Removal of Phenol by Expanded Bed Airlift Loop Reactor

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ABSTRACT: *The exaggerated release of industrial wastes especially those containing phenol into the environment led to the contamination of both surface and groundwater supplies. In the present work a synergistic and combined system technique between three operations, adsorption of phenol via (rice husk or granular activated carbon GAC as adsorbents) together with stripping by airflow and advance oxidation via hydrogen peroxide as the oxidation agent, to evaluate the possibility of using a proposed new design for internal airlift loop reactor for removing the phenol from wastewater. The experiments were set up in a cylindrical Perspex column consisting of a transparent outer column having a 15 cm inside diameter and 150 cm height that included an internal draught tube of 7.5 cm and extending vertically to 120 cm top contains a bed having a dimension (7.5 x 30 cm) filled with adsorbent materials (rice husk, granular activated carbon GAC) and a volume capacity 25 liters. The experiments were conducted under the influence of both of the following variables air flow rate (2-20) (L/min), treatment time (5-60 min), the molar ratio of hydrogen peroxide to phenol,(1:10, 1:15, and 1:20)). The results showed the success of the proposed design with obtaining a removal efficiency (83%),(81%)when using GAC and the rice husk as adsorbent materials respectively, with a minimum remediation time 60 minutes, airflow rate of 18 L/min, and molar ratio(20) hydrogen peroxide to phenol. This study demonstrated that the proposed synergistic system could be utilized for the remediation of contaminated aqueous systems.*

KEYWORDS: *Loop reactor, Expanded bed, Hydrogen peroxide, Rice husk, Phenol.*

INTRODUCTION

The discovery of chemical pollutants in industrial and municipal wastewater is one of the most important tasks of water quality in recent years. The arrival of pollutants, whether natural or artificial, to the water streams by various means depending on their solubility in water as they are used and transported with rotated in the water cycle creates a serious threat to the aquatic environment [1].

The excessive release of industrial wastes especially those containing phenol into the environmental water the system represents the main threat to health where the oil refineries, pharmaceutical industries, pesticide industry, and petrochemicals in addition to the paper, and textile industries represent the main sources of phenolic compounds waste [2-4]. Phenol and its compounds with its degradation products are major and highly dangerous water pollutants due to a harmful poorly biodegradability and negative impact on living organisms and humans even at low concentrations they are toxic and carcinogenic in addition

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to severe side effects such as diarrhea and nervous breakdown [5-7]. The levels of presence of phenol and its compounds in drinking water and effluents were determined due to the difficulty of treating it biologically and its high toxicity level [8-10].

The permitted concentrations of phenol were determined according to the US Environmental Protection Agency and the World Health Organization to not exceed 1 μg /L and 0.002 mg / L in drinking water respectively, and not exceed the 500 μg / L in industrial water [9, 10].

Contaminated water forms complex mixtures requiring novel methods of treatment other than conventional methods. The flexibility and high capacity of airlift loop reactors in ensuring mass and heat transfer processes make them the most widely used in the biological and chemical industries [11-14] The rated energy consumption and low shear stress of cells in addition to low risks of pollution are considered from the sensitive operating conditions in biotechnology applications, (wastewater treatment, microalgae industry) all these requirements represent the features of the loop reactors in addition to engineering flexibility as a permanent factor to improve the effectiveness of these reactors. The presence of the draft tube in these reactors improves the regularity of the periodic pattern of flow inside the reactor and improves the mixing of the liquid in addition to reducing the collection behavior of the bubbles [13-16].

Phenol is one of the pollutants that reach the microscopic level when soluble in water which causes the difficult treatment of these wastewater containing microorganisms and non-biodegradable compounds due to their short retention periods depending on phenol degrading bacteria as *Tziotzios et. al.* have notified in many research about biological phenol removal [17].

