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EXPERIMENTAL INVESTIGATION OF EPOXY/POLY(AMINO AMIDE)/PHTHALIC ANHYDRIDE:

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International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 EXPERIMENTAL INVESTIGATION OF EPOXY/POLY(AMINO AMIDE)/PHTHALIC ANHYDRIDE:

MECHANICAL PROPERTIES AND THERMAL STABILITY Eduart Wolok1,

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Engineering, State University of Gorontalo, Kota Gorontalo 96128, Indonesia Abstract Epoxy resin is one of materials engineering that is widely applied especially in the shipping industry, aviation, automotive, and others. With a variety of applications led many scientists in the field of materials engineering work to improve the properties and ability of the epoxy either by changing the structure of epoxy. One way to improve the properties of epoxy is adding certain compounds that can react and able to change the properties of the epoxy according to the requirement. In this study, to improve the properties

of the epoxy, then added phthalic anhydride compounds. The epoxy was cured using poly(amino amide) (PAA) as hardener at 40 wt%. The composition of phthalic anhydride (PA) was added at 5, 10, 15 and 20 wt%. The epoxy/PAA/PA was evaluated as the effect of phthalic anhydride composition. The addition of phthalic anhydride increase the elongation at break, energy and impact strength but decreased tensile strength and thermal stability. The optimum of energy and impact strength was at 20wt% phthalic anhydride respectively 0.398 J and 5450,48 J/m2. The addition of phthalic anhydride decrease the thermal properties of epoxy / PAA / PA. Stability of the lowest in the addition of 15wt% PA around 146oC. Keywords: Epoxy, phthalic anhydride, mechanical properties, thermal stability, impact strength INTRODUCTION Epoxy resin has high mechanical properties and hardness, good adhesive, chemical resistance and excellent solvent for a variety of different substrates, thermal

