ARTICLE IN PRESS
Journal of King Saud University – Engineering Sciences (2016) xxx, xxx–xxx

King Saud University
Journal of King Saud University – Engineering Sciences
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ORIGINAL ARTICLE

Optimizing homogenization parameters for improving ethylene vinyl acetate emulsion stability in pour point depressant application

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Received 1 December 2015; accepted 24 January 2016

KEYWORDS
Emulsion;
Ethylene vinyl acetate;
Homogenization;
Stability;
Pour point depressant

Abstract Considerable attention is currently devoted to producing a cold-resistance pour point depressant (PPD) via the emulsification process. The aim of this study is to optimize the emulsification process parameter as to yield a stable emulsion. Shearing intensity, temperature and time of the emulsification were studied as the parameters to optimize the process. The influence of these parameters on the emulsion properties i.e. particle size, emulsion morphology and freeze–thaw stability was investigated. The particle size of the emulsion is reduced from 0.7103 μm to 0.5185 μm when shearing intensity increased and maximum emulsion stability was achieved by 120 days at 5000 rpm. It was also identified that the particle size and emulsion stability are smaller and longer respectively when the homogenization temperature increased. Emulsion produced at 80 °C presented superior emulsion stability than other homogenization temperature. Prolonged homogenization time showed a positive effect on the emulsion stability from 20 to 30 min. Morphological studies by microscopy illustrated that smaller and uniform emulsion particle was achieved. The results outlined that the optimum homogenization parameters are: stirring intensity, 5000 rpm; homogenization temperature, 80 °C and homogenization time, 30 min.

1. Introduction

Emulsions are viewed as one of the mass practical areas of interest because of their extensive application in food, cosmetics and pharmaceutical industries (Vignati et al., 2003). An emulsion is a type of dispersion in which two normally immiscible substances are stabilized by another substance, called an emulsifier. While we can agitate to form a suspension, it is temporary and the oil and water will eventually separate into distinct layers. Tzoumaki et al. (2011) stated that emulsion

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Peer review under responsibility of King Saud University.

Production and hosting by Elsevier

http://dx.doi.org/10.1016/j.jksues.2016.01.006

preparation and stabilization can conventionally be achieved by prolonged mechanical agitation and addition of emulsifiers or other surface active polymers.

On the other hand, in PPD chemical production, emulsion technology being less explored and exposed is quite beneficial to be further studied in this area since there are only a few studies regarding PPD emulsion product. Becker (1999) pinpointed that PPD emulsion products are more advantageous for the use in sub-ambient temperature as it improves the physical handling characteristics compared to traditional products. Many studies have been conducted to describe the efficiency of EVA copolymers as pour point depressants compared with other additives to treat crude oil (Machado et al., 2001; Pedersen and Rønningsen, 2003; Taraneh et al., 2008).

Yet, there are no further studies on the homogenization factors to maintain or improve PPD emulsion stability for example homogenization intensity, temperature and time. Hence, this research is focusing on all those factors which indirectly relate to emulsion properties and science which is quite uncommon compared to the above applications as applied in oil and gas industries. Hall et al. (2011) also mentioned that formulated emulsions are highly dependent on the manufacturing process and the chemical formulation. Both the material and process parameters therefore control product properties. Maintaining the emulsion product quality is crucial to ensure that emulsion separation does not occur after changes in temperature. Jennings and Newberry (2008) stated that a pour point depressant applied in cold temperature, for example in deep water, must remain stable and completely maintain its fluidity, so that the injection line is not compromised.

Given the ingredients of an emulsion and the emulsifying machine, there are several parameters that need to be controlled in order to obtain the desired emulsion properties. The first parameter is the shearing intensity. As the shearing intensity increases, it enhances the colloid break-up process as the particle size becomes smaller (Rámirez et al., 2002). Therefore, efficient agitation gives better emulsion. Particle size is a key factor that affects emulsion properties, such as emulsion viscosity (Heldmann et al., 1999) and stability (Ghosh and Rousseau, 2009). Particle size mainly depends on the processing conditions (Walstra, 1993), especially the mechanical force or stirring rate during the emulsification process. Degner et al. (2014) emphasized that the destabilization of emulsions through sedimentation can be avoided through a reduction in particle size.