Techniques for treating the contaminated phenol for wastewater are divided into two main parts: - decomposition as in oxidation processes in the presence of ozone, hydrogen peroxide, or manganese oxide, and the most common processes such as adsorption in porous solids, separation using membranes, exchange of ions and solvents [18-20]. Phenolic wastewater treatment processes are successfully applied *via* the implementation of integrated biodegradation of aerobic and anaerobic processes by using internal airlift loop reactors that also give a good performance of aerobic degradation in phenol, high mass transfer rates of liquid and (gas-liquid) which lead to promote the activity of the biodegradation rate [21,22]. When the reactor design ensures to predict the key of the best operational parameters (liquid circulation velocity, mixing intensity such as the suitable airflow providing at sufficient quantity) that will enable the successful implementation of many complex processes such as the removal of bio-sulfur from wastewater produced by the oil industry [23-27]. The need to find a simple design with pretty efficacy and a low cost of implementation as an alternative to the traditional methods of treating wastewater contaminated with phenol is essential to make the environment safer when recycled. In this study, the performance of a new design for the internal air loop reactor was evaluated in the synergy of three processes, namely stripping, oxidation and adsorption, and using two types of adsorbents:(rice husk, granular activated carbon GAC) to remove phenol from synthetic wastewater and with different operating conditions in terms of treatment period (5-60) minutes, airflow rates (7-20) L/min, in addition to the molar ratio of phenol to hydrogen peroxide (1 / 10,1 / 15 and 1 / 20).

EXPERIMENTAL SECTION

Materials

The rice husks utilized were acquired from (Al-Shanafia) farms for rice in southern Iraq. The common structure analyses of rice husks are recorded in Table 1. The rice husk was washed with surplus quantities of distilled water to strip the disband materials existent in the rice husk that brings of the field, then heated reaching to boiling to expel color and other fine impurities probably found in the rice husk and then dried until 105 ^{0}C for reaching to constant weight. The dried husks were put in desiccators until to use.

To prepare the stock solution of phenol (1000mg/L) that will be used to provide a sequenced five desired concentrations (10, 20, 50,100,150 mg/L) that which tested in this study, so the required phenol specification (chemical formula C6H5ClO, the molecular weight of 128.5 and 99.9% purity) was imported from Sigma-Aldrich company, one gram of phenol was consumed in one liter of distilled water to get the stock solution and adjust the acidity for each test solution through the addition of a suitable amount of 0.1 N hydrochloric acid or 0.1 N sodium hydroxide solutions.

The test runs were conducted with commercial Granular Activated Carbon (GAC)(8–20 mesh, 0.85–2.4 mm

Table1: Rice husk properties.

Compound	Composition wt %
SiO2	90.70
LOI	4.71
K2O	2.64
P2O5	0.73
CaO	0.61
MgO	0.25
Al2O3	0.13
Na2O	0.09
Fe2O3	0.06
TiO2	0.015
S.A (m^2/g)	57.5

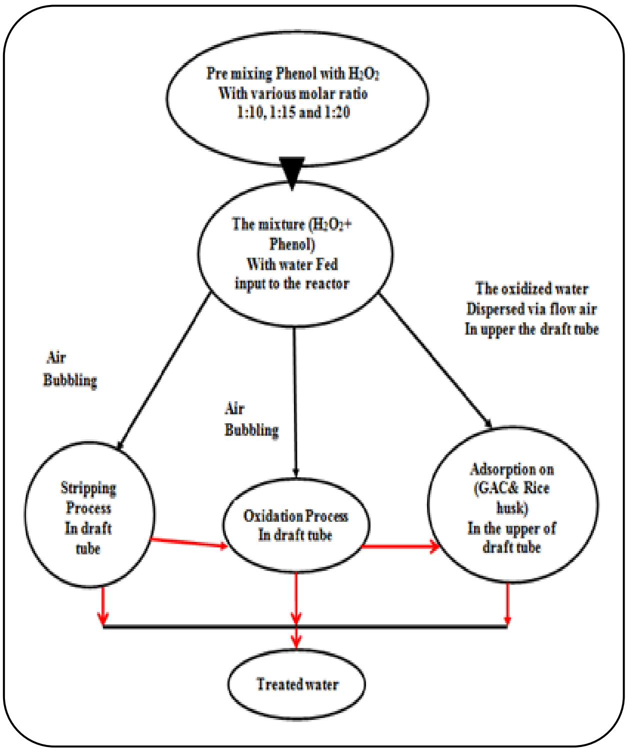


Fig.1: The sequence of the steps within the proposed design.

particle size, surface area 1050 m^2/g, solid density 1.153 g/mL) acquired from Sigma Chemical Co. The GAC was washed several times with distilled water to remove fine particles, then dried *via* oven to 105 ^{0}C.

