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SCIENTIFIC PAPER

UDC 547.56:628.3:502:66.081.3

PHENOL REMOVAL USING PULSATION BUBBLE COLUMN WITH INVERSE FLUIDIZATION AIRLIFT LOOP REACTOR

Article Highlights
- The study is an integrated system of a pulsation bubble column with an inverse fluidization reactor
- The effectiveness of the present design was tested with one of the chemical contaminants, namely phenol
- The method works for a variety of airflow rates, residence time, and molar ratio of phenol to H_2O_2
- Results showed that the phenol removal efficiencies are ~90%

Abstract

Phenol and phenolic compounds are omnipresent organic contaminants which are sent out to water bodies and wastewater systems produced from industrial processes, and they require specific attention due to their extraordinary features such as high toxicity, carcinogenic characteristics, and ability to accumulate, which affects the health of humans and the environment. In this practical study, the integrated system of a pulsation bubble column with an inverse fluidization air loop reactor was tested to remove phenol. The test platform was made and operated with a bubble column containing at its upper end an electrical solenoid valve engaged via at least two timers, and connected to the air loop reactor consisting of an outer rectangular tube and an internal draft tube by one-way valve, where the granular activated carbon is put as an adsorbent material in the annulus region between the inner and outer tube. The effects of various parameters [molar ratio of Phenol to H_2O_2 (1/10, 1/15 and 1/20), airflow rate (5-20 L/min), remediation time (5-60 min), initial phenol concentration (10-150 mg L^{-1}) have been studied. Removing 90% of the contaminated phenol as a result of this study may represent a partial solution to the ecological problem.

Keywords: phenol, pulsation bubble column, inverse fluidization, stripping, oxidation, adsorption.

Wastewater remediation *via* the removal of organic contaminants stills an earnest environmental and general problem. Moreover, in the face of tougher laws, polluted water has become a major concern and a priority for most industrial sectors. Phenolic compounds and its derivatives are usually found in the environment as a result of their vast industrial uses [1]. Several remediation techniques applied for removing the chemical, oil, and petrochemical wastes,

can be categorized as three types: physical, chemical and biological. The choice of the best technology is based on many factors, like the chemical nature of the waste, the impact of the effective cost, space occupied by equipment, discharge and reusing policy, permanent operation and side products. The main wastes produced from these categories are composed of hydrocarbon compounds, phenolic compounds, dyes, heavy metals, etc. [2-6]. Most of these compounds have pernicious ecological effects and may be labeled as serious waste; also, they are carcinogenic and have a malignant impact on human health. They are the largest contributors to the pollution of surface and groundwaters [7,8]. Centrifugal separation, adsorption, membrane separation, reverse osmosis, nanofiltration, ultrafiltration and micro-

filtration are particularly physical treatment methods; however, these methods have multiple restrictions, like producing great volumes of muds, expensive cost of equipment, in addition to installation and operation costs [9-12]. Air stripping is a technology that uses air flow jet for volatile organic contaminate removal from wastewater by increasing the contact surface area of the polluted water that is exposed to air; an organic pollutant which having low Henry's constant will require a much higher air/liquid ratio or steam stripping for efficient removal [13]. In the literature of wastewater remedy by adsorption, activated carbon adsorbents interact with adsorbates, keeping the adsorbate on the adsorbent surface and thus eliminating it from the aqueous phase. Adsorption *via* activated carbon is a prevalent wastewater treatment style because of its vigourous ability to remove organic materials even at low concentrations [14]. Powdered activated carbons may be directly used in the wastewater resource tanks, while granular activated carbons can be packed into a bed inside columns through which wastewater flows continuously [14]. The cheap carbon materials created from a residue material - which can noticeably minimize the cost of water remedy and supply an evaluation way for varied waste streams - have been studied through recent decades as another wastewater remedy choice [15--17]. The large consumption of chemicals and the costs of operation and maintenance in addition to skilled labor make the chemical processes such as oxidation, electrochemical deposition and advanced oxidation processes less economically feasible [18-25]. The advanced oxidation processes are characterized by the creation of reactive sorts, such as hydroxyl radicals (HO$^{\bullet}$), which are strong oxidation agents [26]. AOPs based on the production of HO$^{\bullet}$ have a higher oxidation potential than those of ozone and hydrogen peroxide, and are responsible for the complete oxidation for organic pollutants [27,28]. Although biological treatment processes use multiple types of algae, fungi, yeast, genetically modified organisms, and biologically active substances, some problems however, such as the difficulty of introducing the above materials into the reactor containing a filling in addition to the bypass phenomenon as a result of channeling, have led to limited use of biological treatment. Therefore, the need to find a new, efficient, inexpensive and environmentally friendly design is necessary to reduce the negative impact of hazardous pollutants on water and to preserve the natural water cycle in the environment. The main objective of the current research is to test the effectiveness of the new design that integrates the performance of two devices and two techniques, the bubble column having a solenoid valve controlling to the pulsation-time of treated water and the inverse fluidization loop reactor under the effect of three processes of oxidation, stripping and adsorption, with various scenarios and operating conditions.