stability and good dimensional 1-4. In its application, the curing agent is

very important in changing the low-molecular of epoxy

oligomers to very high crosslink network and provide high mechanical strength in epoxy. With these properties, epoxy resins are used in a large variety of application areas, including coatings, electronic insulation materials, high-performance composite materials, adhesives, and construction5-7. However, high crosslink system causes the epoxy usually brittle and has limited capability in applications requiring impact strength and high fracture8-11. The type of amine curing agent is one of the basic curing agents for epoxy resin and can be classified into three main categories: aliphatic, aromatic and cyclo-aliphatic amines12-15. The curing agent of amine type reacts with epoxide ring by nucleophilic addition reactions. The tertiary amine is often used as accelerators curing agent for other epoxy resins. Aliphatic amines are widely used to curing epoxy resin for coatings and adhesives because of its low cost and high reactivity. But the traditional aliphatic polyamide has problematic of low boiling point, the poor nature of the surface is smooth, easy absorption of moisture and carbon dioxide, as well as high irritation. These defects have limited thermoset applications for the better in the high-tech industry16,17. During the last years, many studies have been done to reinforce the epoxy resin by adding flexible curing agent. During past few years, various attempts have been made to introduce flexible chains into the epoxy structure such as polysiloxane, polyether, epoxy soybean, and others18,19. Due to incorporate flexible polyether chain to modify the structure of epoxy backbone, crosslinking density of the epoxy resin has decreased, resulting in the decrease in tensile strength and Young's modulus, and improve the impact strength and elongation. One of the epoxy resin curing agents is an anhydride20,21. The anhydride is used as a curing agent for epoxy resin electrical insulating materials. Anhydride requires severer conditions of an amine- based curing agent, but it is suitable for making large-sized molds because it has a long pot life form cured resin which has a relatively good balance of electrical, chemical, and mechanical properties while generating a small amount of heat. Anhydride curing agent has excellent electrical properties, good chemical resistance, and good physical properties. Epoxy resin cured with anhydride compounds generally have better properties than the resin is cured using amines. They are less poisonous, shows a high glass transition temperature, absorbs less water, low exothermic reaction, and their reaction shrinkage22. Curing epoxy with the anhydride of carbonic acid and base catalyst has been studied intensively in many laboratories for a long time. The use of an alkaline catalyst as the curing caused the road of reaction to be selective. The reaction catalyzed by tertiary amine begins with activation of the anhydride with amine23,24. Anhydride compounds generally include phthalic anhydride (PA), 3,4,5,6-tetrahydrophthalic anhydride, methyl tetrahydrophthalic anhydride, hexahydrophthalic anhydride, methyl hexahydrophthalic anhydride, and25,26. The anhydride is not reacted with the epoxy group directly, but anhydride chain will open the first time because it reacted with hydroxyl groups. And then, the carbonyl group of the monoester reacting with epoxy. After the first reaction to occur more quickly, monoester accumulated in the reaction mixture. The author also assumes, in small part, the reaction of an epoxy group with hydroxyl has occurred22. In this study, investigators used phthalic anhydride as a curing agent to be reacted with the epoxide ring of the epoxy resin. Selection of phthalic anhydride because they are cheap and can improve impact properties and elongation at break. The use of phthalic anhydride not done conventionally as has been done in previous studies. At first, phthalic anhydride will be reacted with a secondary amine. The compounds that formed later to be reacted with the epoxy resin to form thermosets. A good combination between epoxy and phthalic anhydride in the presence of poly(amino amide) very promising. Therefore, we wanted to evaluate mechanical properties and thermal stability of combination from epoxy and phthalic anhydride. MATERIALS AND METHODS Materials Epoxy resins type of diglycidyl ether of bisphenol A (DGEBA) (brand name: Eposchon®), EEW = 161,64 g. (equiv.epoxy)-1 as determined by acid titration. Poly(amino amide) (PAA) (brand name: Eposchon®), equivalent weight: 419,036 g.(equiv. active hydrogen)-1. Both the epoxy resin and PAA was purchased from the PT. Justus Kimiaraya, Surabaya. Phthalic anhydride was purchased from PT. Sumber Utama Kimiamurni. Chemical structure of DGEBA, PAA4 and PA can be seen in

figure 1. O CH3 O O CH3 n CH3 O O OH CH3 O Epoxy diglycidyl ether of bisphenol A (DGEBA) resin ISSN: 2005-4238 IJAST Copyright ⓒ 2020 SERSC 1012 International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 H H O H3C H (CH2)11 N CH3 H CH3 H (CH2)2 N CH3 CH3 H CH3 H3C N H CH3 Poly(amino amide) (PAA) O O O Phthalic anhydride (PA) Figure 1. Chemical structure of DGEBA, Poly(amino amide) and Phthalic Anhydride Preparation of epoxy curing system Firstly, PA reacted with PAA by adding 0, 5, 10, 15, and 20wt% PA into 40, 35, 30, 25, and 20wt% PAA in acetone. The mixture was then stirred for ± 5 minutes and left until the cooling process is complete. PA and PAA mixture then added to the epoxy (60wt%), stirred and put in the oven. Heating is carried out gradually beginning from the pre-curing temperature: 50 ° C 2 hours, 1 hour 70 ° C, 100 ° C 1 hour, and the temperature post-curing at 140oC 1 hour27. After curing, thermosetting epoxy then characterized. Characterization of Epoxy/PAA/PA Fourier transforms infrared spectroscopy The Fourier transform infrared spectroscopy (scientific Thermo Nicolet iS10) at room temperature was used to determine the chemical structure of the epoxy which has been prepared both before and after a curing process. FTIR range of 4000-500 cm-1. Scanning

electron microscopy Scanning electron microscopy (SEM) was used to

identify the microstructure of epoxy specimens that have undergone the curing process. SEM testing is done by coating the sample with Au / Pd and analyzed using apparatus XL30 SEM microscope (Phillips). Tensile strength and elongation at break Testing of tensile strength and elongation at break using Universal Autograph / Tensile Testing Machine Shimadzu with Type-

