



## An Experimental Investigation of Synergistic Pulsation Bubble Column with Inverse Fluidized Loop Reactor for Removing Chloroform from Wastewater

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### PAPER INFO

#### Paper history:

Received 04 March 2020

Received in revised form 21 August 2020

Accepted 03 September 2020

#### Keywords:

Stripping

Oxidation

Pulsation Bubble Column

Adsorption

Inverse Fluidization Airlift Loop Reactor

Chloroform

### ABSTRACT

In this study, the feasibility of using the developed design for the removal of organic pollutants from wastewater was examined. The design includes the integration of the work of both pulsation bubble column (PBC) and the inverse fluidization airlift loop reactor (IFALR). The experimental podium was fabricated and installed which that consists of a bubble column with a diameter of 5 cm and a height of 210 cm, contains at the top a solenoid valve which is electrically turned via at least two timers, and its connection with the loop reactor by a one-way valve. The loop reactor consists of an outer rectangular tube with dimensions (29 cm long x 15.5 cm wide x 150 cm high) and an internal draft tube with 9 cm diameter and 120 cm long as granular activated carbon is put as an adsorbent in the annulus region between the inner and outer tube. Experiments were conducted using one of the organic pollutants namely chloroform, with a work scenario that includes changing both the airflow rate (2-20) liters/minute, the total survival time of the treatment (5-60) minutes, the molar ratio of the chloroform pollutant to the oxidizing agent of hydrogen peroxide (1/10 - 1/20). The results showed removal efficiency near to 89%, and it gives an indication of the success of the proposed design, with the possibility of recycling the treated water and releasing it to the environment due to the low risk of the organic pollutant in it.

doi: 10.5829/ije.2020.33.11b.01

## 1. INTRODUCTION

It is known that there are trace amounts of chloroform accompanying water of all types whether, drinking water, surface water, tap water, and domestic sewage, cooling water in industrial plants, groundwater, as a side product in the chlorination stage during the remedy process for wastewater to decrease the hazard of diseases due to microorganisms or viruses [1]. Through the previous periods, chloroform was utilized as a doped for operations and in many products such as cough suppressants and toothpaste, in addition, that chloroform is used also in industrial processes producing many typically materials of various uses such as lubricating oils, cleaning solvents, bleaching paper, refrigerants, rubber, intermediate materials in the pharmaceutical industry for herbicides and fungicides [2]. Emission of

chloroform causes damage to the environment, in particular, the destruction of the ozone layer [3]. In addition to being toxic and carcinogenic; it represents a direct threat to health and a direct cause of kidney and liver cancer and damage that may affect the nervous and reproductive system [4, 5]. Several studies carried out regarding treatments methods used to remove hydrocarbon compounds of low molecular weights including chloroform have been classified into three methods, firstly the physical methods like adsorption via activated carbon or by many different other adsorbents (Nano adsorbents or zeolite) [6], agglomeration, Flotation's, abstraction, and ion exchange, [7-10] secondly the chemical methods, such as oxidation with many catalysts and finally, the conventional biological methods have failed to abstract poisonous compounds or recalcitrant organic pollutants [11-13]. Therefore, one of

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the most important and dangerous challenges at the present time is to protect water resources via removing dangerous and toxic organic materials by unconventional, effective and inexpensive methods [14, 15]. Each of the three mentioned methods has advantages and at the same time has negative aspects, so the need has become necessary to find cheap and effective alternative methods that collecting the majority of the advantages of those operations with one device [16-28]. The aim of this research is to test the effectiveness of a proposed new design for treating water-contaminated organic compounds by combination of a bubble pulsed column with an internal air loop reactor with different operating conditions in terms of treatment time (5-60) minutes and various airflow rates (2-20) L/minute and the molar ratio of chloroform to hydrogen peroxide (1/10 - 1/20).