EXPERIMENTAL

Materials and methods

The application of the proposed system required a synthetic wastewater model containing phenol as a polluting source. The chemical specifications of commercial formula phenol C_6H_5OH that was imported from Sigma-Aldrich which was employed in this study: the pureness is more than 99%, molecular weight (g/mol) 94.144, molar volume (cm^3/mol) 90, solubility in water (mg/L) 50-100 at 19 °C. Five synthetic solutions with varied successive concentrations (10, 20, 50, 100, 150 mg/L) were prepared from the prepared stock solution of phenol (1000 mg/L with acidic pH 3.5-4).

The experiments were carried out using a commercial granular activated carbon (GAC) (8-20 mesh, 3-4 mm particle size, specific surface area 1050 m^2/g, solid density 1.153 (g/ml)) obtained from Sigma-Aldrich. The GAC was washed many times by deionized water to eliminate the undesired particles, then drying in a furnace until reaching 105 °C. The oxidizing agent hydrogen peroxide with 35 vol.% concentration was obtained from HiMedia Laboratories Pvt. Ltd., Sigma Chemical Co.

Experimental setup

The proposed design model consists of two units (Figure 1). The first is the pulsation bubble column (PBC) with an effective volume of 4 L that was made from diaphanous acrylic that allows us to see what's going on inside. The outside diameter and height of the reactor were 5 and 210 cm, respectively. At the upper end of the PBC, a solenoid valve was placed and worked under the control of two timers which control the residence time for the contaminated solution, and created a pulsation flow during the period of opened and closed valve to transfer the treated water to the inverse fluidization air loop reactor (IFALR). The aqueous solution of phenol and hydrogen peroxide solution was premixed before entering the PBC via the two dosing pumps (P1 and P2 called SECO, chemical dosing type, country of origin: China). The compressed air was continuously fed through the compressor (C1) to the column with various flow rates of 2 and 20 L/min by an air sparger placed at 50 cm above the base of the column to

vigorously mix the phenol and hydrogen peroxide solution for giving sufficient time for the stripping and oxidation process to occur. During the opening and closing of the solenoid valve, a difference in pressure was created between the water in the PBC, and IFALR makes the water passes through a one-way valve into the loop reactor. The solenoid valve is reopened and closed at different times, up to stability condition in the water flow, and samples of treated water are taken at intervals through the valve model No. 1.
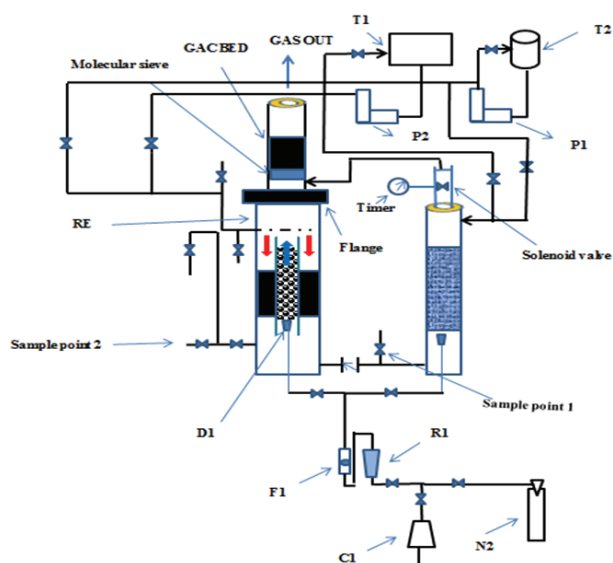