100kNAG SFL. Testing using the standard ASTM D 638 type I. The tests

were conducted at a crosshead speed of 10 mm / min . Impact strength Impact strength was performed using a Charpy impact tester according to ASTM D 6110. The specimen size was 127.0 x 12.7 x 3 mm. Impact strength was tested on a specimen that has been cured. Thermogravimetric analysis Thermogravimetric analysis (TGA) is used to determine the degradation temperature of the specimen has been cured. TGA was performed with a Mettler Toledo TGA with Gas Controller GC 200 under the N2 atmosphere at a heating rate of 10 °C/min from 50oC to 800oC. ISSN: 2005-4238 IJAST Copyright ⓒ 2020 SERSC 1013 International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 RESULTS AND DISCUSSION Curing Mechanism of epoxy/poly(amino amide)/phthalic anhydride Mechanism of curing of epoxy / PAA / PA occurs in two stages. First, the reaction between poly (amino amide) and phthalic anhydride (Figure 2). The reaction begins with the dissolution of the C-O bond in the cyclic chain (C-O-C) of phthalic anhydride in which the electrons will move and cause an excess of electrons on the oxygen becomes O- due to the influence of amine. Anhydride in phthalic anhydride are reactive to the amide groups in the PA and then carbon will lack an electron and becomes C* (C-star). Electron deficient carbon binds to the nitrogen of poly (amino amide) to form amides zwitterion intermediates, R2HN+ - C. This reaction occurs in which the exothermic reaction temperatures can reach 40-

60oC. CH3 CH3 O CH3 O H CH3 O H H O H3C 6 N H

3CH3 CH3 acetone, 27 oC 6 O 3CH3 O + N N H3C N + N N CH3 Phthalic anhydride Poly(amino amide) O: - O Figure 2. Reaction between phthalic anhydride and poly(amino amide) Second, the reaction that occurs between the PAA-PA with an epoxy resin DGEBA type (Figure 3). Oxygen with a charge of negative electrons in PAA-PA will attack epoxide ring (C-O-C) in the epoxy and resulting epoxide ring will open where a covalent bond between the C-O in the chain of cyclic break and the electrons would be attracted to the oxygen more electronegative become O- unstable. O- ionic on PAA-PA would binds to C+ on the open epoxy ring and form a covalent bond. Another epoxide ring in the epoxy resin will also be open because of the influence of amine and binds to the secondary R2NH on PAA. Hydrogen atoms in R2HN+ would break and bind to the O ions in the chain of cyclic epoxides have open. CH3 O H H3C N + 6 O O: -

O PAA-PA CH3 O H H3C N + 6 O O O CH3 H N N 3CH3 CH3 H N N

3CH3 O : - O Bis A H3C CH3 + CH3 O + CH3 O 6 Bis A O DGEBA Bis A O O HO N O O O CH3 CH3 O H3C ? (50, 70, 100, 140) oC 6 O H N + H N N

