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A new method for isolating pyromellitic dianhydride

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Abstract

Pyromellitic dianhydride of high purity is now one of the most popular products of organic chemistry and is widely used in the production of heat-resistant polyimides, alkyd resins, effective plasticizers, as a hardener of epoxy resins, in the paint industry, etc. The development of highly efficient methods for isolating pyromellitic acid dianhydride is a topical task with special attention given to its purification.

A new method for isolating pyromellitic acid dianhydride is proposed to yield the desired product of high purity, which meets the necessary quality requirements of consumer organizations. With this purification method, the optimal composition and solvent mixture ratio have been found, by which we have succeeded to reduce the content of impurities in the target pyromellitic dianhydride. It is indicative of the effectiveness of the proposed method of pyromellitic dianhydride isolation to produce raw materials for polyimide production.

The availability of raw materials, small waste, simple facility design, as well as the low level of material and energy costs in product separation and solvent recovery are advantages of the proposed method of purifying pyromellitic dianhydride. A schematic process flow diagram of the purification of pyromellitic dianhydride is provided, comprising the stages of treatment of raw pyromellitic dianhydride with a binary solvent mixture, filtering the complex of pyromellitic dianhydride with the solvents, and decomposition of the complex to give the targeted pyromellitic dianhydride.

The composition of the isolated product was characterized by gas chromatography–mass spectrometry, photometry, and titrimetry. The content of benzocarboxylic acids in the obtained sample of pyromellitic dianhydride was controlled using gas chromatography–mass spectrometry; titrimetry and photometry were applied for quantitative analysis of pyromellitic acid and coloring impurities, respectively.

Our designed method for pyromellitic dianhydride isolation is promising not only for preparative applications but also for industrial ones and enables isolating product of the required quality with the basic substance content at least 99.7%, suitable for obtaining polyimide resins with high strength, durability, heat resistance, and elasticity.

References

[1] Lu Ming, Hu Yu Lin, Liu Xiao Bin, Zhang Sheng Bin, Ji Zhan Hui, and Lu Ting Ting. An inexpensive and efficient synthetic method for the preparation of pyromellitic dianhyride in ionic liquid. *AKRIVOC*. **2010**. Vol.IX. P.63-74.

[2] Henry Lee, Donald Staffey, and Kris Neville. New linear polymers. *McGraw-Hill, New York.* **1967**. 374p.

[3] M.I. Farberov, G.S. Mironov, N.V. Pavelko, V.D. Shein, A.V. Bondarenko, I.V. Budniy, and O.S. Kozlov. Technical synthesis of pyromellitic dianhydride based on chloromethylation of xylenes. *Chemical Industry*. **1968**. Vol.44. No.8. P.3-7. (russian)

[4] E.P. Babin, V.I. Lozovoy, K.B. Pan'kovskays, N.A. Goryunov, and N.I. Danilova. Xylenes as raw materials for the synthesis of pyromellitic dianhydride. *Journal of Applied Chemistry*. **1973**. Vol.46. No.3. P.598-603. (russian)

[5] *Pat.* DE1768881 *Germany*. Verfahren zur reinigung von durch gasphasenoxidation von 1,2,4,5-tetraalkylierten benzolen gewonnenem pyromellitsaeuredianhydrid. *Priority of* 13.01.**1972**.

[6] *Pat*. 2314301 *Russia*. A method of producing and purifying pyromellitic dianhydride. *Priority of* 10.01.**2008.** Bulletin No.1.

[7] *Pat*. 2412178 *Russia*. A process for preparing intramolecular anhydrides of benzopolycarboxylic acids. *Priority of* 20.02.**2011.** Bulletin No.5.
[8] *Pat*. 2567397 *Russia*. A process for preparing 2,4,6-trimethylbenzoic (durylic) acid. *Priority of* 11.10.**2015**. Bulletin No.31.
[9] S.S. Zakharov, V.A. Belikov, T.V. Chelyuskina, and L.V. Kaabak. Thermochemical study of the process of obtaining 2,4,6-trimethylbenzoic acid. *Theoretical Foundations of Chemical Engineering.* **2015**. Vol.49. No.3. P.355-360. (russian)