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Kinetic study on the adsorption of pollutants from olive mill wastewater onto granular activated carbon

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ABSTRACT

This research aimed to examine the effect of contact time on total organic carbon (TOC) removal rates associated with adsorption of pollutants from different olive mill wastewater (OMW) samples onto activated granular activated carbon (GAC). The first sample was a raw OMW that was microfiltered through a 100 µm membrane and the second sample was an OMW's permeate from a 50 kDa filtration. The TOC removal rate (%) of pollutants from prefiltered OMW increased over time, reaching its peak after 34 h (2040 min). Subsequently, the system reached adsorption equilibrium, corresponding to a removal rate of 60%. Then, it stabilized at this value till the end of adsorption at 48 h (2880 min). TOC removal rates (%) (corresponding to adsorption at different concentrations of GAC) of pollutants permeating 50 kDa also increased over time, reaching their peaks after 2040 minutes. The highest TOC removal rate was around 85%. This study also investigated the kinetics associated with this adsorption. To gain a comprehensive process understanding, pseudo-first-order (PFO) and pseudo-second-order (PSO) were employed as kinetic models. The second-order model best expressed the adsorption process which achieved equilibrium within 34 h.

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1. Introduction

The expansion of olive oil production has been accompanied by a significant increase in the creation of olive mill wastewater (OMW). These wastewaters constitute a primary environmental concern for the olive oil sector. It is assumed that approximately 107 m³ of OMWs are produced annually on a global scale, with the majority originating from the Mediterranean area [1]. Mitigating the volume of OMWs created each year presents a substantial environmental challenge for countries within this area [1,2]. OMWs are characterized by an acidic pH and appear as dark liquids, with values ranging from 4.5 to 6.0 [3,4]. The primary factor that contributes to the pollution load of these wastewaters is their elevated organic matter content. The chemical oxygen demand (COD) and the biochemical oxygen demand (BOD₅) of the OMWs can reach values as high as 220 and 100 g/L, respectively. Polyphenols, with concentrations ranging from 0.5 to 24 g/L, are a source of OMW phytotoxicity and resistance to biodegradation [4,5]. Until now, Morocco has not had particular laws regulating the management of OMWs. In the absence of formal regulations, most Moroccan and international olive oil mills dispose of

OMWs into the ponds or discharge them in the environment without a preliminary treatment. This procedure presents multiple environmental challenges, including the release of unpleasant smells, the emission of gases, soil infiltration, and the proliferation of insects. Furthermore, the elevated concentration of lipids in OMWs promotes the formation of films that cover the surface of the ponds, inhibiting natural evaporation. The growing international need for olive oil is expected to worsen this situation in the coming years [2,6].

Research has focused on the creation of biotechnological uses for the synthesis of metabolites from OMWs [5,7]. De Carluccio *et al.* [8] investigated the impact of the electroassisted Fenton (EAF) process on the bacterial community within a moving-bed biofilm reactor (MBBR) during co-treatment of OMW with urban wastewater (UWW). Metagenomic analysis revealed that EAF pre-treatment, which achieved a total phenol removal of 84%, promoted an appearance of bacterial genera in the MBBR which were absent, in a condition without pre-treatment. Notably, under EAF pre-treatment conditions, *Candidatus* competibacter emerged as the dominant denitrifying bacterium, replacing *Amaricoccus*. Other pretreatment techniques of OMW are the coagulation / flocculation and