Hydrogen peroxide was used at a concentration of 35% volume/volume obtained from Olloweg Ltd. Germany.

Experimental setup

The treatment scenario for phenol-polluted water according to the sequence of the steps within the proposed design can be clarified in Fig. 1. The treatment scenario for phenol-polluted water according to the sequence of the steps within the proposed design can be clarified in Fig. 1. The synergy with the details of the three operations performance (stripping, oxidation, absorption) and reactor geometry in terms of engineering dimensions, the material of reactor construction, bed location containing the packing of the adsorbent materials, air distributor specifications, all these specifications are shown in Fig. 2: (a, b, c, d, and f).

The reactor is a two concentric cylindrical Perspex tube the inner one called (draft or riser) with an internal diameter of 7.5 cm and a height of 120 cm open end from both ends, the center of the bottom end is the place of the gas distributor multi-hole with a free space 80% and the upper end is the place of the bed containing the padding of porous materials adsorbent (granular activated carbon or rice husks) with dimensions (7.5 * 30) cm, the draught tube is fixed via three supports, from the upper and lower sides in a way that makes it in the center of the outer tube (column) for any vertical height above the base.

The outer tube is 15 cm in diameter and height 150 cm, this makes the practical volume of the reactor 25 liters. The hydrogen peroxide is pre-mixed with the polluted water before entering the loop reactor via gravitational. The flow pumped from the fed tank prior to entering the reactor and that outed from it was controlled by using the gate valves as illustrated in Fig. 3. All experiments were conducted using various airflow rates ranging between (2-20) L/min and the working temperature not exceeding (30 ± 2^{0}C).

Experimental procedure

Contaminated water containing phenol was prepared at a concentration of 150 ppm in the feed tank T2 at the same time 1000 ml of hydrogen peroxide was added to 4 L of distilled water in the feed tank T1 located above the air loop reactor (RE) to prepare the oxidizing agent solution at a molar ratio 1 / 20 phenol to hydrogen peroxide at a temperature of 25 °C while placing 0.2 kg equivalent to 175 cm^3 volume of adsorbent material (activated granular carbon or rice husks) in the bed designated for padding at the upper end of the (riser) or draught tube. The polluted water with phenol was pumped from