Figure 1. The design of the proposed treatment system;
▶◀ - valves, P1, P2 - dosing pumps, T1 - waste water tank,
F1 - flow meter, R1 - regulator, RE - reactor, C1 - compressor,
T2 - hydrogen peroxide tank, N2 - nitrogen cylinder,
D1 - distributor.

The inner tube for IFALR reactor is fixed in a manner that allows for creating empty space under the column to allow the water to circulate, in addition to its centering in the outer box for any distance extended above the base of the outer box, by three supports fixed *via* screws for each region: top, middle, and bottom of the reactor. Each support is comprised of 16 punctures, and every hole has a 1 mm diameter; these holes permit water to pass and do not allow the passage of activated carbon due to their small size (1 mm) compared to the size of the granulated carbon (3-4 mm). Screws participate in the installation of the draft tube from one side and loading the granulated carbon on the prop on the other. The direction of movement of the circulation of dispersed water will be from the top of the inner (draft) tube passing through the annulars to penetrate the activated carbon layer to complete the adsorption process for the remainder

of the phenol, with the occurrence of synchronization of both stripping and oxidation processes.

Experimental procedure

The integrated system operates in the following sequence.

The first step is a preparation of a synthetic solution contaminated with phenol at a concentration of 150 ppm, in the feed tank T1 with acidity ranging from 3.5-4. One liter of hydrogen peroxide is added to 8 L of deionized water in tank T2 to prepare a solution of hydrogen peroxide, depending on the molar proportion, as operational condition required 20 mol of hydrogen peroxide per 1 mol of phenol. 4.4 cm^3 from the assigned bed size for the adsorption process is filled with 5 kg of granular activated carbon. Installation of the flow rates for each pump: 0.3 L/min for contaminated synthetic wastewater for pump P2, and 0.2 L/min for hydrogen peroxide solution for pump P1. The two pumps operate at a pressure of 2 bar. For reaching stability, the system must be operating for an hour before conducting the experiments.

After installing the timer control for operating the solenoid valve (at 30 s to open the valve + 5 s to close), a sample is taken to test the water treated for the first experiment at the sample-taking point No. 1.

Five samples are taken within thirty minutes from the sample-taking point of the treated water No. 1, at the rate of one sample for every five minutes, to check the concentration of phenol *via* the UV spectrophotometer (Chrome Tech UV-VV-1100, UEBO9025, Japan) at a wavelength of 254 nm.

As a result of the difference in density of oxidized water due to the dispersion via the flowing air between the draft tube and the outer box, the treated water passes from PBC to IFALR at the same time the water forced to pass and recirculate through (GAC) padding at the inverse direction from top to bottom, to give full chance for efficient conjunction for stripping, oxidation and adsorption processes.

One sample every six minutes was taken from the sample checking point No. 2, to compute the concentration of phenol in the treated water.

The same steps were repeated by changing the following parameters.

A: The residence time ranged for all processes from 5 to 60 min;

B: various timers set the range for opening the solenoid valve (40, 50, 60, 120, 180 s), while the closed valve period remained 5 s during all experiments;

C: the rate of airflow ranged from 2 to 20 L/min;

D: three mole ratios were used in all experiments: 1/10, 1/15, 1/20 phenol to hydrogen peroxide.

