INTISARI

Pemungutan asam tereftalat (TA) dari limbah cair proses *finishing* kain poliester merupakan salah satu upaya pengelolaan dengan memanfaatkan kembali limbah menjadi bahan baku industri. Penelitian ini bertujuan untuk memungut dan memurnikan TA dari limbah cair proses pengurangan berat dengan cara kristalisasi serta karakterisasi kristal TA yang terbentuk dari kedua metode tersebut. Kristal TA juga diuji reaktivitasnya dengan resin epoksi berupa Di Glycidil Ether Bisphenol A (DGEBA).

TA kasar dipungut dengan menambahkan asam ke dalam limbah cair. TA kasar dimurnikan dengan dua metode yaitu kristalisasi reaktif dan kristalisasi pendinginan. Pada kristalisasi reaktif, TA kasar direaksikan dengan larutan NaOH menjadi larutan Na_2TP yang kemudian diasamkan dengan H_2SO_4 sampai mencapai pH target. Dilanjutkan dengan penambahan larutan H_2SO_4 dan larutan sekunder Na_2TP dengan konsentrasi yang sama pada pH tetap. Kristal TA yang terbentuk kemudian dipisahkan dan dikeringkan. Dipelajari pengaruh waktu, pH, konsentrasi larutan sekunder, kecepatan volume reaktan, suhu kristalisasi dan kecepatan pengadukan terhadap distribusi ukuran dan massa kristal TA.

Pemurnian dengan kristalisasi pendinginan dilakukan dengan melarutkan TA kasar dalam Dimethyl Acetamide (DMAc) pada suhu 90°C sampai jenuh, kemudian larutan jenuh difiltrasi pada suhu tetap. Filtrat jenuh kemudian diturunkan suhunya sampai 70°C dengan pendinginan alami, kemudian suhu dijaga tetap dengan menggunakan *waterbath* selama dua jam, selanjutnya difiltrasi. Kristal yang terbentuk dikeringkan, sedangkan filtrat kemudian diproses kembali seperti langkah sebelumnya yaitu diturunkan suhunya sebesar 10 ˚C kemudian suhu dijaga tetap konstan selama dua jam. Proses tersebut diulangi kembali sampai suhu pendinginan mencapai 30°C. Distribusi ukuran dan massa kristal TA digunakan untuk mengevaluasi pengaruh metode pendinginan dan kemurnian kristal pada proses ini.

Hasil percobaan menunjukkan bahwa kristalisasi reaktif terjadi sangat cepat. Variabel waktu, pH, kecepatan volume reaktan, suhu, dan laju pengadukan tidak berpengaruh nyata terhadap ukuran kristal rerata. Sementara itu, konsentrasi reaktan berpengaruh positif terhadap ukuran kristal TA. Rata-rata ukuran kristal pada pada konsentrasi 0,5, 0,3 dan 0,1 M masing-masing adalah 7,57, 3,24 dan 3,09 μm. Hasil penelitian menunjukkan bahwa kristalisasi reaktif cenderung menghasilkan kristal dengan ukuran yang halus. Hal ini disebabkan oleh laju nukleasi yang tinggi dibandingkan dengan pertumbuhan. Hasil kristalisasi pendinginan menunjukkan bahwa ukuran kristal dipengaruhi oleh suhu pendinginan, dimana semakin tinggi suhu yang dijaga konstan maka ukuran kristal akan semakin besar. Kemurnian TA juga mempengaruhi rendemen TA, TA murni (PTA) menghasilkan nilai rendemen 80,26%, sedangkan TA rekristalisasi (TA-Re) dan TA mentah (CTA) menghasilkan nilai rendemen 70,30 dan 61,52 %. Ukuran kristal TA rata-rata dari PTA, TA-Re, dan CTA masing-masing adalah 22,55, 21,95, 21,13μm.

