



Waterborne polyurethane dispersions synthesized from jatropha oil



Sariah Saalah^{a,b,*}, Luqman Chuah Abdullah^{a,c}, Min Min Aung^{c,e}, Mek Zah Salleh^d, Dayang Radiah Awang Biak^a, Mahiran Basri^e, Emiliana Rose Jusoh^c

^a Department of Chemical and Environmental Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

^b Chemical Engineering Department, Faculty of Engineering, Universiti Malaysia Sabah, Jalan UMS, 88400 Kota Kinabalu, Sabah, Malaysia

^c Institute of Tropical Forestry and Forest Products, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

^d Radiation Processing Technology Division, Malaysian Nuclear Agency, 43000 Kajang, Selangor, Malaysia

^e Department of Chemistry, Faculty of Science and Technology, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

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ABSTRACT

A series of waterborne polyurethane dispersions derived from jatropha oil-based polyol (JOL) with different OH numbers ranging from 138 to 217 mgKOH/g, were successfully prepared. Jatropha oil-based polyols were synthesized by epoxidation and oxirane ring opening using methanol. The JOLs produced were then used to prepare jatropha oil based waterborne polyurethane (JPU) dispersions by reaction with isophrene diisocyanate (IPDI). Dimethylol propionic acid (DMPA) was used as an internal emulsifier to enable the dispersion of polyurethane in water. The influence of the OH number, DMPA content and hard segment content on the stability of the wet JPU dispersions, as well as the physical, mechanical and thermal properties of the dry JPU films were investigated. The results reveal that with increasing OH number, the DMPA content and hard segment content significantly decrease the particle size from 1.1 μm to 53 nm, indicating increasing stability of the dispersions. JPU films exhibit the stress–strain behavior of an elastomeric polymer with a Young's modulus ranging from 1 to 28 MPa, a tensile strength of 1.8 to 4.0 MPa and elongation at break ranging from 85 to 325%. The polyurethane dispersions synthesized in this work possess good pendulum hardness, water repellence and thermal stability with promising application as a binder for wood and decorative coatings.

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1. Introduction

Many of the commercial polyurethane adhesives and coatings are known to contain a significant amount of organic solvents and some also contain volatile isocyanate which is harmful to human health as well as the environment. Increasing concern on this issue has pressured the urethane industry to move toward water based systems. Waterborne polyurethane (PU) dispersion is a recent class of binder that is easily diluted with water and air-dried, stoved or crosslinked to produce volatile organic component (VOC) compliant coatings (Athawale and Nimbalkar, 2011). As with their solvent based counterparts, waterborne PU dispersions find application mainly in the ink, surface coating and adhesive industries. Polymers derived from waterborne PU dispersions exhibit an excellent combination of physical properties such as high tensile and tear

strength, high elasticity, a range of hardness, excellent abrasion resistance, good resistance to chemicals and good low temperature stability (Davies and Some, 1990).

Waterborne PU dispersions can be prepared by adding water to a segmented polyurethane ionomer containing a very small amount of solvent as a diluent agent. Ionomers which contain hydrophilic groups are incorporated into the side chain or backbone of the polymer to enable dispersibility of water-insoluble polyurethane. The ionomer could be an anionic or cationic type but an anionic type such as dimethylol propionic acid (DMPA) is commonly used. The DMPA acts as an emulsifier to provide dispersion stability for longer storage of the waterborne PU dispersion. The hydrophilicity of ionic groups in the DMPA is reported to improve the mechanical properties, but tends to make the dispersion film more sensitive to water and chemicals (Bullermann et al., 2013). Therefore, the amount of DMPA should be controlled to be as low as possible in the polyurethane prepolymer.

Currently, most waterborne PU dispersions are derived from petroleum based polyol which is non-renewable. As consumers are becoming more aware of environmental issues, they are changing their preference toward bio-based products. In addition, people

* Corresponding author at: Department of Chemical and Environmental Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia. Tel.: +60 3 89466288.

E-mail address: s.sariah@ums.edu.my (S. Saalah).

in industry are constantly looking for alternatives as the price of fossil oil is typically increasing. Recently, the synthesis of waterborne PU dispersions derived from vegetable oil such as soybean oil, castor oil, rapeseed oil and linseed oil have been reported (Chang and Lu, 2012, 2013; Lu and Larock, 2008; Ni et al., 2010). However, to the best of our knowledge, no research has been reported concerning jatropha oil regarding its promising properties to produce waterborne dispersions. In Malaysia, jatropha become one of the most important crop after palm oil and rubber, mainly planted for biodiesel production. BATC Development Berhad was actively engaged in jatropha plantation and bio-fuel industry since 2007. Up to 2011, about 600,000 acres planted areas, 3.3 million areas landbanks and more than 300 nurseries and collection centers were reported in Malaysia (Bionas, 2011).

Jatropha oil (JO) which is extracted from the seeds of the jatropha fruit is a promising candidate for chemical purposes as it contains 78.9% unsaturated fatty acids, mainly of oleic acids (43.1%) and linoleic acid (34.4%) (Sarin et al., 2007). This high degree of unsaturation provides a broad alternative for chemical modification to produce polymers with various properties. Furthermore, it is an advantageous to use jatropha oil because it is a non-edible oil and thus its usage will reduce the consumption of edible oils for chemical purposes (Rios et al., 2013). Previous researches have revealed the potential of producing alkyd resin (Boruah et al., 2012), polyurethane coatings (Sugita et al., 2012), polyurethane adhesive (Aung et al., 2014) and polyurethane elastomer (Hazmi et al., 2013) from jatropha oil. In this research, an attempt is made to produce a waterborne PU dispersion using a jatropha oil-based polyol.