The efficiency of the integrated system was calculated *via* the percentage of phenol removal through the following equation:

$$\eta = 100 \frac{C_{ph,in} - C_{ph,out}}{C_{ph,in}} \qquad (1)$$

where η is the phenol abstraction efficiency (%), $C_{ph,in}$ and $C_{ph,out}$ are the initial and final concentrations of phenol in ppm, respectively.

RESULTS AND DISCUSSION

The results can be discussed in two stages: the results obtained from the pulse bubble column during the occurrence of two processes (stripping and oxidation) and the effect of four variables.

A: The curing time is associated with the opening and closing times of the solenoid valve located above the bubble column;

B: the mole ratio of the pollutant to the oxidizing agent;

C: the rates of airflow;

D: the pollutant concentration in the feeding input;

The results are based on the models taken from the Model No. 1 pull point.

From Figure 2 it is clear that by increasing the shutdown time of the solenoid valve, the contact period increases, thus increasing the processing time, this giving sufficient time to the $^{\bullet}$OH free radicals resulting from the decomposition of hydrogen peroxide to attack the phenol ring with increased capability for completely mineralizing the phenol pollutant to H_2O and CO_2 [29]. This is consistent with findings (Haroun and Idris, 2009) in that the increase in the residence time leads to an increase in the reactor performance [30]. At the same time, the process of mass transfer of phenol from the liquid phase (water) to the gas phase (air) occurs in the stripping process. The increase in the removal percentage *via* increasing the concentration of the pollutant in the feed stream is due to the increase in the driving force of the mass transfer process generated by partial pressure reduction on the permeate side, in the stripping process [31,32]. The percentages of removal of phenol for thirty minutes for 10, 50, 100 and 150 ppm were 22.7, 43.2, 60.8 and 63.3%, respectively.

Figure 3 shows that the mole ratio of hydrogen peroxide to phenol is the key to controlling the dissociation rate of phenol in the oxidation reaction, based on the literature for oxidation processes in the presence of a single oxidizing agent (hydrogen perox-
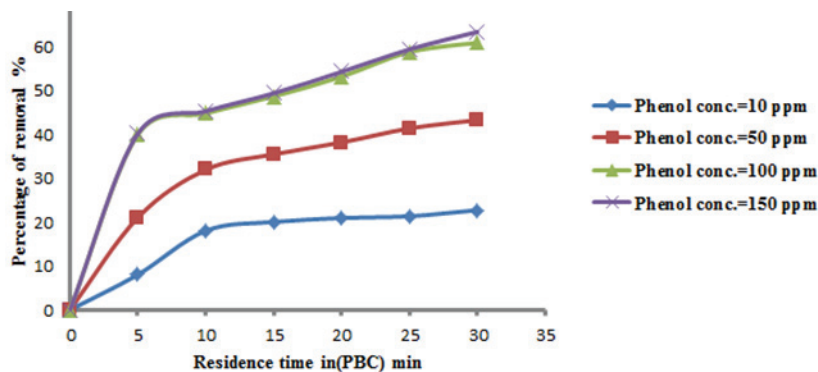

Figure 2. Impact of time curing on the percentage of elimination of phenol, for different concentrations of phenol inputs.
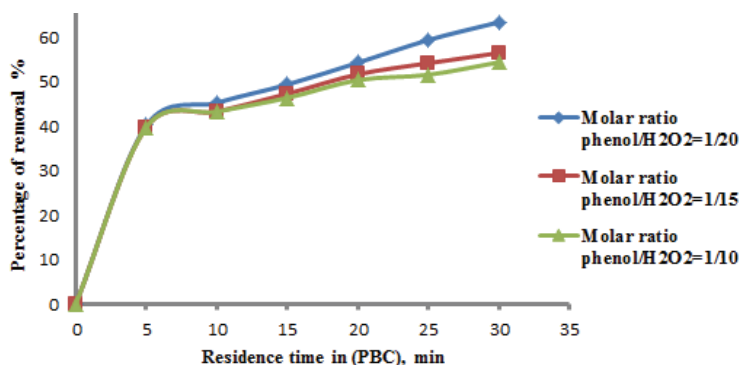

