

**PREPARATION, CHARACTERISATION AND
SOME POTENTIAL WATER TREATMENT
APPLICATIONS OF ACTIVATED CARBON
PRODUCED FROM JORDANIAN OLIVE STONES**

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Abstract

This work was carried out at Department of Chemistry/School of Science and the Environment. Although it is not easy nowadays to separate between different parts of chemistry, sciences and applied sciences; however, this work is essentially based on chemical basics (analytical and physical), in which chemical methods were used for producing material (activated carbon and activated carbon modified with titanium dioxide) suitable for treatment of water samples polluted with chlorinated phenols. Additionally, chemical methods were used in the water treatment process and analytical chemical methods were modified and used for analysis of the activated carbon surface and analysis of the water samples during the treatment process.

This work can be seen from an analytical chemistry point of view, in which some analytical techniques were modified to study properties of the treated water samples, such as the one dealing with analysis of chlorinated phenols (typical water pollutants) by gas chromatograph, in addition to analysis of chloride ion concentration and total organic carbon for the treated water samples. Additionally, various analytical techniques were involved for studying the surface properties of the activated carbon and activated carbon modified with titanium dioxide, such as metal analysis by inductively coupled plasma and x-ray fluorescence, thermal gravimetric analysis, Boehm titrations, infrared spectroscopy, x-ray diffraction, nitrogen adsorption and methylene blue adsorption/spectrophotometric analysis. Additionally this work can be seen from a physical chemistry point of view in which water treatment techniques involved adsorption and degradation of the pollutants. On the other hand, there is an environmental importance for this work, since it is dealing with water treatment and solid waste disposal problems.

The yearly production of olives in Jordan is more than 100 kilo ton of which a significant proportion is de-stoned prior to sale. In this work, a single variety of Jordanian olive stones were used for the preparation of activated carbon with the aim of producing a water treatment product suitable for treatment of contaminated groundwater. The preparation conditions were varied to study their effects on the surface area, porosity, morphology, functionality and crystal structure. Variables studied included time of carbonisation, time of activation, activating agent, particle size, sample pre-drying and the effect of the activation process itself. Further treatment of activated carbon included oxidation with hydrogen peroxide, oxidation with nitric acid or de-ashing using HCl/HF. X-ray diffraction analysis showed that the prepared activated carbon is essentially amorphous and indicated the presence of traces of oxides of calcium and magnesium, while infrared spectroscopy showed peaks relating to hydroxyl, aliphatic, ether, aromatic and phenolic groups. Nitrogen

adsorption experiments showed that the active carbon is mainly microporous and of moderate surface area. Scanning electron microscopy showed the development of the pores during different treatments. The most noticeable effect on the texture was that when the sample was not pre-dried. The adsorption capacity and surface acidity/basicity of all the samples have been studied by methylene blue (MB) adsorption method and Boehm titrations, respectively. Results showed that the surface is mainly of basic nature, and also indicated that methylene blue was adsorbed following a pore filling mechanism. The properties of the produced carbons were considered normal properties. Thus it was considered useful to follow the stepwise carbonisation of the olive stones, in which various properties were followed, including mass loss; appearance-disappearance of any crystalline matter and functional groups, and elemental analysis. This was in addition to studies of the texture and relative surface area by scanning electron microscopy and methylene blue adsorption, respectively. It was found that carbonisation is almost complete at 600°C. The results of this part of the work may help in the future development of the carbonisation process, or it may help to obtain a product of specific properties for specific applications.

In an attempt to modify the properties of the produced activated carbon, titanium dioxide was deposited on its surface to produce a potential photocatalyst capable of degradation of 2-chlorophenol. Three different techniques were used for the deposition process: chemical vapour deposition (CVD), direct air-hydrolysis (DAH) and high-temperature impregnation (HTI) techniques. The total TiO₂ deposited and amount crystallized into anatase form by each technique was estimated. The effect of each deposition procedure on surface area, porosity and thermal stability of the sample was studied by nitrogen adsorption, mercury intrusion, methylene blue adsorption and thermal gravimetric analysis. Surface texture of the potential catalysts was studied by electron microscopic techniques. Results showed that different procedures gave different yields of total and anatase TiO₂. On the other hand, there was significant reduction, which may be due to TiO₂ deposition, in porosity and surface area. The reduction in MB adsorption was proportional to anatase TiO₂ but not to the total TiO₂ deposited. Chemical vapour deposition gave more anatase TiO₂ than the direct air-hydrolysis technique, which is thought to form the deposit within the micro-meso pores, and caused more porosity-surface area reduction. It is thought that TiO₂ particles inside the pores have more opportunity to crystallize into the anatase form. The deposited layer of TiO₂ apparently catalyzed slight weight loss at low temperatures but generally gave more thermal stability for the support. Non-activated carbon (C) was also used as a support and its thermal stability, porosity

and surface area were not affected by TiO_2 deposition. The deposition, in non-activated carbon, is thought to occur on external surface, which may be due to absence of large micropores.

Adsorption of 2-chlorophenol on the prepared potential photocatalysts was studied dynamically and statically (equilibrium). The potential photocatalysts failed in producing any photocatalytic activity probably due to strong adsorption of the pollutant on the catalyst surface. Thus, it was decided to concentrate on the adsorption of various chlorinated phenols on activated carbon surface. The prepared activated carbon samples were used for adsorption of various chlorinated phenols, viz. 2-chlorophenol (2-CP), 3-chlorophenol (3-C), 4-chlorophenol (4-CP), 2,4-dichlorophenol (2,4-DCP) and 2,4,6-trichlorophenol (TCP). Effect of activated carbon properties, pH of the solution, number and position of chlorine atoms on the adsorption process was investigated. Special attention was given to a sample prepared by fast heating rate pyrolysis, which was expected to give better properties. Commercial samples were also included. Langmuir and Freundlich adsorption isotherms were applied to the adsorption data and adsorption constants were calculated. The aim of this process was to determine if there are any relationships exist between the properties of the prepared activated carbons and the adsorption constants of Langmuir and Freundlich. These properties included burn-off, apparent surface area, micropore volume, total pore volume and methylene blue (MB) adsorption surface area. Total basicity had no relationship with any adsorption parameter. However, total basicity was found to have a relationship with total pore volume, micropore volume, MB adsorption and burn-off. BET surface area showed only a very weak relationship with total basicity. Burn-off does have a relationship with the relative affinity of adsorbate and the surface and Langmuir constants, but not the capacity of adsorption. De-ashing the sample using HF / HCl decreased the total basicity and MB adsorption but increased the total acidity and the capacity for 2-CP uptake.

The activated carbons produced appear to have attractive properties for the sorption of acidic pollutants but do not appear to make a suitable photocatalyst support because of interferences in the catalytic effect caused by strong sorption of acidic pollutants.