observed with specimens sub-merged in water for longer period of time as the values escalated between 305% and 325.87% for specimens with 36 wt. % of nano-clay at eight and twenty-four hours respectively as illustrated in Fig. 5(b-c). Water absorbency after 48 hours is illustrated in fig. 5(d) shows no significant changes in absorption for specimens immersed for 24 h. Absorption behavior in saline water also showed negligible change. Maximum water absorbency was 407.87% for superabsorbent composites with 36 wt. % of nano-clay. It is worth to mention here that absorbency in tap and de-ionized water is greater as compared to brine. This phenomenon is due to the presence of valence cation in the brine solution which halts the matrix of the polymer to bind with the molecules of water [33]. Cross-linker has great influence on the water absorption Fig. 6 illustrates the detrimental water absorption properties with respect of changing wt. % of cross-linker i.e., 0.5-2.5 wt. % in the superabsorbent polymer composites. The effect of lower absorption is due to higher cross link densities that ultimately decreases porosity with increasing concentration of cross-linker. However, the effect of initiator concentration was also studied which effects the absorption as the cross-linking activation is directly proportional the concentration of initiator. Fig.7 illustrates the effect of initiator on the absorption behavior, showing maximum absorption measured 34 folds. The findings are well in line with the reported literature [34].

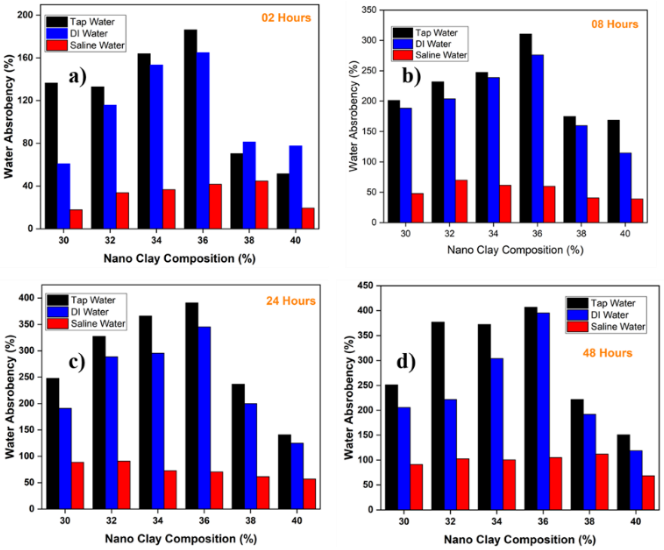

Fig. 5. Water absorption % of SAPCs absorbed in various media for (a) 02 hours; (b) 08 hours; (c) 24 hours; (d) 48 hours

4. Conclusion

By adding nano-clay, superabsorbent polymer composites are prepared for the copolymerized graft of acrylic acid and acrylamide. Increased in filler proportion resulted in higher water absorption and thermal stability. Composites comprises 36 wt. % of nano clay shows optimal water absorbency. The chemical reactions of the carboxyl and hydroxyl groups were further confirmed by FTIR spectroscopy, that evident the grafting of polymers. Surface of the synthesized composite was analyzed by SEM that illustrated more homogenous and permeable structure after graft copolymerization reaction. It is also established from this study that by increasing the weight percentage of crosslinker dwindled the water absorption, however escalated amount of initiator had an ameliorated effect on the water absorbency of the SAPCs. For water absorbency applications, the developed composite can potentially be used.

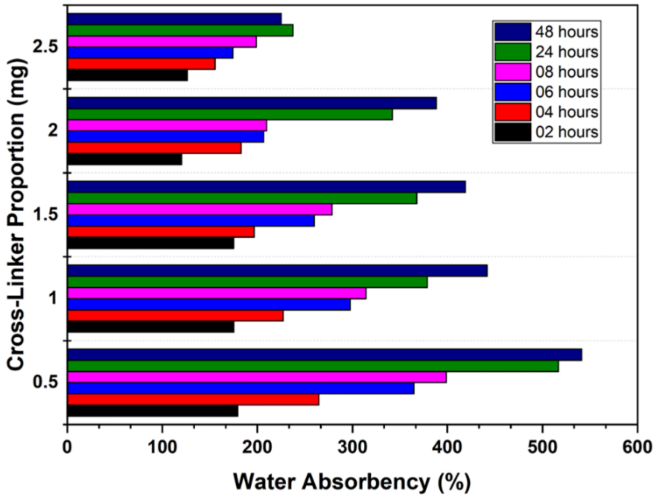

Fig. 6. Influence of cross-linker concentration on SAPCs water absorption at different time intervals

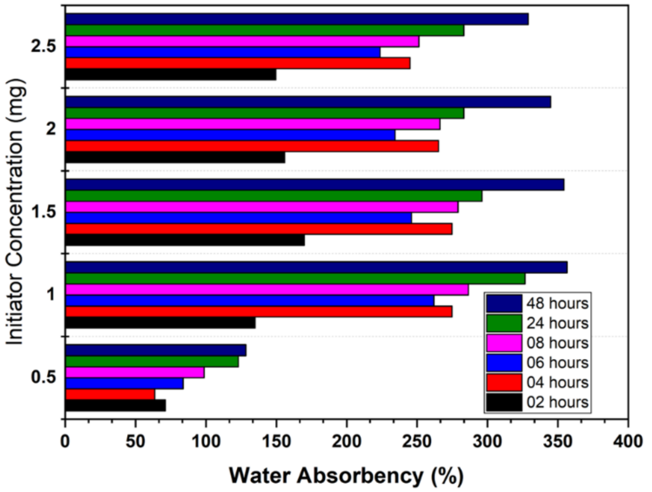

Fig. 7. Influence of initiator concentration on SAPCs water absorption at different time intervals

Acknowledgment

We would like to acknowledge Mehran University of Engineering & Technology, Jamshoro for financial support. We also thank High-Tech advanced research laboratory, University of Sindh, Jamshoro for providing us the facility of FTIR Spectroscopy and Thermogravimetric analysis (TGA).

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