Figure 3. Influence of the remediation period on the percentage of removal, for a different range of molar ratios of phenol to hydrogen peroxide in PBC.

ide) [33]. Redoubling the molar number of hydrogen peroxide approximately twenty times twice the number of moles of phenol, with a 30 min remediation period, showed that the removal rate did not exceed 63.3%. The increase or decrease in this molar ratio caused in both cases a decrease in the dissociation rate of phenol, as the increase leads to what is known as the sweeping or scavenging the free radicals of hydrogen peroxide, where the decrease causes the numerical diminution of the free radicals that attack phenol and, consequently, the rate of dissociation in the oxidation reaction. The increase or decrease beyond the optimal concentration leads to decreasing the process efficiency due to the scavenging effect of the HO$^\bullet$ [29]. The ascending order of percentages of phenol removal for a treatment period of approximately 30 min, according to molar ratios 1:10, 1:15 and 1:20, are 56.4, 54.3 and 63.3%, respectively.

Figure 4 shows the limited effect of the change in the rate of airflow on the percentage of phenol removal for a 30-min treatment period, where at the flow rate of 5 L/min, the percentage of removal was approximately 52%, while at 18 L/min the percentage for removal was approximately 63%, at the same treatment period of 30 min. The difference in removal efficiency does not exceed 16%. This confirms the limited effect of a relative change in flow rate on the percentage of elimination efficiency. The percentage of removal for phenol with airflow rates 5, 10 and 18 L/min within 30 min remediation time was 52.8, 59.7 and 63.3%, respectively.

The second stage of the results appears in the IFALR reactor that are represented via the samples taken from checking point No 2. These results are illustrated by the continuous effect of both stripping and oxidation processes, in addition to the new important factor represented by the adsorption process, with continuous changing in operating conditions (treatment period, concentration, airflow rates and the molar ratio of the pollutant to the oxidizing agent) [34].

From Figure 5 the effect of adsorption on the phenol removal rate for different phenol concentrations in the entering feeding stream appears evident. Furthermore, when concentration was 150 ppm, the removal rate was 89% for a 30-min treatment period in the second stage remedy, occurring in a reverse fluidization reactor (IFALR), *i.e.*, with a total period of stay in the treatment system of 60 min, with the synergy of oxidation and stripping processes, in addition to the adsorption process which has the main effect in the work of the IFALR reactor. The dispersed water in the inner column of the reactor (draft) creates a differ-
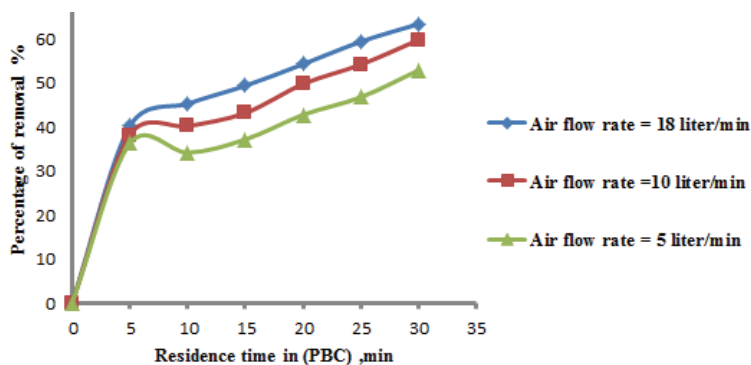

