



J. Serb. Chem. Soc. 88 (4) 437–450 (2023)
JSCS–5637

Full factorial design methodology approach to optimize the elimination of gallic acid from water by coagulation using activated acorns barks as coagulant-aid

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(Received 23 June, revised 20 November, accepted 9 December 2022)

Abstract: This study investigated the elimination of organic matter from water by the coagulation process using a biomaterial “acorns barks” as a coagulant-aid with the presence of aluminium sulphate in low concentration. The removal of gallic acid from water was first studied by two processes: the adsorption on activated acorns barks, and coagulation by aluminium sulphate, separately. The hybrid system was then studied, and the optimal operating conditions were determined. The performance of the hybrid system (coagulation/adsorption) mainly depends on the initial concentration of gallic acid, the coagulant dose and the mass of coagulant-aid. A full factorial design 2^3 was used to determine the optimum conditions for gallic acid removal. The maximum removal of gallic acid in water was 92.48 %, achieved at 20 mg L⁻¹ of initial gallic acid concentration, 50 mg L⁻¹ of aluminium sulphate coagulant concentration and 1.5 g of activated acorns barks adsorbent mass. The application of these optimal conditions on urban wastewater for the elimination of organic matter has shown the performance of this hybrid system treatment.

Keywords: adsorption; biomaterial; hybrid system; optimization; urban wastewater.

INTRODUCTION

The increase of the quantity of wastewater containing various chemical substances and solid particles, caused by economic development, has become a serious threat to human health and sustainable development. Consequently, the treatment of polluted wastewaters remains a topic of global concern because

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<https://doi.org/10.2298/JSC220623084B>

wastewaters collected from municipalities, communities and industries must ultimately be returned to receiving waters or to the land.¹

Wastewater contains a mixture of pollutants, among which phenols and their derivatives are the most common organic pollutants of wastewater that require careful treatment.^{2,3} Phenols and its derivatives are toxic to humans, animals, and aquatic life, and they increase the oxygen demand of receiving water.⁴ Phenolic compounds are very harmful to organisms even at low concentrations due to their high toxicity and carcinogenic properties.⁵ Gallic acid is one of the most important phenolic components used in medical applications, food and the cosmetic industries.⁶ Consequently, a great deal of wastewater containing gallic acid discharges into water bodies. Therefore, it is necessary to remove gallic acid from water. Several studies have been carried out for the elimination of gallic acid by different processes.⁷⁻¹⁰ Moreover, it is difficult to remove gallic acid by the traditional water treatment processes due to its water solubility and small molecular weight.¹¹

The selection of a particular treatment technique depends on the nature of the effluent, waste type and concentration, presence of other constituents, level of removal required and economics.¹² Traditionally, biological treatment, activated carbon adsorption and solvent extraction are the most widely used methods for the removal of phenols and its derivatives from wastewater.¹³ Among them, adsorption is a well-established and powerful technique for treating domestic and industrial effluents.¹⁴ The adsorption method is characterized by low initial cost, simple operation, insensitive to toxic substances, and high effectiveness.¹⁵

Activated carbon is the most widely and effectively used adsorbent because of its large surface area, micro-porous nature, and high adsorption capacity. However, due to its high price and its regeneration cost, activated carbon becomes inconvenient as an adsorbent.¹⁶ Nowadays, one of the great challenges in the adsorption technologies is the exploring of new potential and low-cost adsorbents using biological material. These materials should be biodegradable and are presumed to be safe for human health.¹⁷ Producing activated carbons usually entails carbonization, pyrolysis and activation (either chemical or physical).^{18,19}

Coagulation flocculation technology has proved its advantages in separation processes. The process does not only separate suspended solids from water, but also remove colour and certain organic matters from diverse sources of wastewater.²⁰ Aluminium and iron salts are the most widely used coagulants in water treatment due to their high efficiency, as well as synthetic and natural organic polymers, which find application also.²¹ However, Al-based coagulants are hazardous and can cause health problems, for example Alzheimer's disease.²² In addition, it was reported that coagulation is not effective for some synthetic organic matters due to their physicochemical properties.²³ In recent years, there has been considerable interest in the development of biomaterial to become an alter-

native for conventional coagulants. Several studies referred to the use of alternative coagulants from recyclable materials, due to their cost effectiveness and good removal performance.²⁴