Kristal TA hasil kristalisasi reaktif dan pendinginan sebagian besar telah memenuhi syarat mutu SNI 06-2153-1991 tentang asam tereftalat murni (PTA), sedangkan metode kristalisasi reaktif masih melebihi syarat mutu untuk parameter kadar abu dan 4-Karboksi benzal-dehida (4-CBA). Metode pemurnian kristalisasi reaktif dan kristalisasi pendinginan memberikan perbedaan morfologi, ukuran kristal, titik lebur dan rapat curah. Di sisi lain, metode pemurnian tidak memberikan pengaruh yang signifikan terhadap reaktivitas terhadap resin epoksi berupa Di Glycidyl Ether Bisphenol A (DGEBA).

ABSTRACT

The recovery of terephthalic acid (TA) from polyester fabric finishing wastewater is one of the chemical treatments of wastewater as well as the utilization recovered TA into an industrial raw material. This study is aimed to recover and purify of TA by the crystallization methods (reactive and cooling crystallization) and the characterization of TA obtained from both methods. Reactivity of TA crystals were conducted by react TA with epoxy resin of Di Glycidyl Ether Bisphenol A (DGEBA).

The crude TA is recovered by adding acid to the wastewater. The TA purification was carried out by two methods i.e., the reactive crystallization and the cooling crystallization. In a reactive crystallization, the crude TA was reacted with sodium hydroxide solution to form a salt of disodium terephthalate solution, then acidified with sulfuric acid until it reaches the targeted pH solution. An equimolar secondary solutions of 0.5 M disodium terephthalate and 0.5 M sulfuric acid were then added continuously to the reactor. The TA crystal formed then separated and oven-dried at 70°C. In the reactive crystallization, the effect of time, pH, concentration and volumetric feeding rate of secondary solution, temperature and stirring speed on the size and mass distribution of TA crystals were studied. The cooling crystallization used in this experiment was an unseeded multi-stage cooling crystallization. The saturated TA in dimethyl acetamide (DMAc) at 90˚C was cooled in multi-stages where each stage consisted of lowering the temperature (cooling) followed by holding the temperature constant for 2 hours. The crystals were then separated and dried in an oven at 100˚C. Meanwhile, the mother liquor was cooled down as the same as the previous stage where at each stage the temperature was lowered 10°C followed by a constant cooling at a temperature of 60°C for 2 hours. The process was repeated until the cooling temperature reached 30°C. The effect of cooling temperature and purity of TA on the size and mass distribution of TA crystals were studied.

The experimental results showed that the reaction and crystallization of TA occurs very quickly. The time, solution pH, volumetric velocity of reactants, temperature, and stirring rate had no significant effect on crystal size. The concentration of reactants have a positive effect on the crystal size of TA. The mean size of crystals concentrations of 0.5, 0.3 and 0.1 M were 7.57, 3.24 and 3.09 μm respectively. The result showed that reactive crystallization tends to produce crystals with a fine size, which is caused by the high nucleation rate compared to growth. The results of cooling crystallization showed that the crystal size was affected by a cooling temperature, where the higher the isothermal temperature was, the larger the crystal size would be. The purity of TA affected the yield of TA, the purified TA (PTA) resulted in a yield value of 80.26%, while the recrystallized TA (TA-Re) and the crude TA (CTA) resulted in a yield value of 70.30 and 61.52%, respectively. The average TA crystal sizes from PTA, TA-Re, and CTA were 22.55, 21.95, 21,13 μm, respectively.

Most of the quality requirements of Indonesian Standard of PTA (SNI 06-2153-1991) have been fulfilled by purified TA by cooling and reactive crystallization, but the TA purified by reactive crystallization still exceed the quality requirements for the ash content, and 4-Carboxybenzaldehide (4-CBA). The TA purified by reactive crystallization and cooling crystallization have differences in morphology, crystal size, melting points and bulk density. On the other hand, the purification methods did not have a significant effect on the reactivity of TA when reacted with epoxy resin in the form of DGEBA.