sedimentation processes. Fleyfel *et al.* [9] used 8 g/L of CaO in combination with 7 g/L of $\text{Al}_2(\text{SO}_4)_3$, in a first coagulation / flocculation process, and achieved removal rates of 10% for electrical conductivity (EC), 41% for total solids, and 48% for COD from an OMW. In the second process, they employed 5 g/L of CaO and 4 g/L of $\text{Al}_2(\text{SO}_4)_3$, resulting in lower removal rates for total solids (37%) but higher removal rates for COD (67%) from the OMW. In another study, Vaz *et al.* [10] highlighted that 12-hour sedimentation led to removal rates of 45, 50, and 64% for TOC, COD, and BOD_5 , respectively, from an OMW. Membrane processes have been investigated for OMWs treatment, aiming to generate liquids of sufficient quality for eco-friendly disposal [5,11]. Ultrafiltration (UF) has appeared as a highly efficient alternative to conventional separating techniques. It offers numerous benefits, such as effective oil removal, high selectivity, continuous and automated operation, cost-effectiveness, and low energy consumption. In particular, UF is considered the most effective method for managing stable emulsions [12-14]. Cifuentes-Cabezas *et al.* [15] investigated OMWs treatment employing 5, 15, and 50 kDa ceramic membranes. The 15 kDa membrane achieved substantial removal of color (72%) and turbidity (99%), while also decreasing the organic load by 54%, under 3 m/s and 3 bar as working conditions. However, this study found that UF alone did not result in a significantly elevated COD rejection rate, which ranged from 48 to 63%. This suggests that a coupled process is required to improve the COD removal efficiency [16,17]. For example, Ouadah *et al.* [18] treated OMWs by combining ultrasonication with UF. This technique resulted in removal efficiencies of 88% for COD, 85% for total organic carbon (TOC), 74% for polyphenols, and 91% for ammonium nitrogen. The application of ultrasound resulted in changes to the dynamic properties of the OMW, such as a reduction in viscosity, which facilitated an improved permeate flux. Several studies highlight recent single and coupled processes, revealing that a single treatment method is insufficient to achieve a high COD removal efficiency [10]. Granular activated carbon (GAC), characterized by its high porosity and substantial surface area, has attracted considerable attention for its potential in the adsorption of contaminants from OMW [19]. For example, the use of commercial AC for phenolic compounds [20] and biophenol adsorption [21] has been extensively studied. In a study, Abeer *et al.* [22] proposed a low-cost method to mitigate OMW's phenolic composition by using modified GAC. Commercial GAC was selected for its high surface area and adsorption capacity and was subsequently modified through oxidative treatment with concentrated nitric acid and reductive treatment using a 10% by weight ammonia solution. Optimization of treatment conditions showed that the reduced GAC at pH = 9 achieved the highest phenolic removal efficiency, decreasing the phenolic content by 88% after 48 hours. Furthermore, Odeh *et al.* [23] developed a nanocomposite material that consists of Fe_3O_4 , $\text{FeO}(\text{OH})$ / zeolite and GAC for the remediation of OMW. Additionally, AC coated with milk proteins has been explored for OMW's polyphenols separation [24]. Furthermore, GAC derived from Jordanian functionalized olive cake, modified with $\text{Cu}/\text{Cu}_2\text{O}/\text{CuO}$, has been used for OMW's polyphenols separation [25]. AC produced through the chemical activation of olive pomace and encapsulated in calcium alginate has also been utilized for the uptake of polyphenols from OMW [26]. However, we know that the coupling of adsorption onto GAC with UF for OMW treatment has not been previously investigated.

A previous study showed the benefits of coupling processes to remove pollution from OMW [27]. Unfortunately, the end-of-treatment concentrations were higher than the discharge standards. The aim of this study is to show the effectiveness of GAC as an environmentally friendly adsorbent for the treatment of OMW, especially for lowering TOC concentration. In this study, a hybrid process with microfiltration and ultrafiltration

was developed, followed separately by adsorption onto GAC to treat different OMW samples. The first sample was a raw OMW that was microfiltered through a 100 μm membrane, and the second sample was an OMW that was ultrafiltered through a 150, then through a 50 kDa membranes. The adsorption onto GAC was used as a post-treatment process to lower pollutant concentrations in the OMWs.

2. Experimental

2.1. Analytical methods

The OMW permeate of 100 μm MF and the permeate of 50 kDa UF were analyzed to detect changes in the pollutant concentration. pH and turbidity were measured using a pH meter (HACH sensION + pH3) and a WTW Turb 55Q IR turbidimeter. Electrical conductivity (EC) was measured with a WTW conductivity meter. TOC was measured with a TOC-L type total organic carbon analyzer (Shimadzu, Japan).

2.2. Adsorption onto granular activated carbon (GAC)

Experiments were also performed to identify the optimum GAC mass for OMW post-treatment, based on TOC and turbidity removal efficiencies. Experiments were executed on the pre-filtered OMW, and the 50 kDa permeate, which were mixed with different masses of GAC (C1220G95 in grain mesh, Carbio12, France), a 100% natural product made from walnut shells and coconut. The mixtures were stirred at 400 rpm speed for 48 h. 400 mL of the pre-filtered sample was mixed with 20 g GAC/L while the 50 kDa permeate was mixed separately with 10, 20 and 40 g GAC/L. The mixtures were then shaken to reach equilibrium. The post-treated OMWs were centrifuged at 10200 rpm for 5 min and filtered for residual carbon removal. These experiments were monitored over time. The adsorption of GAC was studied at different time intervals from 0 to 48 h with the adsorbent concentrations mentioned above and at a temperature of 288 K. The adsorption capacity was determined using Equation 1 [26].

$$q = \left(\frac{C_0 - C}{m} \right) \times V \quad (1)$$

where q: the adsorbed quantity in mg/g; C_0 : the initial concentration of pollutants in mg/L; V: the volume of the solution in mL; and m: the mass of activated carbon in g.