The coagulation treatment process can be influenced by several factors, but the failure to consider the interaction of factors may lead to erroneous conclusions. The design of experiments methodology could be a meaningful method for modelling and optimizing the treatment process with a minimum number of experiments in order to identify the effective parameters and their interactions. Factorial design is a more practical technique than other traditional methods for modelling a multi-variable system. Several studies have referred to the use of factorial design to optimize adsorption treatments^{25–27} and coagulation processes.^{28–30}

In this study, acorns barks were used as a coagulant-aid for the elimination of a phenolic acid (gallic acid) from water with coagulation treatment. A factorial design was used to study the individual and interactive effects of three parameters that may affect the removal efficiency of gallic acid, followed by an application of the optimal conditions obtained on an urban wastewater.

EXPERIMENTAL

Preparation of activated acorns barks

For the preparation of carbon from acorns barks, the barks were washed with distilled water and then dried in an oven at 105 °C for 24 h. Ten g of the raw material was immersed in 100 mL of H₂SO₄ solution (40 %) and kept at 105 °C for 2 h. The acid treated acorns barks was washed several times with distilled water and then dried at 105 °C for 24 h. The obtained sample was calcined at 500 °C for 2 h, crushed and sieved at a diameter ≤ 500 μm.³¹

Adsorption experiments

Batch adsorption experiments were carried out in 1000 mL of Erlenmeyer flasks in which 500 mL of aqueous solution was mixing with a determined mass of activated acorns barks.

The mixture was shaken under ambient temperature at 150 rpm for 4 h to attain equilibrium. Samples were collected at predetermined time intervals, then filtered through a filter (0.45 μm) for the determination of gallic acid concentration.

Coagulation–flocculation experiments

Coagulation experiments were conducted in jar-test apparatus by three steps: rapid mixing (200 rpm) for 3 min, slow mixing (60 rpm) for 30 min and 30 min sedimentation. The desired mass of activated acorns barks was added into 500 mL of organic compound solution in rapid mixing step, 30 s after adding the coagulant. Aluminium sulphate was used as a coagulant.

Gallic acid analysis

The concentration of gallic acid in water was determined by measuring the absorbance on a UV–Vis spectrophotometer Shimadzu UVmini-1240, at a wavelength of 254 nm.

RESULTS AND DISCUSSION

SEM and FTIR analysis

Scanning electron microscopy analysis (SEM). The analysis of the pore structure of the raw acorns barks and the activated acorns barks is given in Fig. 1.

It's clear that no porous structure for the raw acorns barks in contrary to the activated acorns barks which possess a good porous structure.

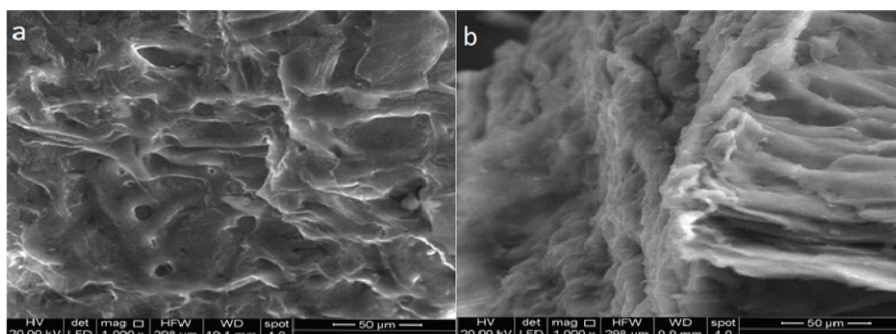


Fig. 1. SEM photographs of acorns barks raw (a) and activated (b).

For further study, the activated acorns barks were used as an adsorbent for the gallic acid elimination from water.

Fourier transform infrared spectroscopy (FTIR). FTIR is the best technique to analyse the chemical structural properties of natural materials.³²

FTIR spectra are a useful tool to identify functional groups in a molecule, as each specific chemical bond often has a unique energy absorption band, and can obtain structural and bond information.³³ Fig. 2 shows the FTIR spectra of the activated acorns barks. A wide absorption band at $3600\text{--}3050\text{ cm}^{-1}$ with a maximum at about 3350 cm^{-1} is due to O–H and N–H stretching groups.³⁴ The band at 1650 cm^{-1} characterizes the presence of carbonyl groups C=O.³⁵ Another band is observed at 1050 cm^{-1} corresponds to C–C stretching.³⁶