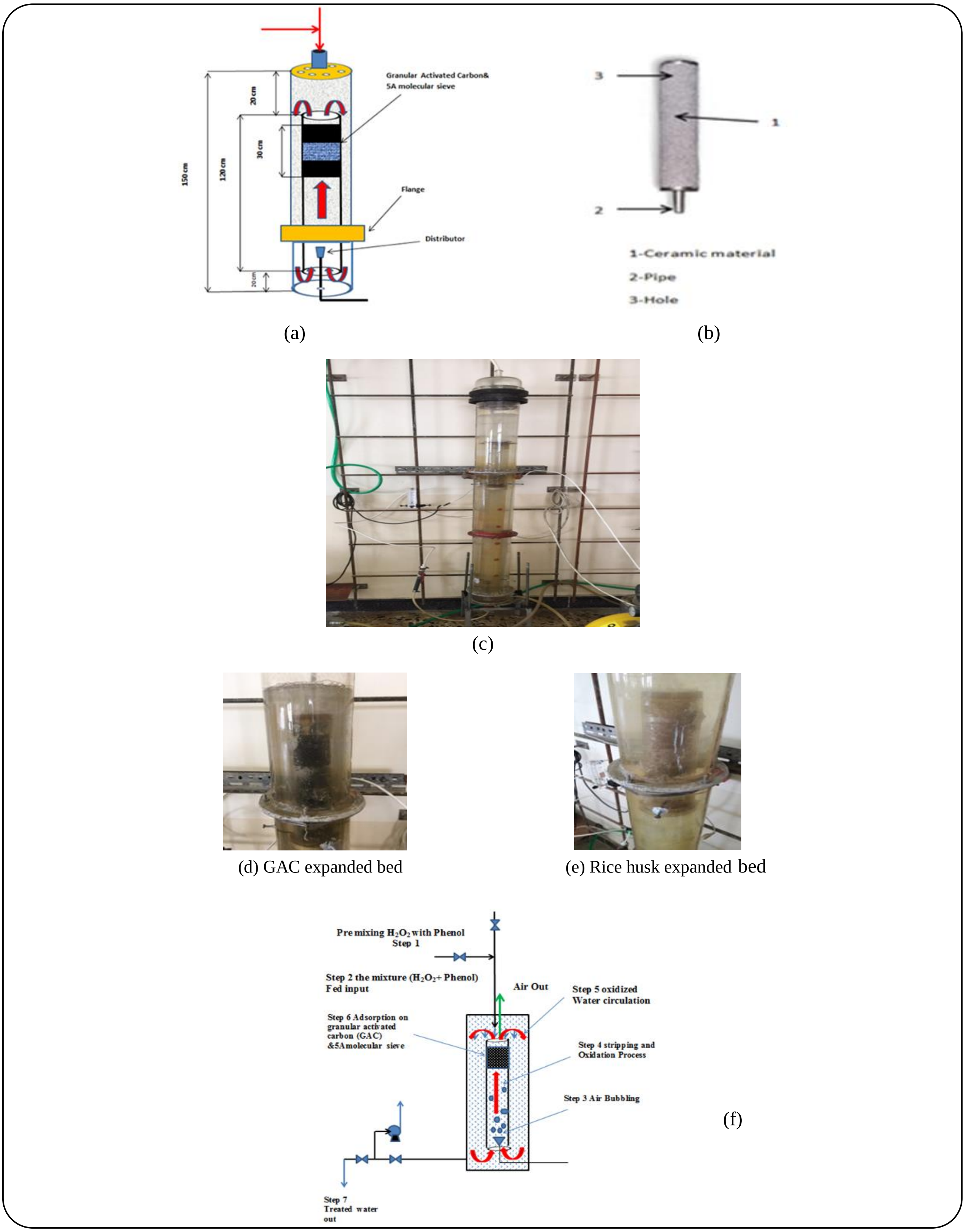
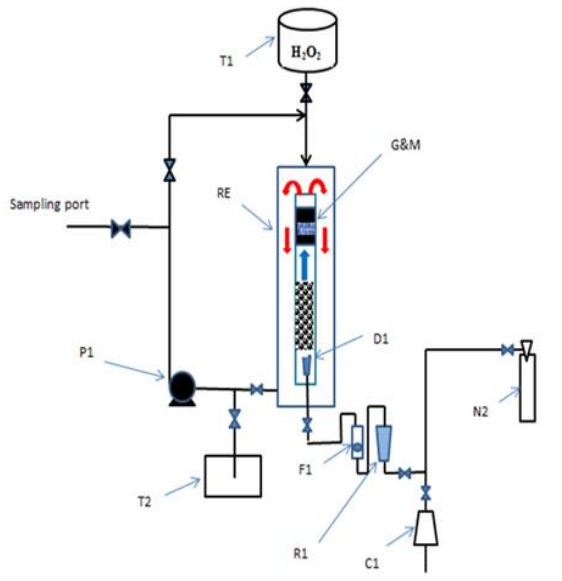


Fig.2: ***(a, b, c, d, and f). The synergy with the details of the three operations performance.***



⋈	Valves	P1	centrifugal pump	T1	hydrogen peroxide tank
R1	regulator	RE	reactor	D1	distributor
N_2	nitrogen cylinder	C1	compressor	T2	wastewater tank
F1	flow meter				

Fig. 3: Synergistic reactor diagram

the Feed Tank T2 with a constant flow rate 18 L/min while continuity of gravity flow of the hydrogen peroxide solution from the T1 tank to be mixing with polluted water pumped via the pump P1 under a pressure of 2 bar just before entering the mixture to the airlift loop reactor (RE). The operation of the process starts for 10 minutes until the flow of the system works steadily before starting the experiments. At the moment the air passes through the gas distributor into the reactor, the polluted water mixture with the oxidizing agent will disperse due to the force of the airflow creating a difference in the density of the liquid between the riser zone and downcomer zone resulting in the regular circulation of water between the draught tube and annulus inside the reactor, giving sufficient time to complete the processes of stripping and oxidation in addition to the adsorption obtained during the passage of dispersed water through the filling containing the adsorbents (granular carbon or rice husks), the treatment time of the polluted water can be controlled by controlling the flow rates for contaminated water stream or treated water through the valves in the pipelines in and out of the reactor, the treated water for 60 minutes was collected through the valve of samples assembly outside of the reactor. The system design was tested for one hour by taking 12 samples of treated water at a rate of one sample per five minutes, pending using a UV spectrometer (U-1800, Hitachi, Japan) to calculate the phenol concentration for each sample at a wavelength of 270 nm, the same steps were repeated working for different periods time ranging from 5 to 60 min with various airflow rates between 2-20 l/min in addition to different molar ratios of phenol to hydrogen peroxide (1/10 - 1/20), all samples of water have a pH between 3.5 to 4, where the percentage of the efficiency for removing phenol from polluted water was calculated via the following equation:

$$F = \left\{ \left(C_{IN} - C_{OUT} \right) / C_{IN} \right\} \times 100 \quad (1)$$