2.3. Adsorption kinetics

Adsorption kinetics is a crucial parameter that reflects the purification efficiency of an adsorbent. It also facilitates the estimation of the amount of pollutants adsorbed over time. Adsorption kinetics can be described using various models. In this study, pseudo-first and second order models were employed to determine nature and kinetic order of OMW pollutants adsorption onto activated carbon [26].

3. Results and discussion

3.1. Effect of time of contact

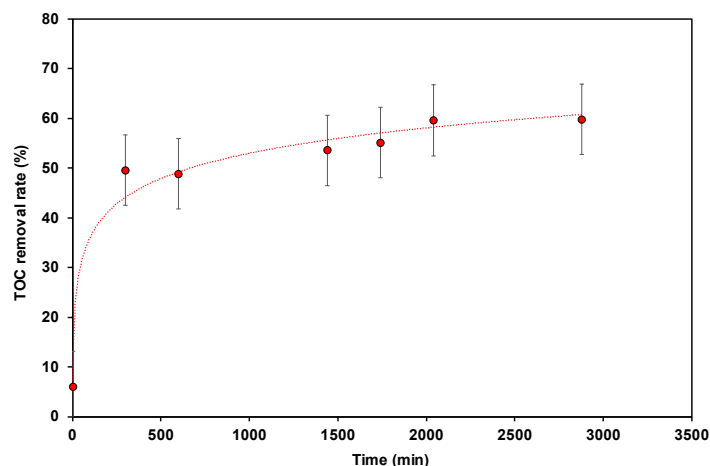
Figures 1 and 2 below illustrate the temporal effect on TOC removal rates of pollutants during adsorption on GAC. The TOC removal rate (%) of pollutants from prefiltered OMW increased over time, reaching its peak after 2040 minutes. Subsequently, the system reached adsorption equilibrium, corresponding to a removal rate of 60%. Then, it stabilized at this value until the end of adsorption at 2880 minutes.

Table 1. Equations and constant values of kinetic models for prefiltered OMW.

Sample	GAC concentration (g/L)	Kinetic model	Equation	Constants	AC results
Prefiltered OMW	20	Pseudo-first-order (PFO)	$\ln(q_e - qt) = \ln(q_e) - K_1 \cdot t$	K_1	0.002327
				q_e	0.002468
		Pseudo-second-order (PSO)	$1/qt = (1/K_2 \cdot q_e \cdot t) + 1/q_e$	K_2	27388
				q_e	0.002468

Table 2. Equations and constant values of kinetic models for 50 kDa permeate.

Sample	Kinetic model	GAC concentration (g/L)	Equation	Constants	Values
Permeate from 50 kDa filtration	Pseudo-first-order (PFO)	10	$\ln(q_e - qt) = \ln(q_e) - K_1 \cdot t$	K_1, q_e	0.002255, 0.000604
		20		K_1, q_e	0.001962, 0.000324
		40		K_1, q_e	0.000637, 0.000139
	Pseudo-second-order (PSO)	10	$1/qt = (1/K_2 \cdot q_e \cdot t) + 1/q_e$	K_2, q_e	68220, 0.000604
		20		K_2, q_e	63119, 0.000324
		40		K_2, q_e	101852, 0.000139

**Figure 1.** Effect of time on the TOC removal rate of pollutants from prefiltered OMW adsorption on 20 g GAC/L.

Comparative studies on the adsorption of OMW pollutants have highlighted the influence of the adsorbent material and process conditions such as pH, temperature, and adsorbent dosage. Research conducted by Ammari *et al.* [28] on clinker (20 mg) revealed rapid kinetics, with a TOC removal rate reaching 81% in 35 minutes. In contrast, the work of Abdoul-Latif *et al.* [29] on olive pomace biochar (2 g/L), which aimed to remove phenolic compounds, showed a much longer equilibrium time of 24 hours, as well as a wide range of removal efficiencies between 10 and 90%. A critical factor identified in the latter study is pH: maximum adsorption occurred under alkaline conditions (pH = 10), highlighting the importance of this process parameter.