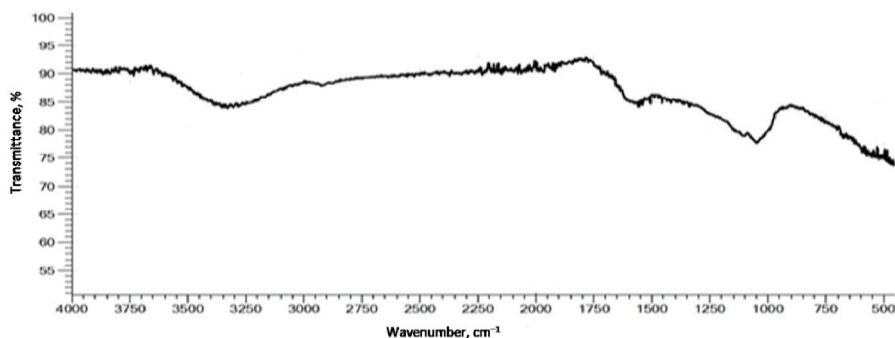


Fig. 2. FTIR of activated acorns barks.

Adsorption

The adsorption experiments were performed out in batch system with different mass of the activated acorns barks for 20 mg L^{-1} of gallic acid concentration.

The adsorption capacity is defined as the amount of gallic acid adsorbed per unit mass of adsorbent, $q / \text{mg g}^{-1}$:

$$q = (C_0 - C_t) \frac{V}{m} \quad (1)$$

where $C_0 / \text{mg L}^{-1}$ is the initial gallic acid concentration. $C_t / \text{mg L}^{-1}$ is the concentration at a defined time. V / L is the volume of solution, and m / g is the mass of the adsorbent.

Fig. 3 shows that that quantity of gallic acid can be eliminated by the activated acorns barks, and the adsorption equilibrium was 240 min. The adsorption capacity was found to be high at low doses, it decreased from 12.05 to 7.48 mg g^{-1} with the activated acorns barks increasing from 1 to 2 g L^{-1} at equilibrium time. This decrease in adsorption capacity with the increasing adsorbent dosage is due to the non-saturation of the adsorption sites.³⁷

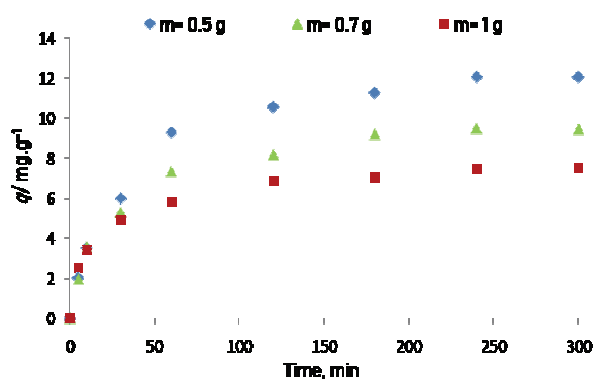


Fig. 3. The effect of adsorbent mass on the adsorption capacity of gallic acid.

Coagulation–flocculation

Different aluminium sulphate coagulant doses (10 to 100 mg L^{-1}) were used to remove gallic acid at a concentration of 20 mg L^{-1} . The variation of the removal of gallic acid ($R / \%$) was calculated as:

$$R / \% = 100 \left(\frac{C_0 - C_f}{C_0} \right) \quad (2)$$

where C_0 and C_f are initial and final concentrations of gallic acid, respectively.

Fig. 4 shows that the removal of gallic acid increases with the coagulant dose due to the growth in the number of binding sites. The straight line after 50 mg L^{-1} indicates that an optimum dose of 50 mg L^{-1} was found at a removal of 50.20 %. The coagulant capacity was steady due to the screening effect between coagulant molecules.³⁸ However, higher coagulant dose can give charge

reversal and destabilization of colloids, and the colloidal products are rather difficult to be removed by precipitation or charge neutralization.²⁴

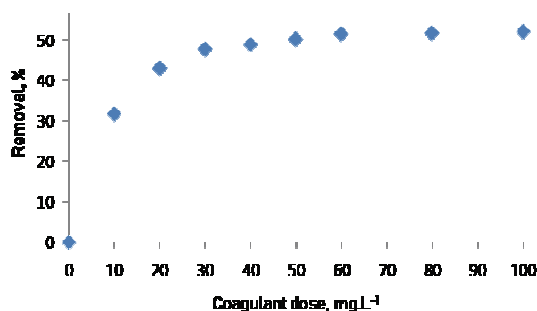


Fig. 4. Effect of coagulant dose on gallic acid removal by coagulation-flocculation.

