

PRESSURIZED HOT WATER EXTRACTION OF BENZOIC ACID AND PHTHALIC ANHYDRIDE FROM PETROCHEMICAL WASTES USING A MODIFIED SUPERCRITICAL FLUID EXTRACTOR AND FACTORIAL DESIGN FOR OPTIMIZATION

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Using pressurized fluid extraction (PFE) technique, benzoic acid and phthalic anhydride were removed from solid petrochemical wastes (i.e. called molten phthalic anhydride, MPA) of Farabi Petrochemical Co. in Iran. More than 65 ton phthalic anhydride and 9.5 ton benzoic acid are lost per year just in this company [1]. The supercritical fluid extractor was modified to be able to pump liquid solvent and CO₂ into the extraction vessel alternatively; PFE is an efficient, rapid, selective, and reliable extraction method [2].

Using a modified supercritical fluid extraction (SFE) apparatus, we have developed a PFE method mainly as an analytical tool to determine phthalic anhydride and benzoic acid content or as a sample preparation method for the phthalic anhydride and benzoic acid quality measurements with full capability of automation when the work load is high [3].

In this study we applied a statistical experimental design [4] based on "Central Composite Design" to evaluate optimized conditions for continuous extraction of phthalic anhydride and benzoic acid from a mixture of benzoic acid, maleic acid, phthalic acid, and phthalic anhydride. Water and water-ethanol mixture (ethanol as a modifier of water) were used for PFE because of its availability, non toxicity, lower cost, and good selectivity of ethanol in dissolving phthalic anhydride and benzoic acid in comparison with other solvents such as CH₃OH, acetonitril, butyl acetate, toluene, and cyclohexane. Parameters such as pressure (60-220) bar, temperature (333-373) K, dynamic time (5-45) min, flow rate (0.2-1) mL/min, and volumetric percent of ethanol in water (0.05-0.25 v/v) coded as x₁, x₂, x₃, x₄ and x₅ respectively, were used. The parameter x₅ was only used in the presence of ethanol as modifier. These parameters were investigated in five levels (-2,-1, 0, 1 and 2). The dependent variables Y₁ and Y₂ were taken as the relative overall benzoic acid yield and phthalic anhydride yield, respectively. Data were analyzed in terms of selectivity, extraction yield for phthalic anhydride and benzoic acid. Results shows that the PFE method is more selective to phthalic anhydride rather than benzoic acid. As phthalic anhydride is hydrolyzed to ortho-phthalic acid by hot water [5] and as a result the system is blocked, we did not use high temperatures. Finally, extracted samples after removal of its solvent were dissolved in acetonitril and analyzed with GC-FID.

References:

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