The second parameter is the homogenization temperature. Homogenization temperature often has indirect effects on emulsification as a result of altering the interfacial tension, adsorption of emulsifier and viscosity. Higher temperature is advantageous for emulsification due to reduction in viscosity and interfacial tension. There is also some evidence that a sharp increase or decrease of temperature tends to coagulate the particles, thereby causing the deterioration of emulsions (Chen and Tao, 2005; Joshi et al., 2012). Lastly, the third parameter that influences the quality of emulsion produced by homogenization is the homogenization time. (Thakur et al., 2010) reported in their study that the colloid particle dispersion becomes more efficient where there is an increment in homogenization time. But beyond a certain level of time, the probability of collision and coalescence of newly formed colloid particle increases as it will produce bigger particle size (Garciaa et al., 2012; Joshi et al., 2012).

Emulsion stability during the freeze–thaw (F/T) cycle has been studied by many research groups, and various methods of determining emulsion stability have been proposed (Lin et al., 2008; Ghosh and Rousseau, 2009; Shahin et al., 2011). One of the methods commonly used to identify emulsion stability is by determining the particle size of the emulsion. Manka et al. (1999) stated that the particle size of an emulsion usually ranges from 0.1 μm to 10 μm in diameter. Emulsions are commonly prepared via a two-step process. In the first step, a coarse emulsion or premix is prepared by thorough mixing of the ingredients in a low-shear device. In the second step, the coarse emulsion is passed through a high-energy mechanical device, such as a high-shear mixer (Maindarkar et al., 2014).

Atiemo-Obeng and Calabrese (2004) indicated that compared with the conventional mechanically stirred vessels, high-shear mixers (HSMs) which are high-shear reactors, rotor–stator mixers, and high-shear homogenizers are the characteristics of high rotor tip intensities (ranging from 10 m/s to 50 m/s) and relatively high shear rates (ranging from 20,000 s^{-1} to 100,000 s^{-1}). In the current study, we examine the effect of using a high-shear mixer in the emulsification process compared with using the conventional mechanical mixer as a function of PPD.

In an effort to have a better understanding of the effect of the homogenization parameter in producing stable EVA PPD emulsion, the homogenization parameter needs to be improved. The effects of several homogenization variables on the stability of EVA copolymer emulsion were studied in the present work. The variables included shearing intensity, homogenization temperature and time.

2. Experimental

2.1. Materials

EVA copolymer (12% of VA), 2-ethyl-hexanol, paraffin wax, and sorbitan monooleate (Span 80) were purchased from Sigma Aldrich. Glycol solution and hexane were acquired from Merck. Distilled water was used as the aqueous phase in the emulsion. All other materials were used without further purification.

2.2. Pour point depressant emulsion preparation procedure

Some parts of the polymers were vigorously mixed with the solvent, with heat applied for 2 h. A surfactant was added into the molten polymer blend and homogenously stirred for 30 min. An anti-freeze (glycol solution) agent, together with some portions of distilled water, were then slowly added in the blend solution and stirred for another 30 min. Lastly, at the homogenization step the premixed emulsion was cooled down a bit to 80 °C before it was homogenized for 30 min at 1000 rpm with the use of an IKA RW 20 digital overhead motor equipped with three-bladed propellers. The steps were repeated while the mixing mechanism was switched to a Silverson L5M-A high-shear mixer unit equipped with emulsor screens, with the shearing intensity varied for the homogenization step. Then, the temperature and time for homogenization step were also studied to optimize the processing parameter. The emulsion formulation is summarized in Table 1.

Please cite this article in press as: Rosdi, M.R.H. et al., Optimizing homogenization parameters for improving ethylene vinyl acetate emulsion stability in pour point depressant application. Journal of King Saud University – Engineering Sciences (2016), http://dx.doi.org/10.1016/j.jksues.2016.01.006