## 2. EXPERIMENTAL

### 2. 1. Materials and Methods

**2. 1. 1. Materials** The essential of prepared a stock chloroform solution by dissolving one gram of chloroform (Sigma-Aldrich, Inc.) chemical structure  $\text{CHCl}_3$ , with more than 99.8% pureness, its molecular weight 119.4, while the dissolved amount in water is 0.005 mL/mL (v/v) in a liter of deionized water to create a stock solution with a concentration of 1000 mg chloroform/L, then prepare the required experimental concentrations of 10, 20, 50, 100 and 150 mg/L. The acidity of the stock solution was regulated to the desirable acidity (7.5-8) via using 0.1M for both HCl and NaOH solution. The trade granular activated carbon (GAC) with a surface area of 1050 square meters per gram and a solid density of 1.153 g/mL, 0.8–2 mm particle size, it's imported by Sigma (Chemicals CO.). It is treated prior before its uses, by means of washing it multiple times with deionized water and drying it in an oven at a temperature of 105 °C. The strong oxidizing agents which have a 35% concentration of hydrogen peroxide generating the shattered free radicals for dissolved organic pollutants in water was supplied by GmbH Germany.

### 2. 2. Experimental Setup

The integrative treatment system consists of two units, the first being the pulsation bubbles column (presented in Figure 1) which was made by using transparent acrylic material of an engineering dimensions embodied via the length of tube 210 cm, the inside diameter (ID) 5 cm, and thickness 0.3 cm, equipped with a solenoid valve located at the top end and it is working periodically opens and closes depending on the electrical timer. The stripping and oxidation processes are performed simultaneously when the wastewater mixture with hydrogen peroxide is fed to the column via a twin dosing pump (P1 and P2). The

compressed air is passed via the compressor (C1) through the air sparger located 50 cm above the base of the column to cause the violent mixing of polluted water with hydrogen peroxide this happens during the solenoid valve opens and given sufficient time for complemented the two processes of stripping and oxidation.

During the period of closing the valve, air will disperse the oxidized water resulting in a pressure difference between the pulse-bubble column and the reverse fluidizing reactor causing the flowing of oxidized water from the pulse- bubble column to the inverse Fluidizing reactor passes through a one-way valve located on a perforated pipe that connects them as shown in Figure 2a. The process of opening and closing the solenoid valve is repeated periodically to reach the stability condition where samples for the treated water are taken at the point of taking samples 1.

The second unit is the inverse fluidizing reactor that consists of a diaphanous rectangular outer column and a draft tube having specifications given in Figure 2b. The draft tube extends vertically from a center of the outer rectangular column leaving a distance allowing the oxidizing water to recirculate between the inner draft tube and the outer rectangular box, the space creating between the draft tube and the outer column was partially filled with a granular activated carbon to working as an adsorbent material.

To create a space between the draft pipe and the bottom of the outer box, three supports are distributed across the top, middle and end of the reactor fixed with screws placed on the outer sides of the reactor. Every support contains sixteen holes, each hole has a diameter of 1 mm, these holes allow water to pass through it and do not allow granulated carbon to pass due to its small size 1 mm compared to the size of the granulated carbon 3-4 mm.

