BCSIR

Available online at www.banglajol.info

Bangladesh J. Sci. Ind. Res. **51(2)**, 155-158, 2016

Short Communication

BANGLADESH JOURNAL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

E-mail: bjsir07@gmail.com

Characterization and surface activity of soluble sulfonated butanone formaldehyde superplasticizer for cement

S. Sultana[1], Z.Yeasmin[2]*, S. Ahmed[2], H. P. Nur[2], P. R. Samaddar[1]

[1]*Eden Mohila College, Dhaka, Bangladesh*

[2]*Institute of Glass & Ceramic Research and Testing (IGCRT), Bangladesh Council of Scientific and Industrial Research (BCSIR), Dhaka, Bangladesh*

Abstract

Water soluble sulfonated butanone formaldehyde (SBF) polycondensate superplasticizer was prepared according to a conventional method. UV-Vis, IR and TGA analyses were performed to analyze the target compound. The surface activity was evaluated. It showed a little surface activity with respect to water as the surface tension decreased very little with increasing the concentration of SBF. Thus there results indicate that SBF behaves like a typical polycondensate superplasticizer such as sulfonated acetone formaldehyde (SAF).

Keywords: Cement; Concrete; Superplasticizer; Surface tension; Thermo gravimetric analysis

Introduction

Nowadays superplasticizer, an important chemical admixture, attracts a great attention of scientists due to its high workability and enhancing strength to the cementitious pastes (Ming *et al.*, 2013). Superplasticizers are organic polyelectrolytes that improve the fluidity of cement by dispersing cement particles present in the paste (Lei *et al.*, 2012) at low cement-water ratio. The superplasticizer broadly classified into four groups: sulfonated melamine formaldehyde condensate (SMF), sulfonated naphthalene formaldehyde condensate (SNF), modified lignosulfonates (MLS) and others including sulfonic acid esters, polyacrylates and polystyrene sulfonates etc. The sulfonic acid or sulfonate groups are responsible for neutralizing the surface charges on the cement particles and causing dispersion (Mindess and Young 1981). Due to higher cost of the synthetic superplasticizer, the usage of it is still not common. Recently less expensive aliphatic ketone based sulfonate acetone formaldehyde condensate (SAF) has been synthesized as a new type of water reducer (Ming *et al.*, 2013; Lei *et al.,* 2012; Pei *et al.* 2004; Zhi *et al.,* 2003).

In this study the sulfonated butanone formaldehyde condensate (SBF) was prepared in the laboratory from the reaction between butanone-formaldehyde and sodium sulfite. There are several synthetic procedures in the literature for SAF. Butanone-2 was used instead of acetone in the present case.

Materials and methods

All commercial products of butanone-2, formaldehyde solution (37% aqueous solution) and sodium sulfite were purchased from Merck, Germany and were used without further purification. Hitachi UV-Vis double beam spectrophotometer (U-2910) was used to record the UV data. Distilled water was used as the solvent. Five different solutions of SBF were prepared having concentrations from 0.02 g/L to 0.1 g/l.

FTIR spectrum was recorded on a FTIR-Prestige 21 (Shimadzu). The spectrum of the SBF superplasticizer was recorded over a wave number range of 4000-400 cm^{-1}. The sample was prepared as a standard KBr pellet.

Surface tension was measured by Sigma 700/701 tensiometer. The liquid was placed in the appropriate vessel. A platinum plate was then suspended in the liquid. The instrument was adjusted to zero with the plate in the liquid. The plate was pulled upward vertically until it broke free from the liquid. The measured value at the break point was recorded as the surface tension.

*Corresponding author. e-mail: zenefar@gmail.com

The thermogravimetric analysis (TGA) was carried out on a EXSTAR TG/DTA 6300 with alpha-alumina powder as a reference sample from 30 to 750 °C in nitrogen atmosphere (flow rate 50 nmL/min). The heating rate was 20 °C/min.

Anhydrous sodium sulfite and water were taken into a three neck round bottom flask. The temperature of the mixture was maintained at 45 °C. When the solution became clear, the pH was adjusted to ~13 by addition of 40% (w/v) NaOH solution slowly. Subsequently 1/10 of total formaldehyde aqueous solution (37%) was added into the vessel. When the temperature of the solution reached to 60 °C, the mixture of formaldehyde and butanone-2 solution were added dropwise with the help of dropping funnel for about 20 minutes. During the addition, the temperature of the solution increased automatically and it was controlled at 60-65 °C. Then the reaction was allowed to continue for 3 hours at 85-90 °C. The reaction mixture was then allowed to cool down at room temperature. The solution was poured into the beaker and dried in oven at 100 °C. The product was analyzed and characterize without further purification.

Results and discussion

Several methods have been reported in the literature (Ming *et. al.*, 2013; Pei *et. al.* 2004; Zhi *et. al.* 2003) to synthesize SAF. The reaction procedure was similar to that described by Ming (Ming *et. al.*, 2013). However, the molar ratio of butanone-2 and sodium sulfite was maintained at 0.55 (Zhi *et. al.,* 2003). In another work Pei showed that good water solubility for SAF was obtained when the ratio of formaldehyde-acetone was 2.0 and the temperature of the reaction was maintained at 85 °C (Pei *et. al.* 2004). The temperature of the condensation reaction was maintained accordingly.

