http://www.doi.org/10.62341/istj-vol38-2-ah60

Received	**2026/06/05**	**تم استلام الورقة العلمية في**
Accepted	**2026/06/28**	**تم قبول الورقة العلمية في**
Published	**2026/06/30**	**تم نشر الورقة العلمية في**

Preparation of Activated Carbon from Olive Mill Solid Waste for Water Treatment Use

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Abstract
Production processes of olive oil usually carry massive amounts of solid waste. In fact, Libya is one of the countries that has a big number of olive trees. It is estimated to have 10–12 million olive trees and Average productivity is reported at around 20 kg of olives per tree per year under favorable conditions. leading to many wastes generated from oil production. This process can produce considerable amounts of olive mill solid waste which can be recycled under special processes for water treatment. Therefore; this experimental study aims to prepare Activated Carbon (AC) from olive oil cake as a cheap natural source. The method conducted for producing this activated carbon was a combination between physical and chemical processes. The solid olive oil mill residue was carbonized at 250^0C and chemically activated using two methods including Model I (25% KOH) and Model II (25% KOH + 25% H_3PO_4) in order to make a comparison between them. To investigate the efficiency of prepared AC, removal of phenol found in an aqueous solution was carried out. The effectiveness of this process was studied via key parameters effects such as contact time, pH, and AC dose. The achieved results showed that the degradation using Model II was more than that of Model I. At optimum conditions the maximum phenol removal was 80%. The results indicate that olive-waste cake has a potential in future water treatment applications due to its high adsorption capacity.

Keywords: Olive oil solid waste, activated carbon, phenol

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تحضير الكربون المنشط من المخلفات الصلبة لمعاصر الزيتون استخدامه في معالجة المياه

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الملخص:
تنتج عمليات تصنيع زيت الزيتون كميات كبيرة من المخلفات الصلبة. وتُعد ليبيا من الدول التي تمتلك أعدادًا كبيرة من أشجار الزيتون، حيث يُقدَّر عددها بنحو 10–12 مليون شجرة، ويبلغ متوسط إنتاجية الشجرة الواحدة حوالي 20 كيلوجرامًا من ثمار الزيتون سنويًا في ظل الظروف الملائمة، مما يؤدي إلى توليد كميات كبيرة من المخلفات الناتجة عن صناعة الزيت. وتنتج هذه العمليات كميات معتبرة من المخلفات الصلبة لمعاصر الزيتون التي يمكن إعادة تدويرها ومعالجتها بطرق خاصة لاستخدامها في معالجة المياه .لذلك، تهدف هذه الدراسة التجريبية إلى تحضير الكربون المنشط (Activated Carbon, AC) من كعكة الزيتون باعتبارها مصدرًا طبيعيًا منخفض التكلفة. وقد تم إنتاج الكربون المنشط باستخدام مزيج من التنشيط الفيزيائي والكيميائي. حيث جرى تفحيم المخلفات الصلبة لمعاصر الزيتون عند درجة حرارة 250 °م، ثم تنشيطها كيميائيًا باستخدام طريقتين مختلفتين: النموذج الأول (25% هيدروكسيد البوتاسيوم KOH) ، والنموذج الثاني (25% هيدروكسيد البوتاسيوم KOH + 25% حمض الفوسفوريك H_3PO_4) ، وذلك بهدف إجراء مقارنة بينهما .ولتقييم كفاءة الكربون المنشط المحضَّر ، تم إجراء تجارب لإزالة الفينول من محلول مائي. كما تمت دراسة فعالية عملية الامتزاز من خلال تقييم تأثير عدد من العوامل الرئيسية، مثل زمن التلامس، والرقم الهيدروجيني (pH) ، وجرعة الكربون المنشط المستخدمة .أظهرت النتائج المتحصل عليها أن كفاءة الإزالة باستخدام النموذج الثاني كانت أعلى من تلك المحققة باستخدام النموذج الأول. وعند الظروف التشغيلية المثلى، بلغت أعلى نسبة لإزالة الفينول حوالي 80%. وتشير هذه النتائج إلى أن كعكة مخلفات

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الزيتون تمتلك إمكانات واعدة في تطبيقات معالجة المياه المستقبلية نظرًا لقدرتها العالية على الامتزاز.