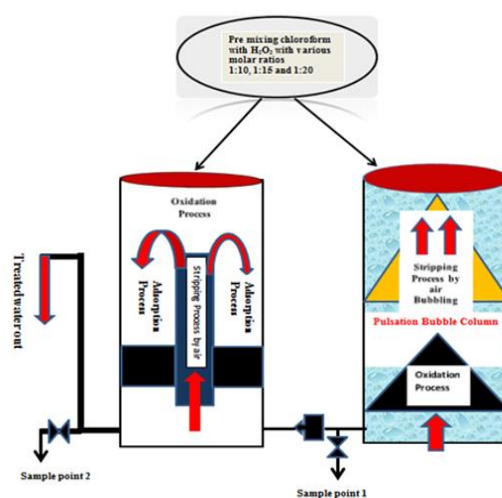
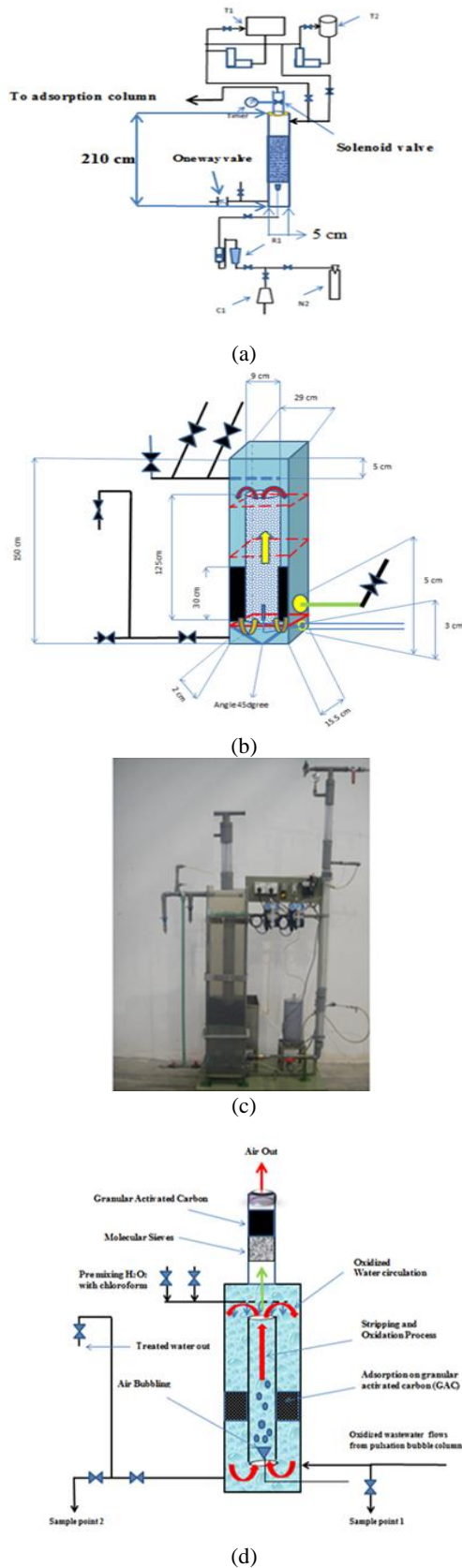


Figure 1. The integrative treatment system



**Figure 2.** Planning and sequence of an integrated system (a, b, c and d)

Pillars cooperate on installing the inner tube from one side and loading the granulated carbon from another side. There is a port for inserting the polluted water mixture and hydrogen peroxide into the inverse fluidization loop reactor. P1 and P2 pumps (called SECO, chemical dosing type, country of origin China) use batches to feed the polluted water and hydrogen peroxide to both the pulsed bubble column and the reverse fluidizing reactor through the same duration as shown in Figure 3.

| Valves |                        |
|--------|------------------------|
| R1     | regulator              |
| N2     | nitrogen cylinder      |
| P1, P2 | Dosing pumps           |
| RE     | reactor                |
| D1     | distributor            |
| T1     | waste water tank       |
| C1     | compressor             |
| F1     | flow meter             |
| T2     | hydrogen peroxide tank |

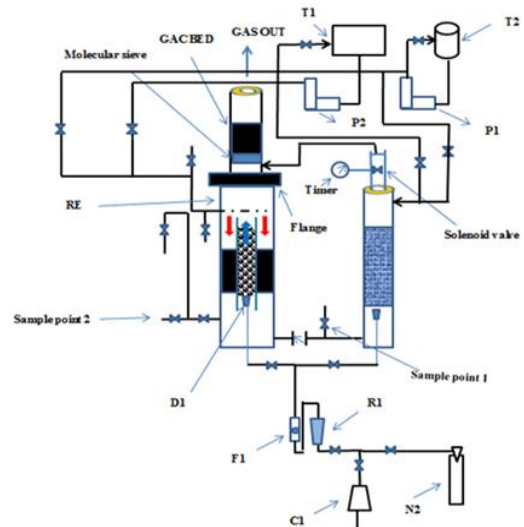
**2. 3. Experimental Procedure** The operation process is carried out by the following steps - :

First: - Preparation of water contaminated with chloroform at a concentration of 150ppm, and an acidity ranging from 7-7.5 in the feed tank T1.