Aluminum sulphate/adsorbent combination in coagulation process. Three masses of the adsorbent were used with the optimal dose of aluminium sulphate (50 mg L^{-1}) for the removal of gallic acid from water at a concentration of 20 mg L^{-1} . The intended mass of activated acorns barks was added into 500 mL of gallic acid solution in rapid mixing steps, 30 s after adding the coagulant in order to follow all the coagulation-flocculation steps.

The efficiency of hybrid system is well illustrated in Fig. 5. The removal of gallic acid from water by hybrid system is higher than the classical coagulation. Also, the removal of gallic acid increases with the activated acorns barks dose, in which the gallic acid removal increases from 64.32 % for 0.5 g to 91.35 % for 1.5 g. Thus, the activated acorns barks are an efficient coagulation-aid to enhance the coagulation performance with aluminium. This was consistent with previous studies in which better elimination performance was obtained when the coagulant-aid is used in combination with conventional coagulants.^{24,39,40}

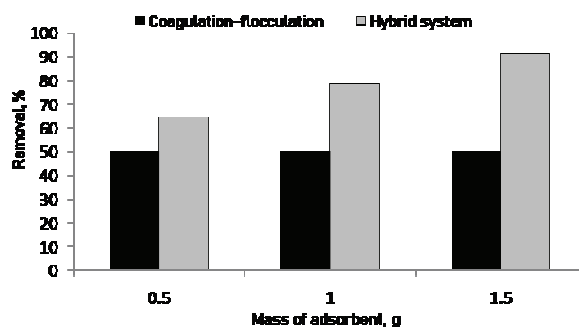


Fig. 5. Comparison of gallic acid removal by coagulation-flocculation and by hybrid system.

Optimization of coagulation hybrid system treatment by full factorial design. In this study, the full factorial design methodology was used to optimize the

treatment process and to establish the correlation between gallic acid removal and three input variables, the gallic acid concentration X_1 , the aluminium sulphate concentration X_2 and the mass of activated acorns barks X_3 . The range and level of each variable are given in Table I.

TABLE I. Factors and levels used in the full factorial design

Factor	Low level (-1)	Hight level (+1)
X_1 – Gallic acid concentration, mg L ⁻¹	20	50
X_2 – Aluminum sulphate concentration, mg L ⁻¹	10	50
X_3 – Mass of activated acorns barks, g	0	1.5

The statistical analyses were applied to validate the model using JMP8. The mathematical model of the first-order polynomial can be given as:

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{123}X_1X_2X_3 \quad (3)$$

Where Y is the theoretical response function (removal of gallic acid), a_0 is the constant, a_i is the linear effect and (a_{ij} and a_{ijk}) are the interaction effects.

The real and coded values are in Table II.

TABLE II. Full factorial design matrix of three variables in coded and real values with the experimental responses

Run	Real values			Coded values			R / %
	Gallic acid concentration, mg L ⁻¹	Aluminum sulphate concentration, mg L ⁻¹	Mass of activated acorns barks, g	X_1	X_2	X_3	
1	20	10	0.00	-1	-1	-1	31.69
2	50	10	0.00	+1	-1	-1	12.48
3	20	50	0.00	-1	+1	-1	50.2
4	50	50	0.00	+1	+1	-1	41.42
5	20	10	1.50	-1	-1	+1	84.6
6	50	10	1.50	+1	-1	+1	70.23
7	20	50	1.50	-1	+1	+1	91.35
8	50	50	1.50	+1	+1	+1	77.48
9	35	30	0.75	0	0	0	64.82
10	35	30	0.75	0	0	0	61.37

By substituting the regression coefficients in Eq. (3), by their numerical values as given in Table III, we get:

$$Y = 58.564 - 7.02875X_1 + 7.68125X_2 + 23.48375X_3 + 1.36625X_1X_2 - 0.03125X_1X_3 - 4.18125X_2X_3 - 1.24125X_1X_2X_3 \quad (4)$$

Fig. 6 shows the predicted values versus the experimental values of the removal of gallic acid. The straight line in red colour represents the model, rectangles points are experimental points and the dotted lines represent the area of acceptable variation.