الكلمات المفتاحية: النفايات الصلبة لزيت الزيتون، الكربون المنشط، الفينول

1. Introduction

In fact, Libya is one of the largest countries in the olive oil production around the world. The production process leads to generate massive amounts of olive solid wastes. The conventional production technique generates three products: olive oil (20%) and two streams of waste: a solid waste (30%) called olive cake and an aqueous waste (50%) called olive mill wastewater. (Niaounakis and Halvadakis 2006). The liquid waste contains high concentrations of organic compounds particularly phenolic that can be either simple phenols or polyphenols. Unfortunately, these effluent wastes discharged into environment without any treatment because the lack of legislation that can be restricted the producers. In contrast, for the solid waste of olive oil production usually used as an animal feed specially in Libya due to its high nutritional value. The recycle of this agriculture waste can contribute to increasing the profitability of the olive oil production process. Therefore, this biomass waste of agriculture by-products can be utilized as a source of activated carbon (AC) due to its cost-effective, which makes it suitable for various environmental applications, particularly water treatment. Different natural sources of AC were used to prepare activated carbon, for example, shells of coconut (Choudhuryet et al 1985) almond (López-González, J.D. et al 1980), stones of peach (Molina-, Sabio et al 1995) husks of rice (Chuah, T.G et al 2005), and stones of olives (López-González, J.D. et al 2000) Figure 1.

However, less attention has been received for the synthesis of AC from solid waste of olive oil production. So, the use of olive cake as an agriculture source for AC preparation not only because its low cost but also to decrease the amounts of solid wastes produced during olive milling processes. These agriculture by products can significantly cause many environmental issues like soil and water pollution.

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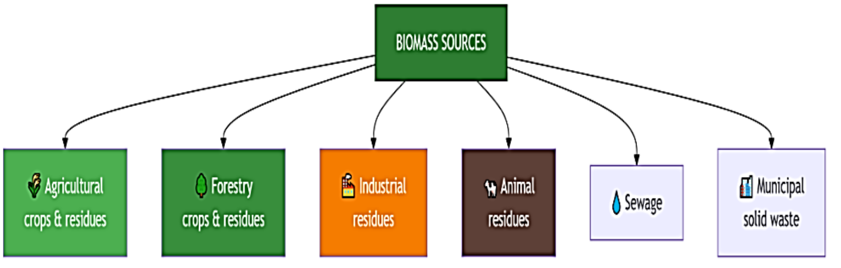

Fig. 1 Different types of biomass feedstock

The preparation of AC passes through several physical stages involving washing, cutting, carbonization, and milling. Furthermore, chemical activation via some chemicals such as nitric acid and potassium hydroxide in order to Functionalize AC Figure 2. Various physical and chemical conditions such as pH, temperature, and contact time were employed during manufacture procedures of AC to get the optimum values of these key parameters.

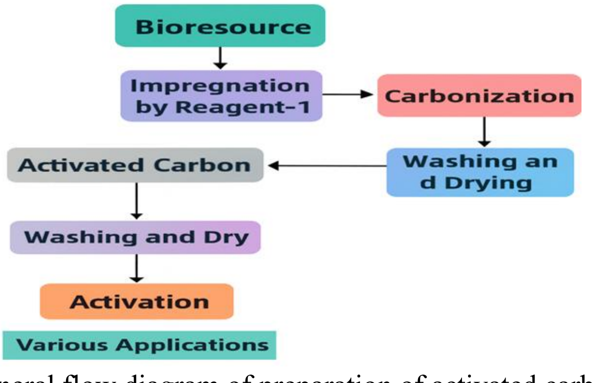

Fig. 1 General flow diagram of preparation of activated carbon from natural source

Drying, grinding and carbonizing are the most important steps of the physical treatment process that control the porous size and surface area of AC. For chemical procedures different chemicals have been used as a dehydrating agent such as $Zncl_2$, ($Zncl_2$ + H_2SO_2), KOH, and H_3PO_4 (Thomas et al 2015). The general

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activation procedures can be categorized as shown in Figure 2 (Sellami, R. et al 2008). The main point in the AC manufacture technique is to create holes inside the carbon particles in order to enhance the porosity leading to increase the surface area available for adsorption, allowing activated carbon to capture and retain a large number of contaminants. In general, the greater the porosity and surface area, the higher the adsorption capacity and treatment efficiency of the activated carbon.