Second: - Preparing a solution of hydrogen peroxide-based on the molar ratio as an operational condition is required 20 moles of hydrogen peroxide to one-mole chloroform, where one liter of hydrogen peroxide is added to 8 liters of deionized water in tank T1.

Third: - The assign bed for the adsorption process is filled with 5 kg of granular activated carbon, equivalent to 4.5cm<sup>3</sup> in size.

Fourth: - Determination of flow rates for each of the contaminated water pump P2 at 0.3 L/min, and the pump of hydrogen peroxide solution P1 at 0.2L/min, and the two pumps operate at a pressure of 2 bars.



**Figure 3.** Schematic diagram of an experimental device

Fifth: - The system should be operational for an hour for reaching stability, prior to conducting the experiments.

Sixth: - The first experiment is being tested by installing the timer controlling the operation of the solenoid valve as follows, 30 seconds to open the valve + 5 seconds to close the valve.

Seventh: - Five samples of treated water are taken in the pulsed bullous column at a rate one sample each every six minutes at the point of withdrawal of samples No. 1, to measure the concentration of chloroform using the UV spectrophotometer (UV-1800, Hitachi, Japan) and at a wavelength of 254 nanometers.

Eighth: - After passing the treated water from the pulsed bubble column to the inverse fluidization reactor, and as a result of the difference in density due to the dispersion of the oxidized water via the flowing air between the draft tube and the outer box, the water recycling and forcing to pass through the granular carbon from the top to the bottom to cause adsorption process and give the entire opportunity to complete the abstraction and oxidation processes.

Ninth: - Five samples are taken at the rate of one sample every six minutes to measure the concentration of chloroform in the treated water at the point of drawing the samples No. 2.

Tenth: - The previous steps will be repeated by changing the following- :

a: The total time of the operation is (5-60) minutes

b: - Solenoid valve opening times (40, 50, 60,120,180) seconds while valve shutdown time remains 5 seconds for all experiments.

c: - Airflow rates range from (2-20) liters per minute.

d: - molar ratios of chloroform to hydrogen peroxide (1/10, 1/15, 1/20).

Eleventh: - The percentage of chloroform removal for the whole process is calculated by the following formula: -

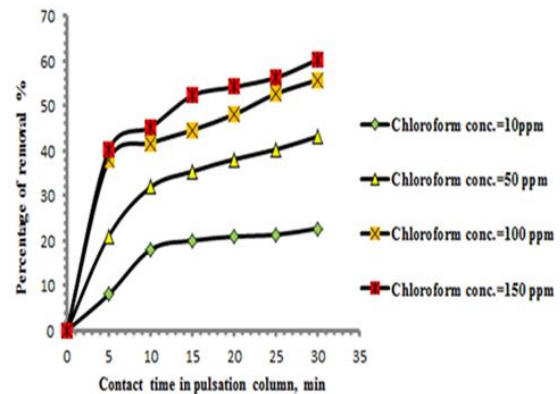
$$\eta = \frac{C_{INPUT} - C_{SAMP2}}{C_{INPUT}} \times 100 \quad (1)$$

where  $\eta$  is  $\text{CHCl}_3$  elimination efficiency (%),  $c_{input}$  and  $c_{samp2}$  are the primary and final concentrations of  $\text{CHCl}_3$  in ppm, respectively.

### 3. RESULT AND DISCUSSION

Figure 4 illustrates the effect of the change in contact time due to the different timing of opening the solenoid valve which locates at the upper of the pulse-bubble column during the occurring of the stripping and oxidation processes on the percentage for the removal efficiency of chloroform at different concentrations for chloroform in the contaminated water which feeding the pulse-column.