3CH3 CH3 O O - O: O O Bis A O Bis A - :O CH3 CH3 O H H + H3C N + N N CH3 6 3CH3 O - O O: O Bis A O Bis A CH3 N N CH3 3CH3 OH Bis A O Thermoset of epoxy/PAA/PA Figure 3. Schematic curing reaction mechanism between PAA-PA and epoxy resin ISSN: 2005-4238 IJAST Copyright ⓒ 2020 SERSC 1014 International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 FTIR analysis of thermoset epoxy/PAA/PA The chemical reaction between the epoxy, PAA, and the PA has occurred. It is characterized by changes in the intensity of the peak with functional groups that react as seen in Figure 4. At peak 3380 cm-1 appears -OH group. The addition of PA increases the intensity of peak 3380 cm-1. Peak 3285 cm- 1 indicate the presence of a secondary amine group R2N-H. The addition of PA lowers the intensity of the peak at 3285 cm-1 because of the secondary amine has been bonded to form a tertiary amine. Peak 1735 cm-1 indicate the presence of a carbonyl group C=O, the addition of PA increased the intensity because of presence C=O groups on the compound phthalic anhydride. Peak 1606 and 1507 cm-1 indicates the presence of benzene groups are more. In addition to coming from epoxy, benzene group also came from the PA. Peak 1235 cm-1 indicates the bond H2C-N. Intensities at peak 1235 cm-1 indicate more C-N bonds are formed. Peak 1035 cm-1 indicate the presence of ether groups C-O-C and the addition of the PA increase the intensity. Peak 826 cm-1 indicate the presence of C-H bond. R2N-H C-O-C 240 e O-H C=C C-N C=O CH2 C-H d Transmittance (%) 180 120 c b a 60 1735 3285 1606 1235 3380 3071 1507 1035 826 4000 3500 3000 2500 2000 1500 1000 -1) Wavenumbers (cm Figure 4. FTIR spectra of (a) epoxy/PAA (40%), (b) epoxy/PAA (35%)/PA (5%), (c) epoxy/PAA (30%)/PA (10%), (d) epoxy/PAA (25%)/PA (15%), (e) epoxy/PAA (20%)/PA (20%) Mechanical Properties of epoxy/PAA/PA The mechanical properties of thermoset epoxy are determined by many factors such as molecular weight, the content of the epoxide groups, the number and type of curing agent used, curing temperature, and others. The mechanical properties of thermoset epoxy / PAA / PA can be seen in Table 1. ISSN: 2005-4238 IJAST Copyright ⓒ 2020 SERSC 1015 International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 Table 1. Effect of the addition of phthalic anhydride to the mechanical properties of thermoset epoxy Phthalic anhydride, PA (%) Tensile strength (MPa) Elongation at break (%) Impact energy (J) Impact strength (J/m2) 0 54,39 4,58 0,108 2358 5 19,61 8,04 0,119 2408 10 8,42 16,96 0,086 1774 15 7,92 21,55 0,280 3514 20 4,98 12,13 0,398 5451 From Table 1 we can see a decrease tensile strength. The main cause of the decrease in the tensile strength of thermoset epoxy / PAA / PA is the presence of a solvent that is still stuck in the specimen and the formation of gelatin phase. Although in theory with the addition of the PA, it will form a crosslinked network in the material, but the addition of acetone which does not bind to the primary material causes the solvent will only reduce the viscosity (concentration of the mixture decreases) and the density of the specimens that have been curing becomes low. Increased curing temperatures above the boiling point of the solvent cause the solvent is driven off in the gas phase, either in the form of acetone gas or other gas (CO or O2) because of acetone has suffered structural damage. Solvents in the gasses coming out phase cause the formation porosity which is one factor of inheritance of a material. Formation of gelatin phase also decreases the tensile strength because of as already mentioned, gelatin phase is the phase in which the material will form a liquid polymer without bond crosslink with other polymers that have been cured28. Gelatin is more like a precipitate formed and disrupt the crosslink materials. 2,0 1,913 1,8 1,6 Swell ratio (w/w) 1,4 1,387 1,2 1,0 1,093 0,8 0,804 0,821 0,6 0 5 10 15 20 Phthalic anhydride (%) Figure 5. The effect of adding phthalic anhydride to swell ratio of thermoset epoxy/PAA/PA The addition of PA initially increase the elongation at break, but the addition of 20wt% PA decreased. In theory, the increase in elongation at break due to more of PA solution is added, the acetone entry is also growing. Solvents are increasingly causing a low concentration and density of the thermoset. Due to the influence of solvent trapped, the atoms in each molecule will be susceptible to vibrations of the atoms. Vibration atom causes elongation at break to be great. The presence of the solvent among crosslink network also facilitates the movement of molecules that will cause the material to become more resilient. In addition 20wt% PA there is a decrease elongation at break ISSN: 2005-4238 IJAST