The TOC removal rates (%) of pollutants from 50 kDa permeate also increased over time, reaching their peaks after 2040 minutes. For adsorption onto 20 g GAC/L, the peak was around 61%. For adsorption onto 10 g GAC/L, the peak was around 62% and for adsorption onto 40 g GAC/L, the peak was around 85%. Abu Dalo *et al.* [30] studied a hybrid process to treat an OMW that combines coagulation-flocculation and adsorption to remove organic pollutants, particularly phenolic compounds. They evaluated the adsorption efficiency of raw and activated volcanic tuff (VT), enriched with magnetite nanoparticles (MNP), for removing COD and total phenolic compounds (TPC) using batch column experiments. The influence of contact time was evaluated for volcanic tuff activated by calcination alone (VTC) and by combined acid-calcination treatment (VTAC). Using an adsorbent dose of 5% (m/m) (VT with 1% m/m MNP) on pretreated OMW for contact times ranging from 6 to 96 hours, the results revealed different kinetic profiles. The nonactivated VT and VTC samples required 48 hours to reach adsorption equilibrium, while the VTAC samples reached it in only 24 hours. In addition, the VTAC medium proved to be more effective at removing COD, with this

efficiency further improved by the addition of 0.5% (w/w) MNPs, achieving a maximum COD removal of 76% at an optimal pH of 8.

3.2. Modeling of adsorption kinetics

The purpose of this kinetic study was to evaluate the impact of time on the removal of pollutants. The rate of uptake determines the adsorbate retention time at the solid-liquid interface, thereby influencing the rate control mechanism. Tables 1 and 2 represent equations and constant values of kinetic models for prefiltered and permeates from 50 kDa filtration, respectively.

Critical insights for the application of activated carbons in adsorption processes were provided. The kinetic equations, along with the corresponding constant values, are summarized in Tables 1 and 2. The pseudo-second order model best describes the pollutants from OMWs onto the GAC. Figures 3 and 4 represent the two models employed as functions of time.

Ammari *et al.* tested the PFO and PSO models [28]. They observed that the correlation coefficient R^2 of the PFO model was far from 1 (0.89). They suggested that this model could not be used to describe the adsorption kinetics of organic matter (OMW) on clinker. In contrast, the PSO model yielded a perfect R^2 of approximately 1 (0.999). Therefore, this model better described the adsorption process of OMW on raw clinker. This model suggested that chemisorption predominated in this process.

El Amraoui *et al.* [31] studied the adsorption of organic matter and polyphenols present in an OMW using raw sawdust and activated carbon derived from *Pinus sylvestris* sawdust. Their experimental results demonstrated a removal efficiency of 47% for COD and 74% for polyphenols under specific conditions.

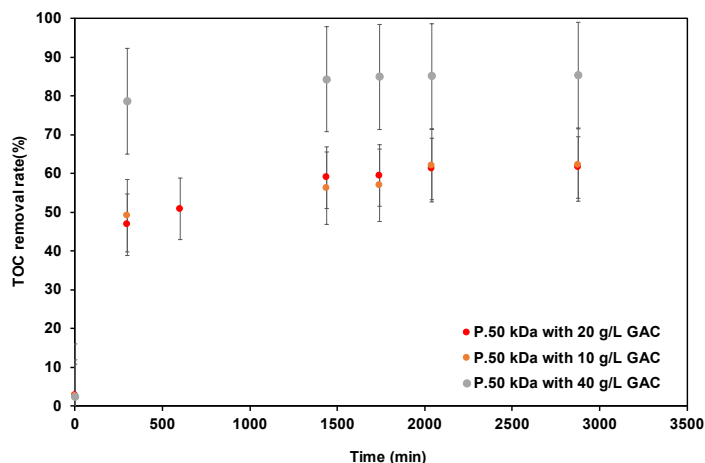


Figure 2. Effect of time on the TOC removal rate of pollutants from 50 kDa permeate adsorption on 10,20 and 40 g GAC/L.

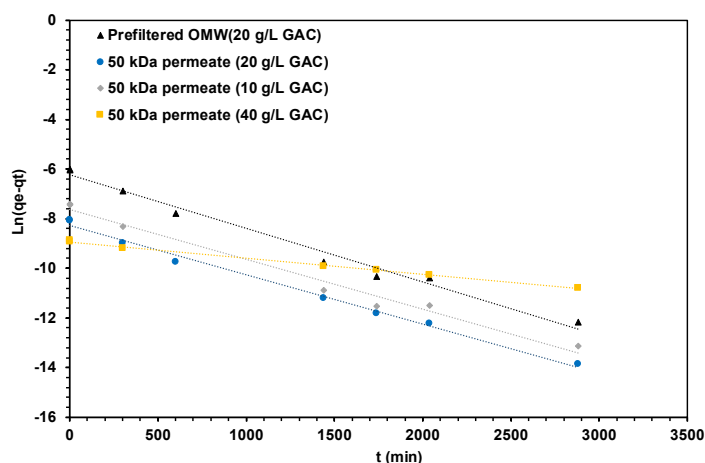


Figure 3. Pseudo-first-order for prefiltered OMW (treated with 20 g GAC/L) and 50 kDa of permeate (treated with 10, 20 and 40 g GAC/L).