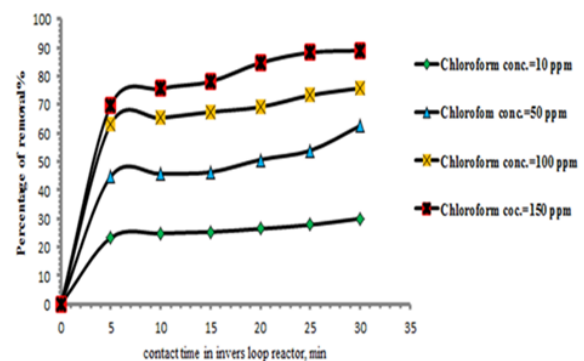
The results indicate an increase in removal efficiency with increased contact time and get maximal removal rate



**Figure 4.** Impact of contact time on the proportion of abstraction of  $\text{CHCl}_3$  with various initial concentrations of  $\text{CHCl}_3$

at the highest concentration of polluted chloroform that feeding the pulsation column. The elimination rate of  $\text{CHCl}_3$  in 30 minutes for, 10, 50, 100 and 150ppm were 22.7, 43.2, 55.8 and 60.3%, respectively. The explanation of this case is the high concentration of chloroform creates the highest rate of driving force for the mass transfer in the stripping process between the pollutant concentration in the liquid phase (water) and the concentration of chloroform in the gas phase (air) [29, 30]. At the same time, the longest contact time gives the complete opportunity to the free radical that produce from decomposing hydrogen peroxide to attack chloroform and decompose it into substances that are fewer hazards to the environment.

Figure 5 shows the effect of the retention time in the inverse fluidization reactor of the various concentrations of the pollutant that feeding the process during the occurring of stripping, oxidation and adsorption processes on the removal efficiency of chloroform. To enrich the process of removing and dismantling chloroform and increasing the efficiency of removal



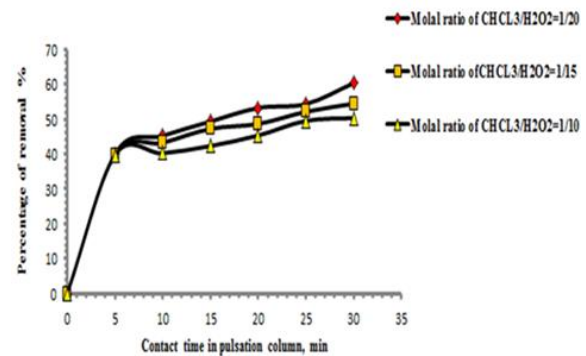
**Figure 5.** Impact of contact time on the percentage of removal of  $\text{CHCl}_3$  with various initial concentrations of  $\text{CHCl}_3$  during the occurring of stripping, oxidation, and adsorption processes in reverse fluidizing reactor

achieved in the pulsation bubble column. The inverse fluidization reactor is designed to ensure occurring the adsorption process of the pollutant or the compounds resulting from its dissociation in the oxidation processes that previously occurred in the pulsation bubble column.

The dispersed contaminated water in the reverse liquefaction reactor is forced to circulate as a result of the density difference between the contaminated water dispersed in the draft tube and the water that in outer box which will passing through the granular carbon layer to adsorb the chloroform or the remaining compounds from the disintegration of the chloroform by free radicals, reaching to reducing the hazard of pollutant on environmental and elevate the efficiency of removal chloroform, subsequently the removal rate of  $\text{CHCl}_3$  in 30 minutes for, 10, 50, 100 and 150ppm were 30, 62.6, 75.8 and 89%, respectively.

The increasing removal efficiency of chloroform for all types of concentrations that fee to the reactor, with increasing contact time is due to three factors. The first is the continued presence of the driving force for mass transfer due to the difference in the concentration of the pollutant (chloroform) between the two phases, (contaminated water) liquid phase and (air) gaseous phase and thus cause continuity of stripping process [30]. The second factor, it is the availability of large numbers of free radicals resulting from the disintegration of hydrogen peroxide. Thus, the continuation of the oxidation reaction to the disintegration of chloroform [31, 32]. The third factor is the continuation of the recycling process of the contaminated water between the inner and outer tubes of the reverse fluidization reactor; which gives the opportunity to continue adsorption of chloroform occurs when it's passing through the granular carbon bed.

