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Novel laboratory extraction method using magnetically micro bubble stream

Emad Kadhim Atiyah
Chemistry Department, Faculty of Science, Kufa University, Iraq
*Corresponding author email: mustafa.rabeea@uokufa.edu.iq

Mustafa Abdulkadhim Hussein
South Refineries Company / Maysan Refinery Department

***Abstract*---**Innovative, sensitive and accurate method for liquid-liquid extraction using bubble phenomenon. is presented in this work. A working system was designed, consisting of two basic parts: the pump air to generate the bubbles and the pump device, to pump or withdraw the organic layer from the liquid-liquid system key variables study was performed in the system containg Dithizone (0.001 M) in $CHCl_3$ solvent and aqueous copper (II) chloride (1000 ppm) solution to find out the extent of their impact on the extraction performance obtained in this way. The effect of copper concentration in the initial sample, shake time, size of bubbles flowrate were studied, fixing the optimal conditions: for a sample of (10 mL) aqueous solution of Cu (II) chloride, the optimum copper (II) concentration in the analyzed sample is (0.0001M) , the size of bubbles 2.6 L\min, the shaking time is (15 min), and the pump flowrate is (50sec/mL). In these optimum conditions, the extraction degree was (91.1±2%). The results of the innovative bubbles extraction method were compared with previous studies, and found they were more efficient than the classical methods of extraction using funnel separation and multiple extraction.

***Keyword*---**novel laboratory, extraction method, magnetically micro, bubble stream.

Introduction

Water is critical for human survival as well as the survival of all living organisms. Water consumption has risen in recent years as the world's population has grown[1]. Meanwhile, residential and industrial agriculture produce significant amounts of wastewater including contaminants such as suspended solids, pathogens, nutrients, and heavy metals that can be hazardous and deadly[2].

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Many pollution concerns will be introduced if they are left untreated and safely discharged back into the sea or rivers[3]. Heavy metal is one of the contaminants found in wastewater[4]. Anthropogenic sources such as electronics assembly and production, textiles, fertilizer and pesticide use, and mining operations all contribute to this wastewater with heavy metals[5] [6]. These heavy metals can be hazardous to species, including humans, even at low concentrations [7]. Ultra-filtration, reverse osmosis, ion exchange, solvent extraction, sedimentation, and chemical precipitation are some of the traditional methods for removing heavy metal ions from wastewaters [7].

However, most of these approaches have drawbacks, such as metal removal that is insufficient [6]. It is critical that we find novel methods for the affordable, energy-efficient cleanup of trace pollutants from water in order to sustain environmental and human well-being [8][9] . Microbubbles (MBs) and Nano bubbles (NBs) are small bubbles with diameters of 10–50 lm and 200 nm, respectively, that have been studied for a variety of uses. In chemical and process engineering, two-phase activities between gas and liquid are frequently carried out using gas blasted into the liquid as bubbles[10][11] . The main objective of these methods is to reduce the extraction time and energy consumption, which is reflected in the final cost reduction, and this is one of the important aspects of these technologies, because they protect the environment and enhance the economic competitiveness and innovation of industries[12]. In this study, an innovative method was proposed to make the extraction easier and accurate and give high rates of extraction.

Experimental

Chemicals

All chemicals used in this study were supplied by companies (Fluka & BDH).Dithizone solution ($C_{13}H1_2N_4S$) (Fluka) was prepared at a concentration of (0.01M) by dissolving (0.0256 g) in (10 mL) of $CHCl_3$ (BDH). For the requirements of the study, another group of Dithizone solutions with a concentration of (0.001M) was prepared by taking (5mL) of the previous Dithizone solution of (0.01M) and diluting it in (50mL) of $CHCl_3$ in a standard volume vial, and keeping the solution in an opaque bottle away from the light[13][14]. An aqueous solution of copper (II) chloride ($CuCl_2.2H_2O$) (Fluka) was prepared at a concentration of (0.01M) by dissolving (0.42625mg) in a small volume of distilled water, then completing the volume to (250 mL) of distilled water, in a standard volumetric vial and saving the solution for use. For the requirements of the study, another group of solutions of $CuCl_2$ with a concentration (0.0001M) was prepared by taking (0.5 mL) of the previous copper (II) chloride solution of (0.01M) and diluting it in (50mL) of distilled water in a standard volumetric vial, when the concentration is Copper (II) standard (0.635 ppm). The acidity of the $CuCl_2$ solution is adjusted to pH=1, the degree of the pH is determined through the pH meter by adding a small amount of HCl (BDH).