Indeed limited studies were carried out to produce AC from olive cake using physical and chemical methods and used to remove pollutants from aqueous solution such as heavy metals (Cimino, G. et al 2005, Baccar, R. et al 2009, Aljundi, I.H. et al 2008), dyes (Cimino, G. et al 2006, Baccar, R. et al 2010, Baçaoui, A. et al 2001), and phenols (Michailof, C. et al 2008, Mameri, N. et al 2000). Organic compounds particularly phenol and phenolic are biorefractory pollutants found in water generating from the effluents of different pollutant sources including petroleum refineries, petrochemical, pharmaceuticals, textile wood, dying, and olive oil production, etc. Furthermore, phenols found in herbicides and insecticides so the excessiveness of these materials in agriculture activities can lead to water pollution (Girodsa, P. et al 2009, Mishra and Poddar 2013). Consequently, drinking water sources and the aquatic life can be negatively affected due to these toxic pollutants. In addition, presence of phenols and chlorine in water at the same time might generate chlorophenols which are very toxic compounds and harmful to human and organisms even at low concentrations (Mahvi et al 2004). WHO states that for safe discharge into surface water the concentration of phenols presented has to be less than 0.001mg/L in drinking water (Yamamura 2000). Several techniques have been used to these pollutants from wastewater such as biological treatment and advanced oxidation processes however, the more effective and low-cost method was adsorption processes via activated carbon. Therefore, this work aims to recycle one of the agriculture wastes as a precursor natural material for the preparation of activated carbon. Removal of phenol from aqueous solution was conducted to study the effectiveness adsorption of the prepared material. In addition, to get a maximum degradation efficiency some

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key parameters were investigated such as pH, contact time, and AC dose.

2. Materials and Methods

2.1 Materials and Equipment

All chemicals used in these experiments were checked for expire dates. Phenol (C_6H_6O) has a freezing point of 39.5-41 °C, a molecular weight of 94.11 g/mol was used. and a solubility of 82 g/l , 25% of phosphoric acid (H_3PO_4), and 25% of potassium hydroxide (KOH) were prepared (Dąbrowski, A. et al 2005). Figure 3 shows equipment used in this work including Spectrophotometer.

Fig. 3 Spectrophotometer.

2.2 Preparation of activated carbon (AC)

Olive-waste cakes were collected from an olive oil mill located in Assaba, Libya. This solid residue was used as a raw material for the production of activated carbon. The activation method can be defined as physical and chemical processes that enormously increase carbon surface area through removing hydrocarbons (Musa K. et al 2019). The process of changing the olive-waste cake into coal is called carbonization while the chemical decomposition of by heating it without oxygen is called pyrolysis (Tseng et al 2008). First of all, 500 g of olive-waste cake was wished with distilled water and dried at 250 ^{0}C for 180 min. Then the carbonized sample was crushed till became homogeneous powder. After that the powder taken into a thermal frying pan and covered by aluminium foil. This

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pan was put in the furnace at 300 ^{0}C for 3hr in order to perform the pyrolysis step. The carbonization material was again crushed to get more smaller particles. For model I (25% of KOH) Chemical activation was performed using which mixed with the powder to make a paste. Then the mixture was taken into the furnace at 100 ^{0}C and 30 min for drying. Finally, the prepared activated carbon was washed using distilled water and filtered to get the final form of the activated carbon. Figure 4 shows the prepared carbon from olive-waste cake.

Fig. 4 Prepared Activated Carbone

All above procedures were repeated for model II (25% of KOH and 25% H_3PO_4). Phosphoric acid activates biomass through a different mechanism. It promotes dehydration and cross-linking of the lignocellulosic structure during carbonization. This helps preserve the carbon framework while developing mesopores in addition to micropores. Both models were leaven to naturally dry for three days at the room temperature.

2.3 Aqueous solution of Phenol (Adsorbate)

Stock solution of phenol (1000 ppm) in distilled water was prepared and stirred for 15 min for more homogeneous. Then, at room temperature different concentrations (5 ppm, 15 ppm, 20 ppm, 25 ppm, 30 ppm, and 35 ppm) of phenol were prepared by diluting the stock solution. Absorbency using spectrophotometer were measured at these concentrations to get a relationship between various phenol concentrations and absorbency. Figure 5 shows the relation between phenol doses and absorbency.