Where F represents removal%, C_{IN} and C_{OUT} are the initial and final concentrations of phenol respectively (mg/L) [3].

RESULTS AND DISCUSSION

The results reflect the effectiveness of the proposed design in terms of the efficiency of the removal of phenol by the synergistic effect of three processes of treatment (stripping, oxidation, adsorption) and different operational conditions in terms of treatment time, airflow rates, molar proportions of the pollutant to the oxidizing agent, in addition to a comparison between the efficiency of the adsorbent (granular activated carbon or rice husks). The two Figs. (4, 5) show an increase in removal efficiency by increasing both the residence time and the inlet phenol concentration the progressive progression of the increasing removal percent is reflected with the increase in the residence period up to 60 minutes for both types of adsorbent materials. When using the activated carbon as an adsorbent material for various inlet phenol concentrations 150ppm, 50ppm and 10ppm removal rates were 29.7%, 62.6%, and 83%, respectively.

In the case of using the rice husks as adsorbent matter, the results showed a clear convergence to the highest removal rate compared to carbon, which amounted to 81%, as well as the gradual increase in concentration and survival time as follows: 29.7% and 60.4%.

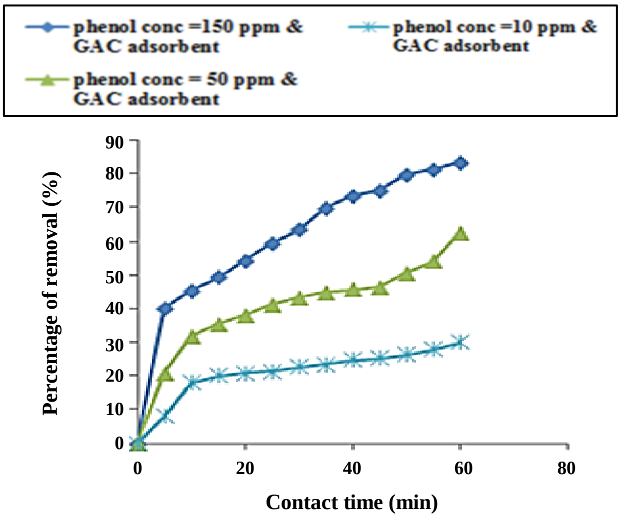


Fig. 4: Influence of residence time on the percentage of removal phenol with a different initial concentration of phenol and GAC as adsorbent material.

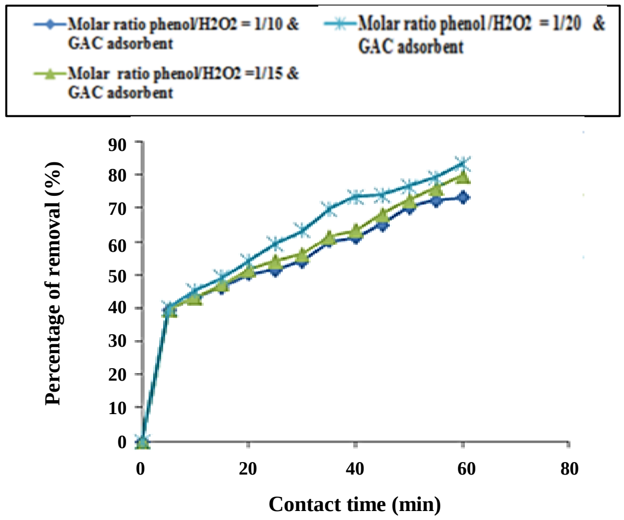


Fig. 6: Effect of treatment time on the percentage of removal with a different molar ratio of phenol to hydrogen peroxide and GAC as adsorbent material.