TABLE III. Estimated regression coefficients for the removal yield of gallic acid

Parameter	Estimate	Standard error	<i>t</i> -value	<i>P</i> -value
Constant	58.564	1.692279	34.61	0.0008
X_1	-7.02875	1.892025	-3.71	0.0654
X_2	7.68125	1.892025	4.06	0.0557
X_3	23.48375	1.892025	12.41	0.0064
$X_1 X_2$	1.36625	1.892025	0.72	0.5452
$X_1 X_3$	-0.03125	1.892025	-0.02	0.9883
$X_2 X_3$	-4.18125	1.892025	-2.21	0.1577
$X_1 X_2 X_3$	-1.24125	1.892025	-0.66	0.5792

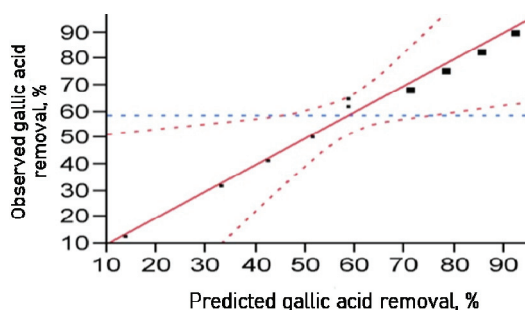


Fig. 6. Observed versus predicted response (removal of gallic acid).

The high value of $R^2 = 0.989$ and R^2 adjusted = 0.972 indicate that the model was successful in correlating the response to the studied parameters.

Student's t-test. The Student's test determines the significance of the regression coefficients of the different parameters. A large *t*-value associated with a low *P*-value (< 0.05) of a variable indicates a high significance of the corresponding model term.

After eliminating the insignificant effects by step-by-step method, Table IV gives only the significant effects. The results suggest that the linear effects of gallic acid concentration, the aluminium sulphate concentration, the mass of adsorbent, and the interaction effect between the latter two are significant.

TABLE IV. Estimated regression significant coefficients for the removal yield of gallic acid

Parameter	Estimate	Standard error	<i>t</i> -value	<i>P</i> -value
Constant	58.564	1.300328	45.04	<0.0001
X_1	-7.02875	1.453811	-4.83	0.0047
X_2	7.68125	1.453811	5.28	0.0032
X_3	23.48375	1.453811	16.15	<0.0001
$X_2 X_3$	-4.18125	1.453811	-2.88	0.0347

The most significant effect is the activated acorns barks with a positive value. It means that the removal of gallic acid increases with the activated acorns bark dose.

The empirical model for the removal becomes:

$$Y = 58.568 - 7.02875X_1 + 7.68125X_2 + 23.48375X_3 - 4.18125X_2X_3 \quad (5)$$

Interactions plot. The interactions plot is given in Fig. 7. An effective interaction was observed between the concentration of aluminium sulphate and the mass of activated acorns barks with negative value (-4.18125), It means that the effect of activated acorns barks dose is very high and positive on the removal of gallic acid when the sulphate concentration is low.

The other interactions seem to be insignificant, the lines representing the effects are parallels in the squares of the diagram.

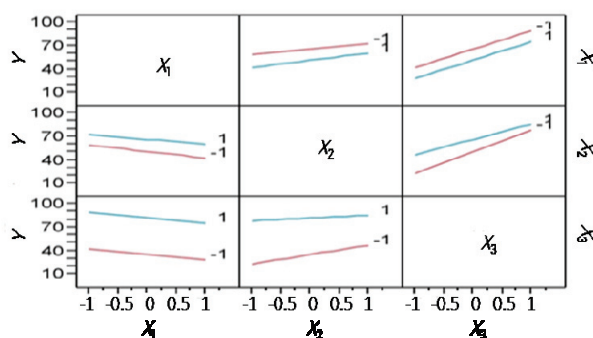


Fig. 7. Interaction effects.

Analysis of variance (ANOVA). The significance of the adjusted response was justified by ANOVA. The ANOVA summary is shown in Table V. The F -value obtained (80.12) and the P -value inferior to 0.05 indicate that the model is valid to predict the removal of gallic acid with the studied variables.