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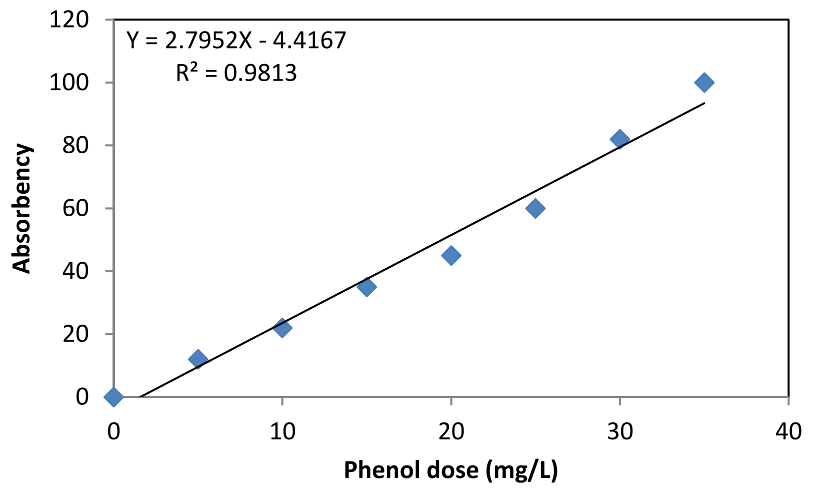

Fig. 5 Relationship between phenol concentrations and absorbency

2.4 Use of activated carbon (AC)

Two models of activated carbon (AC) were used to remove phenol from an aqueous solution at pH 7. Two grams of AC (Model I) was added to 1L of 30 ppm phenol sample and continuously stirred for 5 min before taking first sample for degradation reading. Then five samples were taken at 15 min, 45 min, 90 min, 120 min, and 150 min respectively. To study the effect of pH and the initial concentration of AC the above steps were repeated for various values. Furthermore, the same procedures were implemented for AC (Model II).

3. Results and Discussion

3.1 Comparison between Model I and Model II

The efficiency of phenol degradation for each condition at fixed time interval was determined. The removal percentage of the pollutant from wastewater was calculated using the following equation:

$$\text{Removal (\%)} = [(C_0 - C_e) / C_0] \times 100 \qquad (1)$$

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Where, C_o and C_e are the phenol concentration at initial stage and equilibrium stage respectively (ppm).
To investigate the influence of acid addition on the preparation method of AC, two models were used at the same conditions (2 g AC, pH 7, 150 min) to removal of 30 ppm phenol. Figure 6 shows the obtained experimental results of this comparison.

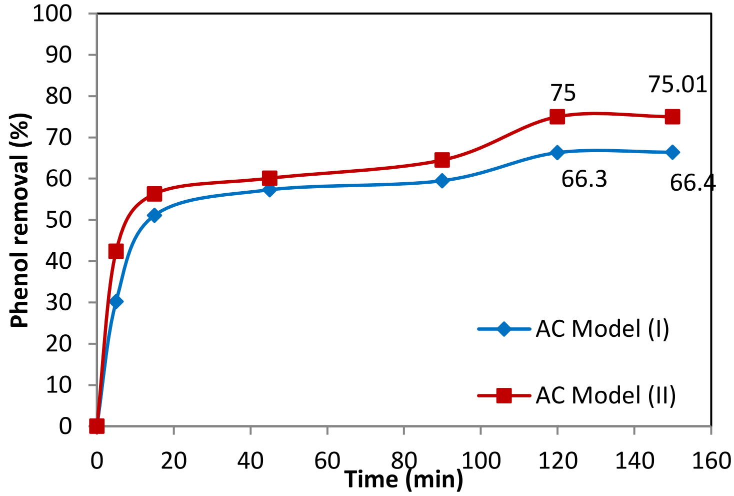

Fig. 6 Comparison between AC models I & II (pH 7, 2 g/L AC, 30 ppm Ph)

It can be seen that the activated carbon treated with phosphoric acid was more effective than zinc chloride alone because it typically produces a higher surface area, and more oxygen-containing functional groups that enhance pollutant adsorption. In addition, his result might be due to lower the total ash content of the carbon and more dry of water content. Acid washed activated carbon is desirable for treating drinking water and food grade applications. Furthermore, the maximum removal for both models was at 150 min of contact time.