Figure 4. Impact of residence time on the percentage of phenol elimination with various airflow rates in PBC.
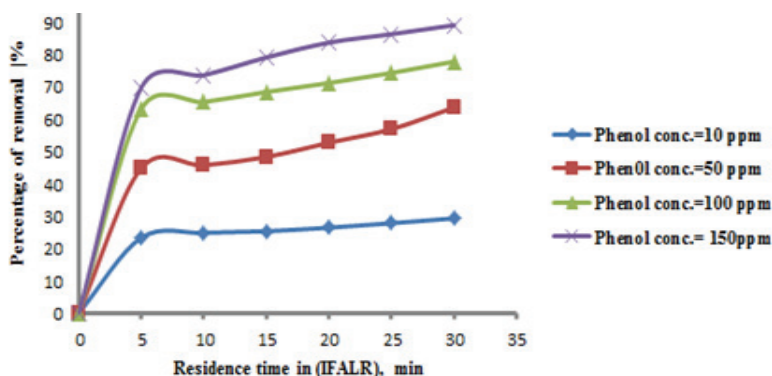

Figure 5. Influence of remediation time on the percentage of elimination of phenol with several initial concentrations of phenol, during stripping, oxidation, and adsorption processes in IFALR.

ence in density between the water treated in the draft tube and the water present in the annulars, forcing the water to circulate in the direction from the top, passing through the packed bed padding with activated carbon (inverse fluidization), where the adsorption of phenol takes place depending on the principle of the mass transfer *via* the concentration difference of phenol between water and carbon, which gives the opportunity to increase the efficiency of the process of elimination of phenol in the contaminated water. Many researchers confirmed that the adsorption process is affected by the gas and liquid velocities, particle size, and initial static bed height, and the initial phenol concentration, flow rate, and bed particle size as well [34,35]. The removal rates of phenol in 30 minu for 10, 50, 100 and 150 ppm were 29.29, 63.6, 77.8 and 89.09%, respectively.

The rate of the oxidation reaction depends on the optimum ratio between the pollutant and the oxidizing agent (hydrogen peroxide). Figure 6 shows that the highest removal efficiency of 89.1% was obtained in the second stage of the water treatment, which occurred in the reverse liquefaction reactor (IFALR) when the ratio of hydrogen peroxide to phenol was twenty, and this is due to the appropriate number of free radicals generated by the dissociation of the

oxidizing agent, which was not too many that it caused the sweep of free radicals and not too few to lead to a decrease in the oxidation rate, so as to ensure the best and highest efficiency of phenol removal [29]. It is noticed that the removal percentage of phenol at 30 min in the second remedy stage in IFALR with a serial molar ratio of 1:10, 1:15 and 1:20, and total residence period in the treatment system of 1 h, was 74.2, 80.1 and 89.09%, respectively.

Reverse liquefaction reactor (IFALR) used in the design of the second stage of the proposed treatment system, uses air to disperse and recirculate the contaminated water between the two parts of the reactor, the riser and downcomer, which leads to control of design factors, such as surface area of contact, mixing intensity, and residence time. Figure 7 shows that the airflow rate of 18 L/min and the use of 5 kg of activated carbon in building the optimum dimensions of the filling bed for getting the highest rate for phenol mass transfer from the liquid state (polluted water) to the solid (activated carbon) [36], has achieved an approximate removal rate of 90% in a treatment period of 30 min in a reverse liquefaction reactor, and the total remedy time was 1 h in the treated system. The removal rates of phenol in 30 min for 5, 10 and 18 L were 80.1, 86.09 and 89.09%,
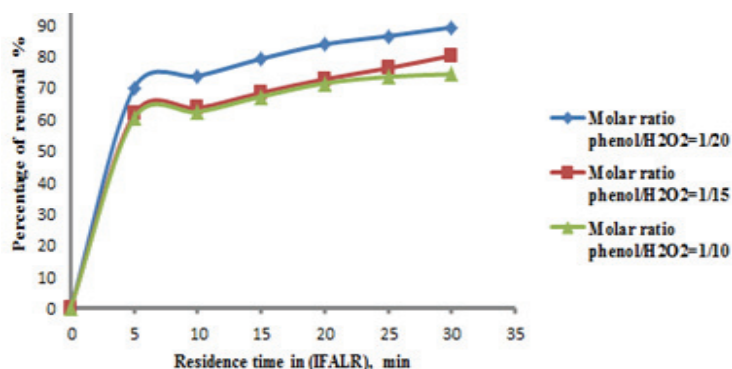