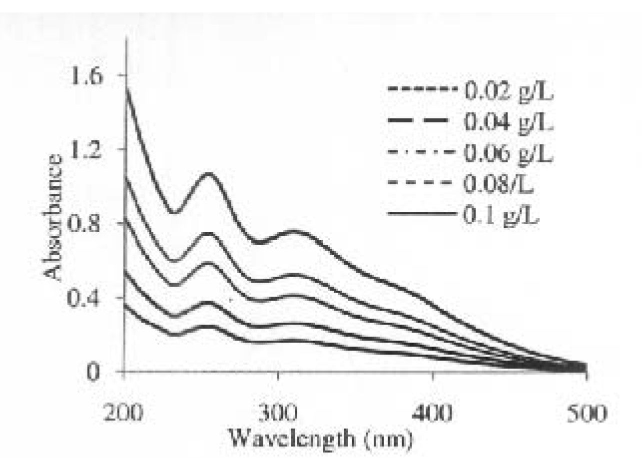

Fig. 1. The overlay spectra of 0.02, 0.04, 0.06, 0.08, 0.1 g/L SBF solutions at 30 °C.

UV-Vis spectra analysis: The λ_{max} was found at 254 nm for all of the spectra at 30 °C. However the observed absorption peak for SAF was found to be 260 nm from literature (Lou *et. al.,* 2012). Fig.1. showed the overlay spectra of SBF solution at different concentrations.

FTIR spectra analysis: Fig. 2 shows the infrared spectrum of SBF. The hydroxyl group appears as a broad peak at 3442 cm^{-1} due to stretching vibrations of O-H group. The stretching frequency at 2929 cm^{-1} is due to the C-H vibration.

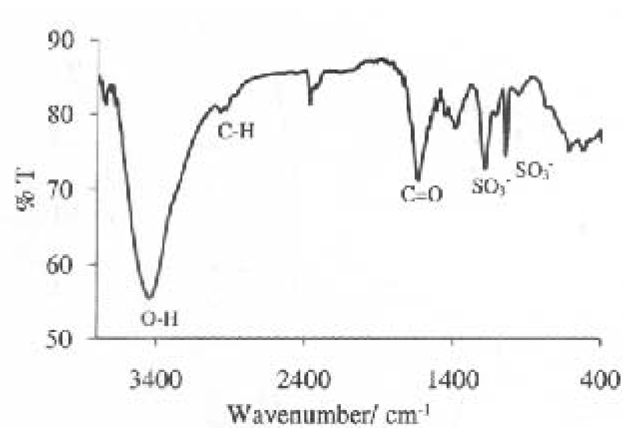

Fig. 2. The FTIR spectrum of SBF resin

The strong peaks appeared at about 1188 cm^{-1} and 1042 cm^{-1} are due to sulfite group. The analyses proved that the synthesized SBF has hydroxyl, carbonyl and sulfonic group in its structure. The spectral data showed good agreement with the literature (Ming *et. al*., 2013). The proposed chemical structure of the SBF resin is shown in Fig. 3 (Zhou *et. al.,* 2007).

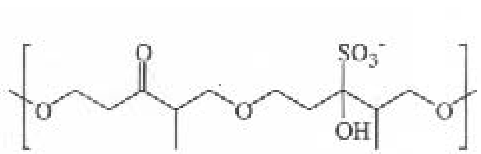

Fig. 3. The chemical structure of SBF resin

Surface tension analysis: To understand the surface activity of SBF, surface tension was measured. The surface tension of SBF solution at different concentrations was measured at 30 °C. Like the SAF, SBF resin is a polymer having many hydrophilic and hydrophobic chains. The results were shown in Fig. 4.

Compared to distilled water (68.2 mN/m), the surface tension of the solution with 1.0 g/L of SBF was found to be 52.5 mN/m. It is quite evident from the Fig. 4 that the surface tension decreases very little with increase in concentration of the SBF resin. There is no evidence observed for critical micelle concentration like other surfactants (Zhou *et. al.* 2007). Lower surface activity was found for polycarboxylate superplasticizer (PCS) compared to SAF (Ming *et. al.*, 2013). As the SBF resin showed similar trend like SAF resin, it can be concluded that SBF would possess little surface activity compared to PCS.