The oxidation reaction using hydrogen peroxide as an oxidized agent leads to an increase in the rate of oxidation depended on optimum molar ratio for the pollutant to oxidized agent, it became clear through Figure 6 that the highest removal rate of chloroform 60% was obtained in pulsation column when imposing the suitable ratio of hydrogen peroxide (twenty times more than chloroform) in the synthetic polluted water fed for pulse column with a treatment time of 30 minutes. The result shows that the percentage removal of  $\text{CHCl}_3$  at 30 minutes for molar ratio 1:10, 1:15, 1:20 were 50.3, 54.4 and 60.3%, respectively. The availability of the optimum molar ratio for hydrogen peroxide to the pollutant is the key to successfully controlling the oxidation process due to the preparation of appropriate free radicals to obtain the highest removal rate. The low number of free radicals reduces the dissociation rate of chloroform and thus reducing the removal efficiency, while if the number of free radicals more than the optimum numbers this cause a phenomenon that is known as sweeping the free radicals that lead to a lower percentage of chloroform removal



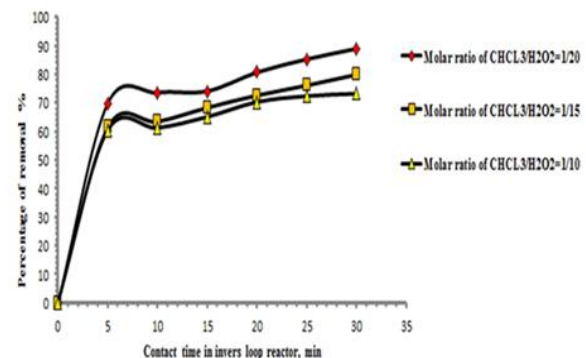
**Figure 6.** Influence of residence time on the proportion of removal at several molar ratios of  $\text{CHCl}_3$  to hydrogen peroxide in the pulsation reactor

[33]. Figure 7 shows the efficacy of the adsorption process associated with enough time opportunity to complete the stripping and oxidation processes, on the removal percentage of chloroform from contaminated water in the loop reactor.

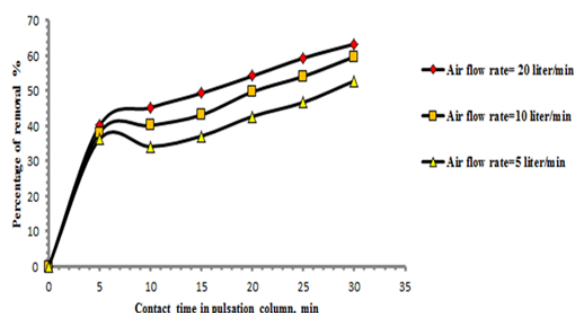
It is observed that the percentage of removal of chloroform at 30 min for molar ratio 1:20 is 89% however for rest molar ratio 1:10, 1:15 it was 73.2 and 79.9%, respectively.

Figure 7 shows that the removal rate reached 89% due to a scenario of synchronization of the best molar ratio (1/20) with the optimal amount of adsorbent activated carbon (5 kg) and the largest area and contact time for complete the adsorption process for chloroform and thus obtain the highest performance efficiency of the system and this matches the proposed design of the treatment system.

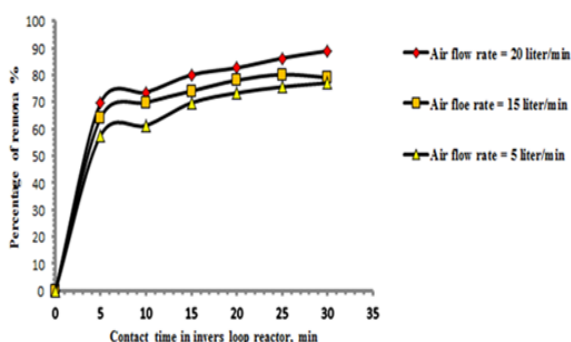
Figures 8 and 9 illustrate the effect of treatment time on removal efficiency when the airflow rate changes for three flows 5, 10 and 20 L/min and for both stages, the treatment via pulse bubble and the second stage of the treatment in the inverse fluidization reactor where the