The aim of this study is to investigate the effect of the polyol OH number, DMPA content and hard segment content on the stability of the wet jatropha oil-based polyurethane (JPU) dispersion and the physical, mechanical and thermal properties of the dry JPU films. The stability of the dispersion was investigated by particle size analysis, pH and viscosity measurement. On the other hand, the physical properties of the JPU films were determined by pendulum hardness and water contact angle analysis. Tensile test and thermogravimetric analysis (TGA) were used to determine the mechanical and thermal properties of the JPU films. These properties will have an influence on the practical design of products as the PU dispersion can be used as a standalone coating or as a binder in wood and decorative coatings.

2. Materials and methods

2.1. Materials

Crude jatropha oil was supplied by BATC Development Berhad, Kuala Lumpur, Malaysia. The hydrogen peroxide 30% and methanol were supplied by Merck, Germany. Isophrene diisocyanate (IPDI), dimethylol propionic acid (DMPA), n-methyl pyrrolidone (NMP), 2-hydroxyethyl methacrylate (HEMA), phthalic anhydride and dibutyltin dilaurate (DBTDL) were purchased from Sigma–Aldrich. Ethyl methyl ketone (MEK), triethylamine (TEA), formic acid, magnesium sulphate anhydrous, pyridine, and sodium hydroxide were supplied by System. All chemicals were reagent grade, and were used as received.

2.2. Preparation of epoxidized jatropha oil (EJO)

The reactions were carried out in a 1000 mL four neck flask equipped with a mechanical stirrer, condenser, a thermometer sensor and an isobaric funnel. The reaction temperature was controlled by placing the flask in a water bath. Jatropha oil (200 g) and formic acid were poured into the flask and heated to 40 °C, before adding hydrogen peroxide dropwise over a period of 30 min. The molar

ratio of the oil double bond to formic acid and hydrogen peroxide was 1:0.6:1.7. The reaction temperature was then increased and maintain at 60 °C. The reaction was quenched after a prescribed time by cooling the reaction mixture to room temperature. The mixture was then transferred to a 500 mL separating funnel to allow phase separation. The aqueous phase was removed, and the oil layer was washed successfully with distilled water to remove the remaining acid. The oil layer was then dried using magnesium sulphate. Four samples of EJO were prepared by varying the reaction time.

2.3. Preparation of jatropha oil-based polyols (JOL) by the oxirane ring opening method

The reactions were carried out in a four neck flask, as mentioned in Section 2.2. A calculated amount of methanol and water was charged into the flask followed by adding sulphuric acid and heating to 64 °C, and then added with EJO. The reaction was kept at 64 °C for 30 min, and sodium bicarbonate was added to quench the reactions. After being cooled to room temperature, the deposit was discarded. Methanol and water were removed by vacuum distillation at 60 °C for 30 min, followed by 80 °C for 10 min. The resulted clear golden yellow polyol was analyzed for OH number according to ASTM D4274-99 (Test Method C-Reflux Phthalation). By using the same hydroxylation procedure for different EJO, a series of jatropha oil based polyols (JOL) with different OH numbers were prepared and coded as JOL 138, JOL 161, JOL 188 and JOL 217. The numbers represent the OH number of the polyol in mgKOH/g.

2.4. Preparation of jatropha oil-based polyurethane (JPU) dispersion

The JOL and DMPA (dissolved in NMP) were added to a four-necked flask equipped with a mechanical stirrer, nitrogen inlet, condenser, and thermometer. The mixture was heated to 70–80 °C and stirred for 30 min to obtain a homogeneous mixture. IPDI was then added dropwise for 30 min followed by adding a few drops of dibutyltin dilaurate as a catalyst. MEK was added batch by batch to reduce the viscosity of the system. After an additional 3 h of reaction, HEMA was added until the NCO peak at 2270 cm⁻¹ of Fourier transform infrared (FTIR) spectra disappeared, showing that all the diisocyanate had been consumed. The reactants were then cooled to 40 °C and neutralized by adding TEA (1.2 equiv. per DMPA), followed by dispersion at high speed with distilled water to produce the JPU dispersions with a solid content of ~25 wt.% after removal of the MEK under vacuum. The reaction scheme for this method is shown in Fig. 1. The formulation for waterborne JPU dispersion is shown in Table 1. The “JPU 138” designation indicates that the OH number of the polyol used for the JPU preparation is 138 mgKOH/g. For all formulation, the molar ratio between the polyol (JOL), IPDI, DMPA and HEMA was fixed which led to JPUs with an increase in the hard segment and DMPA content. The corresponding JPU films were obtained by casting the JPU dispersions into a Teflon mold, and drying at room temperature for 7 days, followed by drying in a vacuum oven at 60 °C for 12 h. The JPU films were stored in a desiccator at room temperature.

2.5. Characterization

The attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectra were recorded on a Perkin-Elmer Spectrum 2000 spectrometer which collects mid-infrared scattered radiation using a single-beam improved Michelson interferometer. The spectra were recorded in the range of 4000–500 cm⁻¹ with a nominal resolution of 4 cm⁻¹.

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