Figure 6. Impact of the remediation period on the proportion of phenol elimination for different molar ratios of phenol to hydrogen peroxide in IFALR.
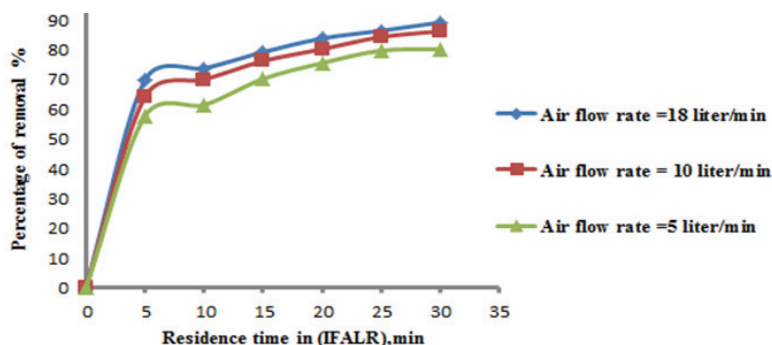

Figure 7. Effects of treatment time on the proportion of phenol removal for various airflow rates in IFALR.

respectively. The difference in efficiency percentage does not exceed 0.1%. This confirms the limited effect of flow rate on the elimination percentage.

CONCLUSIONS

From the results of the research, it is possible to conclude the following.

The success of the design idea based on the use of a solenoid valve with a bubble column to determine the residence time and transport of polluted water without the need to use a pump in what is known as PBC.

The success of the idea of combining PBC with the inverse fluidization reactor IFALR was achieved in one treatment system.

Three processes were achieved: stripping, oxidation, and adsorption.

Three techniques were implemented: pulsation propulsion, inverse fluidization, and use of a single oxidizing agent.

The best operating conditions for the removal system were checked.

The clearance rate of 89% was obtained in the following operational conditions:

A: one hour of total processing time;

B: 18 L/min airflow rate;

C: mole ratio of 1/20 of phenol to hydrogen peroxide.

With the successful removal of phenol, the present treatment system can be used to remove various types of organic pollutants in water.

The system can be used to reduce the risks of organic compounds that have a negative impact on the environment.

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UKLANJANJE FENOLA POMOĆU PULZACIONE BARBOTAŽNE KOLONE SA INVERZNIM FLUIDIZACIJSKIM AIT-LIFT REAKTOROM SA UNUTRAŠNJOM CEVI

Fenol i fenolna jedinjenja su sveprisutni organski zagađivači, koji se ispuštaju u vodo-tokove i sisteme otpadnih voda proizvedenih u industrijskim procesima. Ovaj problem privlači pažnju zbog karakteristika, kao što su visoka toksičnost, kancerogena svojstva i sposobnost sakupljanja koje utiče zdravlje ljudi i životnu sredinu. U ovoj praktičnoj studiji, integrisani sistem pulzacione barbotažne kolone i inverznim fluidizacionim air-lift reaktorom sa sa unutrašnjom cevi testiran je za uklanjanje fenola. Probna platforma napravljena je tako da je barbotažna kolona sa solenoidnim elektro-ventilom na gornjem kraju, koji je uključen preko najmanje dva tajmera, povezana sa air-lift reaktorom parvo-ugaonog poptrčnog preseka sa unutrašnjom cevi dok je anularni proctor ispunjen zrnas-tim aktivnim ugljem. Efekat različitih parametara (molski odnos fenola prema H_2O_2 1/10, 1/15 i 1/20, protok vazduha 5-20 L/min, vreme tretmana 5-60 min, početna koncentracija fenola 10-150 mg/L). Uklanjanje 90% fenola kao rezultat ove studije može predstavljati delimično rešenje ovog ekološkog problema.

Ključne reči: fenol, pulsni stupac mehurića, inverzna fluidizacija, skidanje, oksi-dacija, adsorpcija.