**Figure 7.** Effectiveness of retention time on the proportion of removal at various molar ratios of  $\text{CHCl}_3$  to hydrogen peroxide in the inverse loop reactor



**Figure 8.** Impact of pulsation time on the percentage of removal with different airflow rates



**Figure 9.** Effectiveness of retention time on the proportion of removal at various airflow rates in the inverse loop reactor

efficiency for the chloroform removal for two stages with total residence period 60 min were as follows: 77.1, 79.09, and 89%, respectively. No significant variation was observed for the effect of the volumetric flow rate of air on the percentage of removal where the difference did not exceed 10% in the removal percent when the range of airflow rate changed from 5 to 20 L/min for both phases, the pulse bubble column stage and the next stage of the reverse liquefaction reactor.

#### 4. CONCLUSION

The removal of chloroform by 89% with treatment duration of one hour is evidence of the following:  
 First: The success of the proposed design by combining the pulsating bubble column and the reverse fluidization reactor as a treatment system for water contaminated with hazard and toxic organic materials.  
 Second: The success of using the solenoid valve to convey treated water from the bubble pulsating column to the inverse liquefaction reactor without using a pump.  
 Third: - Determining the best conditions for the removal system to work, which is the airflow rate of 20 liters/minute, the molar ratio between the pollutant ( $\text{CHCl}_3$ ) to the oxidizing agent ( $\text{H}_2\text{O}_2$ ) is 1/20, and the residence time of one hour.

The system can be considered economically feasible for several considerations such, the cheap of construction materials that used in building the system (acrylic) and the simplicity of carrying out maintenance work in addition to the minimal space for the system occupied it makes the possibility for the use of a treatment system to minimize the risk of organic pollutants in wastewater and the possibility of recycling and release into the environment after treatment of a reality fact.

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### Persian Abstract

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#### چکیده

در این مطالعه امکان استفاده از طرح توسعه یافته برای حذف آلاینده های آلی از فاضلاب مورد بررسی قرار گرفته است که شامل ادغام کار هر دو ستون حباب پالس (PBC) و راکتور حلقه هوایی معکوس سیال سازی (IFALR) می باشد. تریبون آزمایشی ساخته شده و نصب شده است که از یک ستون حباب به قطر ۵ سانتی متر و ارتفاع ۲۱۰ سانتی متر تشکیل شده است. در قسمت بالا یک شیر الکتریکی وجود دارد که حداقل از طریق دو تایمر به صورت الکتریکی تبدیل می شود و اتصال آن با حلقه راکتور توسط سوپاپ یک طرفه راکتور حلقه شامل یک لوله مستطیل شکل بیرونی با ابعاد (۲۹ سانتی متر طول  $\times$  ۱۵.۵ سانتی متر عرض  $\times$  ۱۵۰ سانتی متر ارتفاع) و یک لوله پیش ساز داخلی با قطر ۹ سانتی متر و طول ۱۲۰ سانتی متر به عنوان کربن فعال شده دانه ای به عنوان جاذب در ناحیه آنولوس قرار می گیرد. بین لوله داخلی و خارجی. آزمایشات با استفاده از یکی از آلاینده های آلی یعنی کلروفرم انجام شد، با یک سناریوی کار که شامل تغییر هر دو سرعت جریان هوا (۲-۲۰) لیتر در دقیقه، زمان کل بقای تیمار (۵-۶۰) دقیقه، نسبت مولی از آلاینده کلروفرم به عامل اکسید کننده پراکسید هیدروژن (۱۰/۱ - ۲۰/۱). نتایج نشان داد راندمان حذف نزدیک ۸۹٪ است و نشانگر موفقیت در طرح پیشنهادی می باشد که احتمال بازیافت آب تصفیه شده و رها کردن آن به محیط زیست به دلیل کم خطر بودن آلاینده آلی موجود در آن است.

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