

## References

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## Spectroscopic Methods Applied to the Characterization of Phenol Oxidation Intermediates

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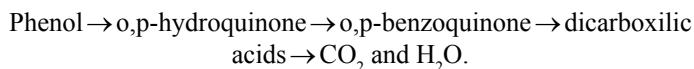
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Phenols and substituted phenols are important raw materials for wide variety of organic compounds, dyes, pharmaceuticals, plasticizers, antioxidants, etc. Phenols are present effluent from the chemical process industries which are either manufacturing or using them.

Combined oxidation of phenolic wastes offers an alternative treatment method to destroy phenolics totally. It consist, in essence, of the electrochemical generation of high-reactivity intermediates, and on either a cathode or an anode (or, possibly, on both the cathode and the anode simultaneously) and their subsequent chemical decay of phenol in an electrolytic solution. In this work oxidizing agent is generated in aqueous sulfuric acidic solutions under direct current. It has been identified that the maximum rate of phenol cleavage achieved with the sulfuric acid concentration of 30%. The assumed scheme of phenol cleavage by electrogenerated oxidizing system is a typical oxidation process of phenol in the liquid phase [1].



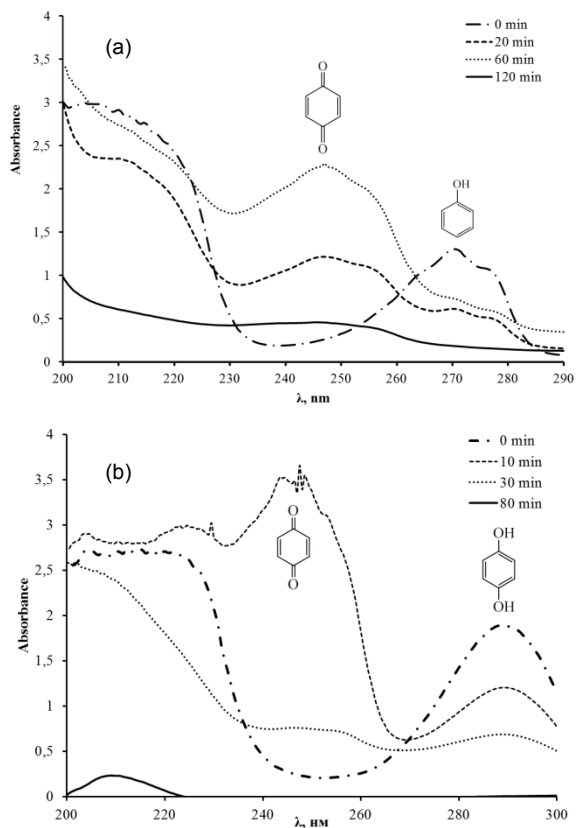
We carried out the identification of intermediates and final products by means of UV spectrophotometry, gas chromatography with mass selective detector and high performance liquid chromatography.

For UV spectrophotometry a samples were prepared by following procedure: 0.1 ml of a solution from electrolyzer diluted to 1 ml with distilled water and then record the spectrum using quartz cell.

A sample preparation for gas chromatography with mass selective detec-

tor is following: 0.2 ml of reaction mixture was placed into a measuring tube, diluted with distilled water to 10 ml. Then the  $\text{BaCl}_2$  was added to precipitate sulfate ions as  $\text{BaSO}_4$ . And further organic matter was extracted by 10 ml ethyl acetate. As an ethyl acetate has the highest extracting ability for phenol and oxidation intermediates.

Phenol has two absorption peaks at 210 and 270 nm, moreover at 210 nm oxygen-containing compounds absorb as well (fig. 1 (a)). Therefore the absorbance at 210 nm is the most intensively. The absorbance peak at 245 nm can be determined as characteristic of p-benzoquinone [3]. During the first hour of electrolyze the concentration of p-benzoquinone is increased and achieved the own maximum, and phenol concentration is decreased. Also the hydroquinone oxidizes through p-benzoquinone as well (see fig. 1 (b))



**Fig. 1.** UV spectra of phenol (a) and hydroquinone (b) acidic aqueous solution during the electrolysis (initial phenol concentration 1 g/l)

However, the data of gas chromatograph with mass spectroscopy show that the oxidation products of phenol are hydroquinone, butyric acid, butenoic acid, and preferably acetic acid. And the presence of p-benzoquinone was not observed. It was found out only p-hydroquinone.

To confirm the results of gas chromatography with mass selective detector we analyzed our samples by high performance liquid chromatography without extraction. Sulfuric acid was neutralized by solution of ammonium hydroxide. Benzoquinone was not observed too. It was identified p-hydroquinone (absorption at 290 nm) and carboxylic acids.

Thus, according to UV-spectroscopy the phenol oxidation intermediates are p-benzoquinone and carboxylic acids, and gas chromatograph with mass spectroscopy determines the p-hydroquinone without p-benzoquinone.

### References

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## Titanium powder segregation from electrolyte precipitate

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This article is a continuation of the work about fluoridation and electrolysis of titanium concentrates. It is dedicated to the development of one of the most important stages of the proposed fluoride technology of titanium – the process of washing the cathode deposit formed during the electrolytic separation of titanium fluoride melts from the electrolyte salts

As a result of titanium powder electrolytic extraction of fluoride salt melt, cathode precipitate containing 42.50% Ti and impurities are formed: LiF, KF, NaF and complex titanium salts such as  $Me_2TiO_3$ , where Me=Na, K, Li, adsorbing on the obtained product during the electrolysis process. There