3.2 Effect of AC dose

The influence of the initial concentration of activated carbon on the phenol removal was investigated. In this part the model II was used to study this effect due to its higher efficiency comparison to model I. Different initial AC doses was used at following conditions

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involving 30 ppm of phenol, pH 7-, and 150-min contact time. Figure 7 shows the obtained results and can be clearly seen the removal efficiency increases with increasing AC concentrations and the maximum value was at 2.5 g/L then the degradation percentage started decreasing which might be due to unsaturated active centers.

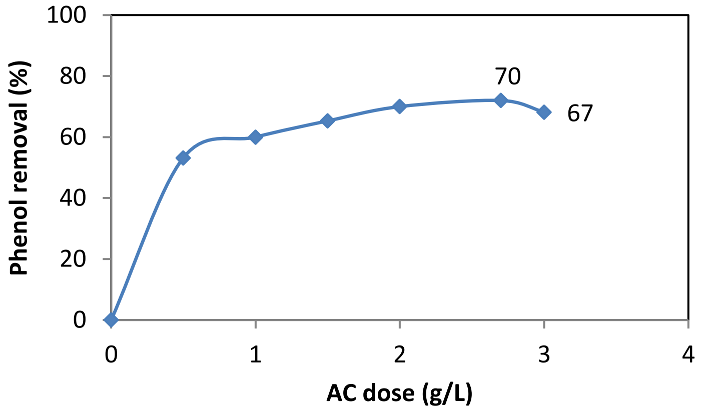

Fig. 7 Effect of AC dose on Phenol adsorption (Model II, pH 7, 30 ppm Ph, 150 min)

3.2 Effect of pH

pH is a key parameter playing important role in adsorption processes. Influence of pH on the removal of phenol from the synthetic wastewater was investigated. The adsorption of the pollutant onto the surface of activated carbon is mainly affected by the surface charge in other world the value of pH. Figure 8 shows the achieved results of the pH effect on the dye adsorption onto AC (Model II) from aqueous solution.

It can be seen that the maximum removal efficiency was detected at pH 5, meaning the surface has a positive charge due to excess protons in the solution. It is known that the decrease of pH value leading to rise in the H^+ concentration in the aqueous solution and the activated carbon surface gains positive charge by absorbing H^+ ions. As a result, when the adsorbent surface is positively charged phenol species own a strong attraction with positively charged of the carbon surface (Hettiarachchi et al 2017). Above this optimum

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pH value, adsorption of the pollutant decreased which might be due to weakening of electrostatic force of attraction between adsorbate and adsorbent.

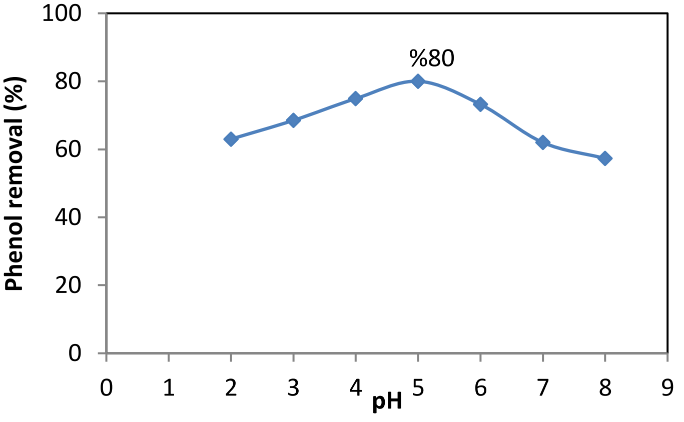

Fig. 8 Influence of pH on the adsorption of 30 ppm Phenol (2.7 g/L AC Modell II, 150 min)

4. Conclusion

- Activated carbon prepared from olive cake proved to be an effective and low-cost adsorbent for the removal of phenol from synthetic wastewater.
- Chemical activation using a combination of potassium hydroxide (KOH) and phosphoric acid (H_3PO_4) produced activated carbon with superior adsorption performance compared to activation with KOH alone.
- The adsorption efficiency was strongly influenced by the solution pH and the adsorbent dosage, making these critical operating parameters.
- Acidic conditions provided the highest phenol adsorption efficiency, indicating that low pH enhances the adsorption process.
- A maximum phenol removal efficiency of 80% was achieved under the optimized experimental conditions.

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- Olive cake, an abundant agricultural by-product, represents a renewable, sustainable, and economical precursor for producing activated carbon with high adsorption capacity.
- The prepared activated carbon shows promising potential for the removal of phenol and could be further investigated for the treatment of other organic contaminants in wastewater.

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