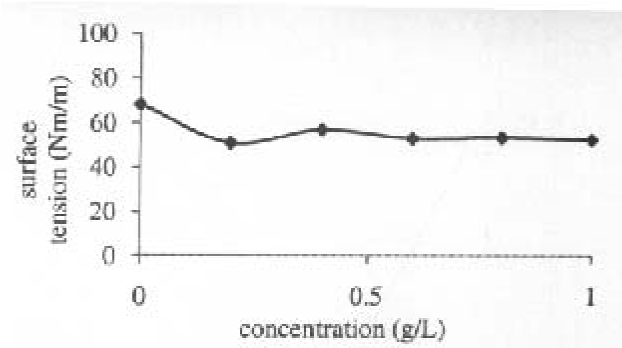

Fig. 4. The effect of concentration of SBF solution on surface tension

Thermogravimetric analysis: Thermal stability of SBF resin was carried out by TGA. Fig. 5 shows the TGA diagram for SBF superplasticizer. First 5 and 10 % of decomposition was observed at 62 °C and 105 °C respectively. A rapid decomposition was found to be at 332.6 °C where 77% weight loss took place. Similar weight loss was observed for SAF (Zhu *et. al.* 2012).

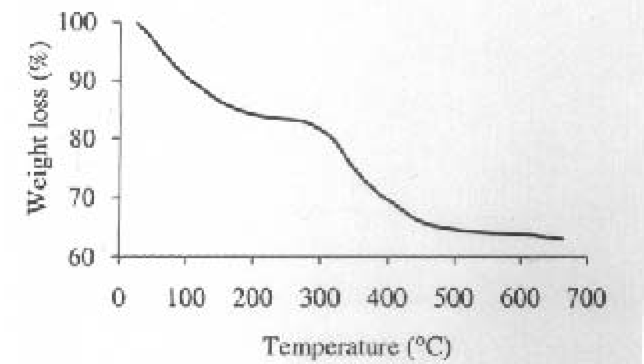

Fig. 5: The TGA of SBF resin

Wang showed that sulfonated melamine formaldehyde (SMF) and new melamine superplasticizer (NMS) possessed good thermal stability as the 80% weight loss has been observed in the range of 290-360 °C (Wang *et. al.,* 2013). The main reason for the higher stability in melamine superplasticizer is due to the triazine ring of melamine which enhances the rigidity of the chain (Wang *et. al.,* 2013). Such rigidity is absent in the chemical structure of SBF superplasticizer. Hence the thermal stability of SBF is slightly lower than that of melamine superplasticizer.

Conclusion

A SBF polycondensate was synthesized from butanone-2, formaldehyde and sodium sulfite. The result shows that SBF behaves like a typical polycondensate superplasticizer such as SAF. It shows little surface activity compared with other superplasticizers. Thermal analysis reveals that SBF shows relatively good thermal stability. Hence it can be used at high temperature environment.

Acknowledgement

Thanks to the Ministry of Science and Technology (Bangladesh) for financial support through Special Allocation Project 2015-16. Authors are grateful to BCSIR authority for funding. Special thanks to Dr. Kazi Nasrin Farooque. Thanks to Prof. Abu Bin Hasan Susan, Department of Chemistry, Dhaka University and Sajib Aninda Dhar, Engineer, PP & PDC, BCSIR, Dhaka for providing tensiometer and TGA facilities respectively.

References

Lei L and Plank J (2012), Synthesis, working mechanism and effectiveness of a novel cycloaliphatic superplasticizer for concrete, *Cem. Concr. Res.*, **42**: 118-123.

Lou H, Ji K, Lin H, Pang Y, Deng Y, Qju X, Zhang H and Xie Z (2012), Effect of molecular weight of sulfonated acetone-formaldehyde condensate on its adsorption and dispersion properties in cementitious system, *Cem. Concr. Res.*, **42**: 1043-1048.

Mindess S and Young J F(1981), Concrete. Englewood Cliffs, N.J.: Prentice-Hall, Inc.

Ming L, Jiaheng L, Xiaodi D, Chengfei F and Bo H (2013), Synthesis, properties and dispersion mechanism of sulfonated acetone-formaldehyde superplasticizer in cementitious system, *J. Wuhan. Univ. Technol.-Mater. Sci. Ed.,* 1167-1171.

Pei M, Yang Y, Zhang X, Zhang J and Li Y (2004), Syntheis and properties of water-soluble sulfonated acetone-formaldehyde resin, *J. Appl. Polym. Sci.*, **91**: 3248-3250.

Zhi Z X, Qing Y Y, Bao D L and Pei M (2003), Synthesis and water-reducing property of water soluble sulfonated acetone formaldehyde resin, *J. Qingdao Univ. Sci. Tech.,* **4**: 315-317.

Zhou M, Qiu X, Tang D andWang W (2007), Synthesis and evaluation of sulfonated acetone-formaldehyde resin applied as deispersant of coal-water slurry, *Energ. Convers. Manage.,***48**: 204-209.

Zhu J, Zhang G, Li J, Zhao F and Qu Q (2012), Synthesis and investigation of sulfonated acetone-formaldehyde polycondensate as dispersant for coal-water slurry, 2nd International Conference on Electronic and Mechanical engineering nd Information Technology (EMEIT-2012), 0610-0613.

Wang H, Yang X, Xiong W, Liu X and Zhang Z (2013), Synthesis and the effects of new melamine superplasticizer on the properties of concrete, ISRN *Chem. Eng.* (2013).

Received:23 November 2015; Revised: 27 December 2015; Accepted: 31 January 2016.