Equipment

Pump air model RS Electric to generate bubbles; Peristaltic pump Ismatec to pump the reagent and withdraw the organic layer; pH - meter WTW - SERLES

Germany, to determine the required pH and atomic absorption device SHIMADZU - 6300, used to measure the proportion of copper (II) remaining in the organic layer.

Bubbles extraction working procedure[15]

This method involves pumping a Dithizone solution at a concentration (0.001 M) by a peristaltic pump, through a rubber tube, after stabilizing the pump velocity at (3mL/30 sec) a certain volume to a certain volume of $CuCl_2$ after fixing the optimum conditions to complete the extraction process which are: a temperature of (25-27 °C) , pH = 1, and the speed of air pump 1.75L\min which works to generate the bubbles by air and with a fixed vibration time at 15 minutes.

Result and Discussion

Bubbles extraction system design

A new working system has been designed using the bubbles technique, it is a bubble generation device by an air pump that operates at two speeds to increase bubbles. This system enables to repeat of the extraction process many times in a short time, and easily monitor the stages of the extraction process while working on it. The design of the work of the system consists of the following main components Figure 1:

- Bubble generation device, which is a two-speed pneumatic pump to pump air through a bubble-generating part and is the main and basic part of the system design.
- The peristaltic pump device, for the transfer of the reagent material to the analyzed sample back and forth.
- A thermometer, which is used to measure the temperature stabilized at (25-27 °C).
- Round flask contains a cover with two holes, the first for pumping the organic matter and the second for pumping the air that generates bubbles by means of the bubble generation device.
- A transparent McCartney bottle volume (50 mL), diameter (29 mm), and height (92 mm) were fixed during the study stages.

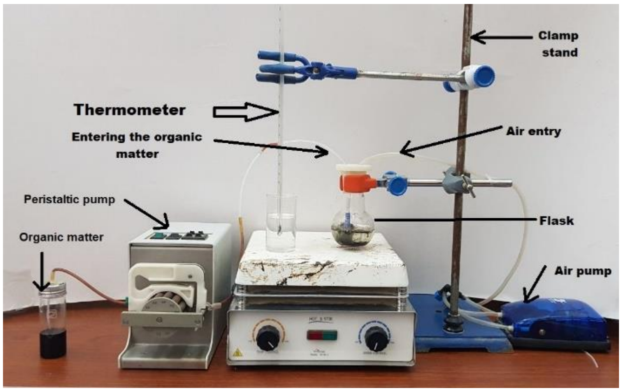

Figure 1. Bubble extraction system design

Stages of the bubbles extraction process

Bubbles generation device, which is a two-speed pneumatic pump to pump air through a bubble-generating part and is the main and basic part of the system design extraction is the process of pumping a specific volume of organic layer Dithizone solution to a specific volume of aqueous $CuCl_2$ solution in a glass container containing the bubbles generation device, which is a two-speed pneumatic pump to pump air through a bubble-generating part and is the main and basic part of the system design component and then the extraction process (complexity) beginning gradually between the two solutions, from green to form a complex Dithizone - dark red copper indicating completion of the extraction process. One of the most important factors that increase the rate of extraction is the increase of the surface area between the organic and aqueous layers, which is evidenced by two principles in this new innovative method. First, the movement of the same bubbles (spherical) results from the pump air device that generates thousands of balls (bubble shape) filled with the organic phase inside the aqueous phase. Secondly, after the pump and the air pump stop, where the surface exchanged between the aqueous and organic layer increases, and then the organic layer is suctioned in a reverse way using the pump, after which the Copper percentage is measured. The remainder is measured in the aqueous layer using a flame atomic absorption device[16], [17]. shows from left to right the stages of the extraction process through the effect of the phenomenon of the bubble Figure 2.

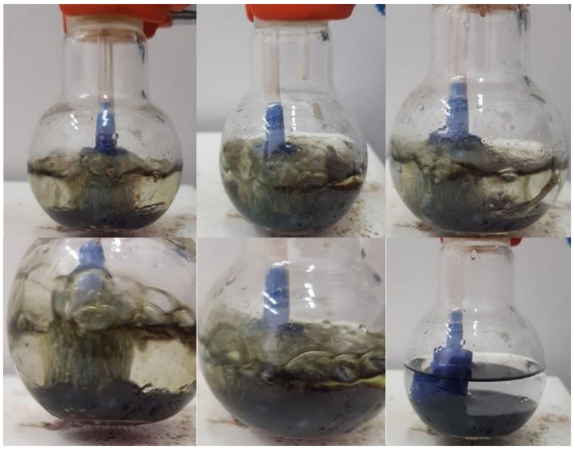

Figure 2. Stages of the bubbles extraction process

The effect of the main variables study

In this work, four main variables were studied to find out the extent of their effect on the percentage of extraction that occurs using the bubbles, as follows:

