The study by spectrometric methods of the azeotropes formed at the distillation of phenolic water

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Abstract. We combined the colorimetric method using 4-amino-antipirine with the GC-MS method, for the study of the azeotropes formed by the phenolic compounds (o, m, p cresols plus phenol) with water. The GC-MS method was optimized for the determination of the phenolic compounds from complex matrices. For the study of azeotropes, a distillation column with a rectification zone of 1 m height was used. The raw was a phenolic wastewater which was in view to be concentrated by distillation. The samples of distillate were analyzed during the distillation process in order to observe the change in composition, the point of the next azeotrope forming. The study of the azeotropes wasn't possible by observing the change in distillation temperature (the TBP curve) since all the azeotropes of phenolic compounds with water formed at approx. 99.5 $^{\circ}$ C.

The conclusions of the study were: i) the azeotropes forming is highly influenced by the presence of inorganic salts and hydrocarbon in the solution, the content in phenolic compounds being much below the concentration indicated in the literature data (in absence of impurifying substances), (ii) the azeotropes are multiple (o, m, p cresols plus phenol), the predominance of compounds being in order: m + p cresols, then o- cresol, then the phenol and in the end only the binary azeotrope phenol-water was detected; (iii) the volume of distillate containing phenolic compounds represents cca. 47% of the raw; the bottom of the column is free of them; (iv) one can use these data in order to size an industrial distillation column for the recovery of the phenols from the industrial waste.

Keywords: phenolic compounds - water azeotropes, GC-MS