TABLE V. Analysis of variance (ANOVA); p : number of significant coefficients; N : total number of experiments

Source	Degrees of freedom	Sum of squares	Mean square	F -ration	P -value
Model	$(p-1) = 4$	5418.9944	1354.75	80.1222	0.0001
Residual	$(N-p) = 5$	84.5427	16.91		
Total	9	5503.5370			

Optimization design using the desirability function. The desirability function approach is the most current and strongly suggested method for the optimization of one or more responses.⁴¹

As shown in Fig. 8, the best combination of factor settings for achieving the optimum response was found to be: gallic acid concentration 20 mg L^{-1} , aluminium sulphate concentration 50 mg L^{-1} and activated acorns barks dose 1.5 g . These conditions lead to a removal of gallic acid of 92.482% with a desirability of 0.900 .

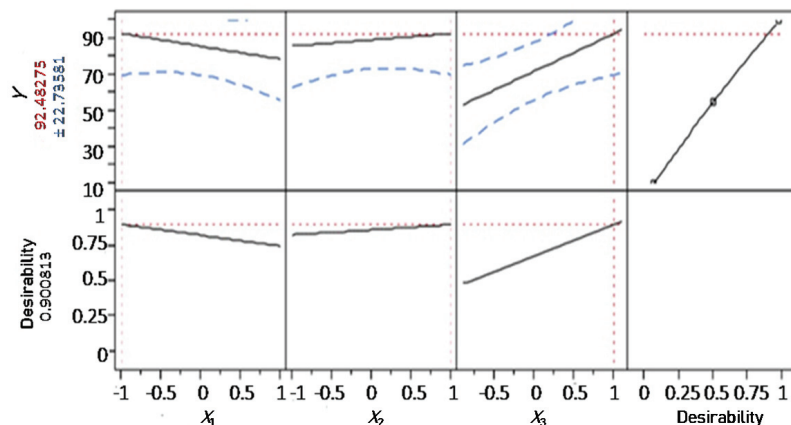


Fig. 8. Desirability function for removal of gallic acid.

Application of the hybrid system treatment on urban wastewater

Firstly, the hybrid system was used for treating a mixture of phenolic compounds: 20 mg L⁻¹ of gallic acid, 20 mg L⁻¹ of phenol and 10 mg L⁻¹ of humic substances under the optimal conditions found previously: 50 mg L⁻¹ of aluminium sulphate with 1.5 g of activated acorns barks.

Simple organic molecules with no functional group or only one non-ionized functional group (as phenol) are not (or very slightly) removed by coagulation, and molecules that have -COOH groups are easily eliminated.⁴² Fig. 9 shows that the use of activated acorns barks as a coagulant-aid improved significantly the elimination of organic matter from water in comparison with the classical coagulation treatment. The high removal (96.72 %) is due to the contribution of the coagulant-aid to the elimination of organic matter by adsorption.

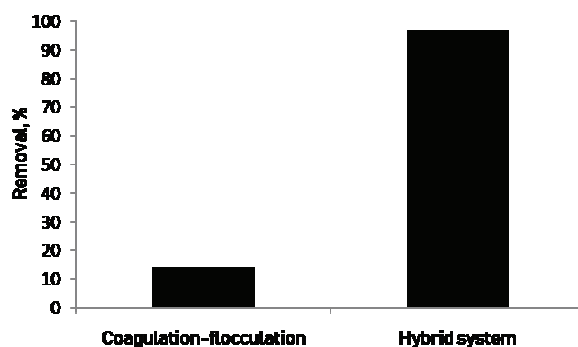


Fig. 9. Removal of organic matter by coagulation-flocculation and hybrid system.

Secondly, the optimal conditions of the hybrid system were applied to urban wastewater. The water sample was collected after pretreatment step at the waste-

water treatment plant which is situated in the Wilaya of Boumerdes (in the north of Algeria, 45 km from the capital Algiers). Its process chain consists of pretreatment, biological treatment and clarification. The treated wastewater is destined for irrigation.

At the treatment plant biological treatment is used for the elimination of organic pollution from water. In our study, organic pollution was eliminated from wastewater by the physicochemical treatment and compared with the biological treatment used.

Fig. 10 shows the organic pollution parameters in wastewater before secondary treatment, wastewater treated by hybrid system, and wastewater treated by biological treatment at the treatment plant.