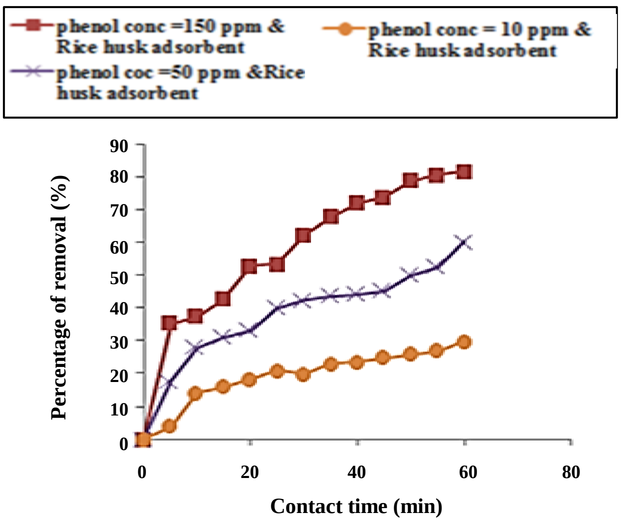


Fig. 5: Impact of retention time on the removal efficiency of phenol with various initial concentrations of phenol and using rice husk as adsorbent material.

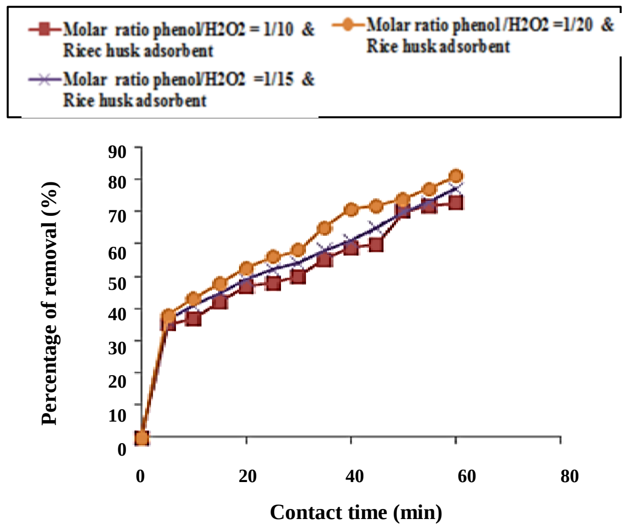


Fig. 7: Influence of treatment time on the percentage of removal with a different molar ratio of phenol to hydrogen peroxide and using rice husk as adsorbent material.

The high concentration of phenol in the feed solution of the reactor has the primary and pivotal role in increasing the driving force of the mass transfer process during the occurrence of adsorption and stripping processes, therefore the rate at which phenol molecules pass from the bulk solution to the particle surface of adsorbent or carrier material is the one that causes the marked increase in the rate of adsorption and stripping processes [28, 29].

This means that the gas phase is represented by the air diffused through the gas distributor, which will disperse the polluted water and strip the phenol from the water, while the adsorbents (rice husks and granular carbon) in the packed area at the top of the draft tube, represent the solid phase, where the adsorption process occurs by contaminated water during it passes and circulates through these adsorbents) [30].

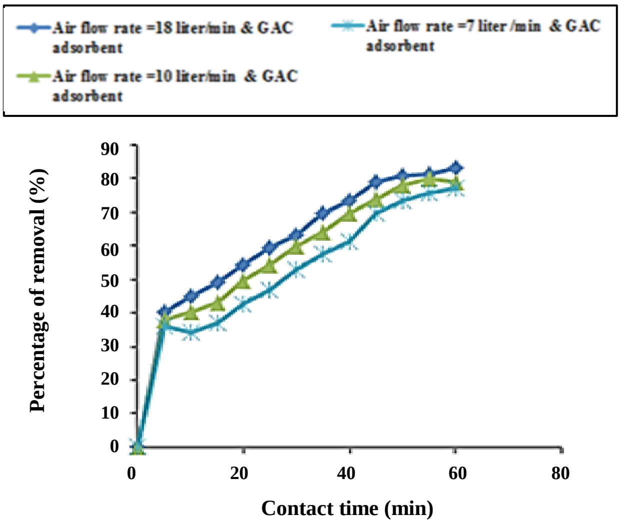


Fig. 8: Illustrate the effect of contact time on the percentage of removal with various airflow rates and GAC as adsorbent material.