Copyright ⓒ 2020 SERSC 1016 International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 properties. This is because the addition of acetone is the most causing movement of the atoms become irregular and the number who have become slightly crosslink network as evidenced by the increasing swell ratio of thermoset with the addition of PA (Figure 5). Increased vibrations of atoms also increase the strength and impact energy of thermoset epoxy. Scanning Electron Microscopy of Epoxy/PAA/PA SEM testing is also carried out on material thermoset epoxy / PAA with the addition of 0, 10, and 15wt% PA to view the microstructure and bonding models formed microscopically (Figure 6). Figure 6. SEM micrograph of the cross section fracture surface from thermoset epoxy/PAA (40%) (a and b), thermoset epox/PAA (30%)/PA (10%) (c and d) and thermoset epoxy/PAA (25%) (e and f) From Figure 6 a and b, we can see a smooth thermoset surface with presence little spots. The smooth white surface indicates there has been a perfect curing of thermoset epoxy / PAA. The spots that appeared is the porosity of trapped air or solvent. Figure 6 c, d, e, and f show porosity because of air ISSN: 2005-4238 IJAST Copyright ⓒ 2020 SERSC 1017 International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 trapped in the thermoset epoxy / PAA / PA. In addition, the solvent was stuck and breaking of covalent bonding forming N2 and O2 gasses can also influence the porosity in thermoset material when heated to the curing temperature. Thermal Stability of epoxy/PAA/PA Thermal stability of thermoset epoxy / PAA / PA who has been curing can be seen in Figure 6 and Table 2 generally indicates changes in temperature of polymer degradation thermoset epoxy / PAA / PA due to the addition of the PA. 100 90 80 70 Weight (%) 60 50 40 30 20 10 0 5% 10% 15% 0% 20% 100 200 300 400 500 600 700 800 Temperature (oC) Figure 7. TG curve of epoxy/PAA/PA with a heating rate of 10oC/min Table 2. Thermal properties of epoxy/poly(amino amide)/phthalic anhydride Phthalic anhydride Temperature (oC) at varied weight loss fractions Ash content (%) (%) 5% 10% 30% 50% 70% 800oC 0 313,21 338,44 372,04 399,99 429,72 4,271 5 301,87 340,02 373,07 399,77 429,47 4,817 10 263,20 332,60 370,22 398,40 429,74 6,764 15 146,00 316,85 366,30 395,18 429,46 6,677 20 164,57 324,15 368,26 394,30 425,71 4,023 From Table 2 and Figure 7 can be concluded that a decline in temperature degradation when adding PA. In other words, the addition of PA decreases the thermal stability of thermoset epoxy / PAA / PA. From the results of TGA, the minimum Td was in the addition of 15wt% PA which is about 146oC. If we compare the results of TGA with and without the addition wt% PA, we can conclude that the addition of PA solution further reduces the thermal stability of thermoset epoxy / PAA / PA. The main cause of the decrease in the thermal stability of thermoset epoxy / PAA / PA namely the presence of a solvent which is trapped in a specimen that has been curing and form a gelatinous phase which does not bind crosslink with thermoset epoxy / PAA / PA. Solvents, air or gelatin have lower stability compared to thermoset polymers and elastomers. CONCLUSIONS Mechanical properties and thermal stability of thermoset epoxy / PAA with the addition of phthalic anhydride were investigated. The results of this study indicate the reaction between the amine group ISSN: 2005-4238 IJAST Copyright ⓒ 2020 SERSC 1018 International Journal of Advanced Science and Technology Vol. 29, No. 5s, (2020), pp. 1011-1021 secondary (R2NH) on poly (amino amide) with cyclic carbon chain on phthalic anhydride (C-O-C). The reaction continued when added the epoxy in which the reaction occurs between epoxide ring with unstable oxygen (O-) on phthalic anhydride and epoxide ring with secondary amines on the poly (amino amide). The addition of PA to the epoxy / PAA decrease tensile strength, but increase the elongation at break, energy and impact strength due to the influence of vibration in the atom. The thermal stability has decreased.

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