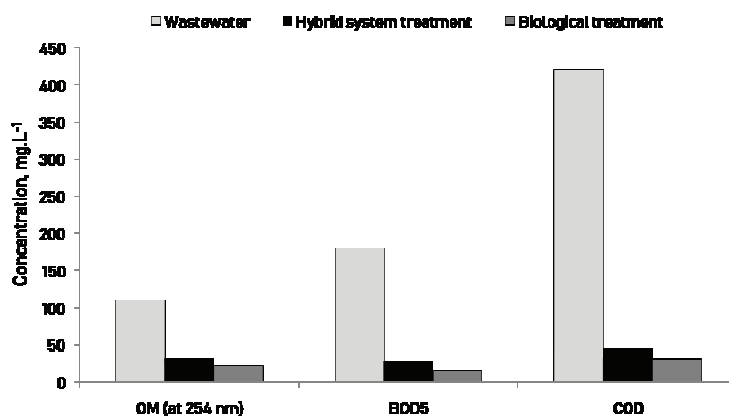


Fig. 10. Organic pollution parameters in wastewater before and after treatment.

With the hybrid system treatment, we observe an elimination of 72.41 % of organic matter (measured by measuring the absorbance at a wavelength of 254 nm), a reduction of 84.37 % for biological oxygen demand (BOD_5) and 89.45 % for chemical oxygen demand (COD). These results confirm the possibility of using acorns barks as a coagulant-aid for the elimination of organic matter in wastewater. The significant reduction of BOD_5 and COD which represent cocktail of many organic compounds, confirm that the obtained model is valid for real wastewater and not only for one compound.

Thus, the efficiency of both hybrid system and biological treatments are very close; this shows that the treatment by combination coagulation/adsorption (physicochemical treatment) can be an alternative to biological treatment.

CONCLUSIONS

The present study analysed the feasibility of activated acorns barks using as coagulant-aid to improve the coagulation process for the elimination of gallic acid from water. The elimination of gallic acid by a classical coagulation was partial,

with a removal of 50.20 % for a concentration of 20 mg L⁻¹ gallic acid. This result was obtained using a maximum aluminium sulphate dose of 50 mg L⁻¹.

The hybrid system of coagulation process for the removal of gallic acid from water using activated acorns barks as coagulant-aid was successfully modelled applying the full factorial design methodology. The optimal conditions for the hybrid system in which the removal of gallic acid reached 92.48 %, were achieved at the initial gallic acid concentration of 20 mg L⁻¹, the aluminium sulphate dose of 50 mg L⁻¹ and the activated acorns barks mass 1.5 g.

The hybrid system has shown high efficiency in the elimination of organic matter in wastewater, the application of the optimal conditions removes 72.41 % of organic matter, 84.37 % of BOD₅ and 89.45 % of COD from urban wastewater. The performance of hybrid system in comparison with the secondary treatment applied in wastewater treatment plant gives the same results, which means that the hybrid system (combination coagulation/adsorption) can be an alternative to biological treatment.

ИЗВОД

МЕТОДОЛОГИЈА ПОТПУНОГ ФАКТОРИЈАЛНОГ ДИЗАЈНА У ОПТИМИЗАЦИЈИ ЕЛИМИНАЦИЈЕ ГАЛНЕ КИСЕЛИНЕ ИЗ ВОДЕ ПУТЕМ КОАГУЛАЦИЈЕ ПОМОЋУ АКТИВИРАНЕ КОРЕ ЖИРА КАО КОАГУЛАНТА

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Ова студија је истраживала елиминацију органске материје из воде процесом коагулације коришћењем биоматеријала, коре жира, као помоћног коагуланта уз присуство алуминијум-сулфата у ниској концентрацији. Уклањање галне киселине из воде је прво проучавано применом два независна процеса: адсорпције на активираној кори жира и коагулације алуминијум-сулфатом. Затим је проучен хибридни систем (коагулација/адсорпција) и утврђени су оптимални услови рада. Перформансе хибридног система у највећој мери зависе од почетне концентрације галне киселине, дозе коагуланта и масе биоматеријала. За одређивање оптималних услова за уклањање галне киселине коришћен је потпун факторијални дизајн 23. Максимално уклањање галне киселине у води износило је 92,48 %, а постигнуто је при 20 mg L⁻¹ почетне концентрације галне киселине, 50 mg L⁻¹ алуминијум-сулфата као коагуланта, и 1,5 g активираних масе адсорбента од коре жира. Примена ових оптималних услова на градске отпадне воде за елиминацију органских материја показала је добре особине овог хибридног система третмана.

(Примљено 23. јуна, ревидирано 20. новембра, прихваћено 9. децембра 2022)

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