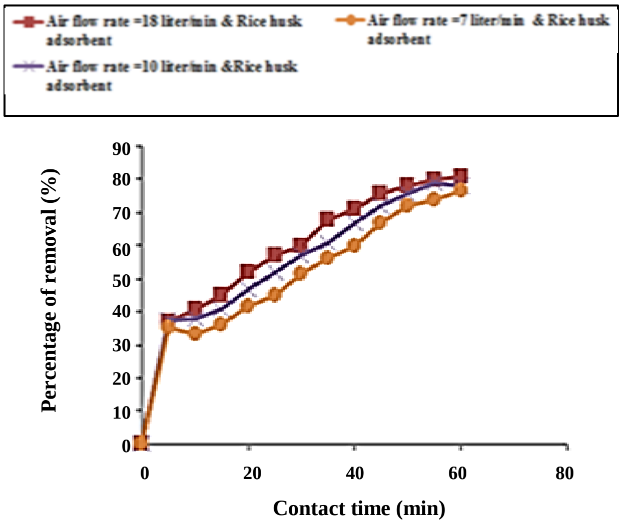


Fig. 9: Illustrate the influence of contact time on the percentage of removal with various airflow rates and rice husk as adsorbent material.

The increase in free radicals $OH^{\bullet}$ resulting from the disintegration of hydrogen peroxide has a decisive role in increasing the rate of destruction of phenol and this is embodied in obtaining the highest removal rate for both substances adsorbed with a very simple relative contrast and at the lowest molar ratio of phenol relative to the strong oxidizing agent hydrogen peroxide 1/20, as for the rest of the ratios The removal when using granular carbon as an adsorbent and at the molar ratios 1/10 and 1/15 were 73.2 and 79.9 respectively and when using rice husks and the same molar ratios above, the removal ratios were 72.6 and 0.77 respectively. One of the most important effective design factors in a bed construction for adsorbent material in the adsorption operation is the selection of EBCT what is known as the (empty bed contact time) which is known as the total size of the(rice husk)or (granular activated carbon) in the bed divided by the liquid flow rate and is usually expressed in minutes, the increases of the bed height leading to increasing the adsorption capacity due to available additional space for the phenol molecules to be adsorbed on these unoccupied areas, in addition, increasing the bed height will give a sufficient contact time for these molecules to be adsorbed on the activated carbon or rice husks surfaces[31-33]. Therefore the appropriate choice of contact time to take full advantage of the filling capacity system has an important role in choosing the dimensions of the bed (7.5 × 30) cm and putting a quantity of 0.2 kg of activated carbon or rice husks [34]. The process of mass transfer of the polluter (phenol) from contaminated water through the bed that containing an of adsorbent materials, whether activated carbon or rice husks, occurs in what is known as the mass transfer area, which is defined as the sufficient depth of the rice husks or activated carbon to reduce the pollutant concentration from the initial concentration of the final concentration at a specific flow rate. The process of recycling the liquid inside the reactor gives the whole opportunity to complete the adsorption process with high efficiency.

From Figs. 8 and 9, the very close effect of airflow on the removal efficiency for the two types of adsorbents (granular carbon and rice husks) is evident, with a slight preference for the flow rate of 18 liters per minute, where the removal efficiency was 83% and 81%, respectively. The results of the effect of three processes [oxidation, abstraction, and adsorption] in removing the organic pollutant (phenol) in a mono device, its manufacturing materials are inexpensive, the cost of installation and maintenance is low, in addition to the small area that it occupies and the short treatment period is a success factor in the device's economic work.

CONCLUSIONS

First: - The success of using the expanded bed packed with rice husks or granular carbon installed at the top of the inner tube of the air loop reactor as a synergistic design to perform three operations performance (abstraction, oxidation, and absorption) in single apparatus for treating polluted water with phenol and using a single oxidizing agent (hydrogen peroxide), where it reached the percentage of phenol removal when using granular carbon is 83% and 81% when using rice husks.

Second: - Determine the best scenario and operational conditions for the treatment system, which is the airflow rate of 18 liters/minute, 1/20 molar ratio phenol to hydrogen peroxide, and 60 minutes as total treatment time.

Third: - Setting the outputs of a less hazardous disposal system for chemical pollutants and safer before recycling and releasing water into the environment.

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