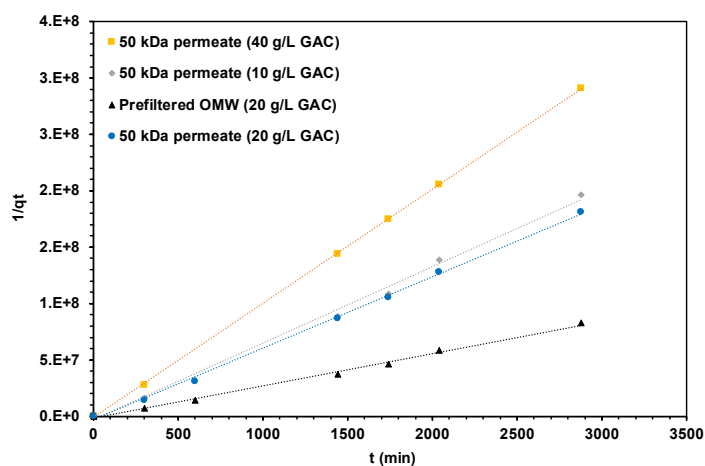


Figure 4. Pseudo-second order for prefiltered OMW (treated with 20 g GAC/L) and 50 kDa permeate (treated with 10, 20 and 40 g GAC/L).

The adsorption mechanism was elucidated through kinetic and isothermal analyses, which revealed distinct behaviors for these two classes of pollutants. The COD removal kinetics were best described by a PSO model, suggesting a monolayer adsorption process. In contrast, polyphenol adsorption was best described by a PFO model, indicating multilayer adsorption. The strong correlation between the COD data and the PSO model ($R^2 = 0.92$) also suggests a chemisorption

mechanism. On the contrary, the adherence of polyphenol adsorption to the PFO model is consistent with a physisorption process, as confirmed by the analysis of the kinetic model.

4. Conclusions

The discharge of OMW poses an important environmental issue, particularly for nations in the Mediterranean area. These

wastewater effluents are characterized by an elevated organic composition and contribute to various forms of pollution. Cross-flow ultrafiltration (UF) of OMW and its adsorption on activated carbon have emerged as promising treatment methods. This study investigated the effect of contact time between OMW pollutants and GAC, on the TOC removal rate; adsorption of pollutants (from prefiltered OMW) on 20 g GAC/L reached 60% as the TOC removal rate after 2880 min. Adsorption of pollutants from a 50 kDa OMW permeate on 20, 10 and 40 g GAC/L reached, respectively, 61, 62 and 85% as TOC removal rates, which correspond, respectively, to COD levels of 0.35, 0.344 and 0.133 g O₂/L in treated wastewaters. These levels of COD remain significantly below the discharge limit of 0.5 g O₂/L, as defined by the Moroccan standard (Official Bulletin No. 6199). To gain a complete understanding of the adsorption process, two pseudo-first and second-order kinetic models were utilized. The findings indicated that the adsorption process occurred, achieving equilibrium within 34 hrs. The pseudo-second-order model best described the adsorption of pollutants from OMWs onto the GAC.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest. Ethical approval: All ethical guidelines have been adhered to. Sample availability: Samples are available from the author.

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CRediT authorship contribution statement

Conceptualization: Philippe Moulin; Methodology: Philippe Moulin, Mohammed Zine; Validation: Philippe Moulin, El Mostapha Lotfi, Nouredine Touach; Formal Analysis: Mohammed Zine; Investigation: Mohammed Zine; Resources: Philippe Moulin; Data Curation: Mohammed Zine; Writing - Original Draft: Mohammed Zine; Writing - Review and Editing: Mohammed Zine, Philippe Moulin, El Mostapha Lotfi, Nouredine Touach; Visualization: Mohammed Zine, Philippe Moulin, El Mostapha Lotfi, Nouredine Touach; Funding acquisition: Mohammed Zine; Supervision: Philippe Moulin; Project Administration: Philippe Moulin, El Mostapha Lotfi, Nouredine Touach.

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