The effect of the concentration of $CuCl_2$ on the extraction process

The thermodynamic and kinetic balance[16][18] for the extraction processes shows that the concentration of the metal ion has a direct impact on the process of forming the extracted complex[19], which is important in getting the best values of the distribution constant D and the extraction degree %E. Therefore, the process of extracting Cu(II) was carried out by taking two volumes of $CuCl_2$ (10ml) each, the concentration of the first being (0.001M) and the concentration of the second (0.0001M), at pH = 1 and after pumping (10 mL) of Dithizone solution at a concentration of (0.001M) each at a pumping speed of (3mL/30sec) , with a time of shaking (10 min), the organic layer being withdrawn from the aqueous layer by the pump. The percentage of copper remaining in the aqueous layer is measured in both samples. After calculating the values of D and %E, it was found that the second concentration (0.0001M) is better for the bubbles extraction process, because it gives higher values than the first concentration (0.001M), as shown in the Table 1.

Table 1
The optimum copper (II) concentration for the bubbles extraction process

sample NO.	Copper(II) concentration	D	E%
1	0.001M	3.8	79
2	0.0001M	13.5	93.1

Study the effect of change the shake time

In this study, four times equal volumes (10 mL) of $CuCl_2$ solution and dithizone solution with varying vibration times for each time (10 min), (15 min), and (20 min) were pumped from the bubbles extraction process, with constant speed The pump at(30sec/mL), air pump size(1.75 L/min). (Figure 1) showed that the values of % E increases with the increase in the vibration time respectively which gives a longer time for the movement of the bubbles to complete the extraction. We consider (15 min) as the most appropriate time to extract because the extraction degree is satisfactory and a long time means more energy consumption. Figure 1.

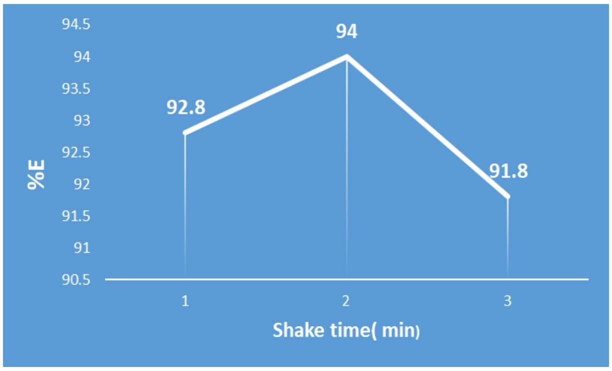

Figure 1. The effect of change the shake time

The effect of changing the size of bubbles on the extraction process

In this study, a set of solutions were prepared with a fixed volume (10ml) of copper(II) chloride solution and dithizone solution, and the fixed volume of dithizone solution was pumped to the same volume of copper(II) chloride solution of (10ml) for each stage of the extraction process. The variable factor that was used in this study is changing the volume of air forming bubbles in two volumes according to the air pump, which pushes the air at two speeds according to the pump installation, which increases the surface area of the bubbles when it is established:

- pump speed
- shake time
- volume of solutions.

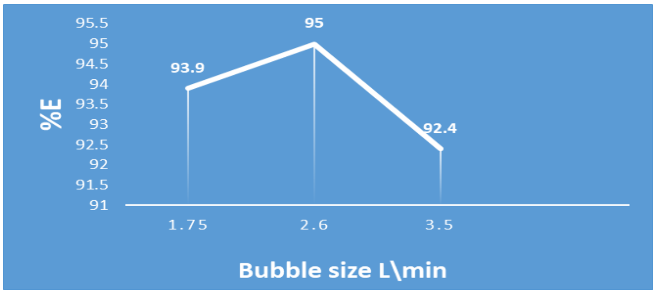

Figure 2. The effect of change the size of the bubbles

The results of this study, as shown in Figure 2, showed that the values of the extraction ratio (E%) and the value of (D) increase with the increase in the size of bubbles, respectively. It increases in the first and second stages with the increase in the size of the bubbles (the increase in the volume of air that generates bubbles) meaning that the air pump speeds (17.5L\min and 2.6 L\min) are appropriate in generating bubbles consistent with the volume of the two solutions, It decreases in the third stage with the increase in the size of the bubbles, meaning that the size of (3.5 L\min) gives turbulent bubbles due to an increase in bubbles and is unstable with the volume of the solutions, which affects the extraction process.

Effect of changing in the rate of pump speed

The change in peristaltic pump velocity to four speeds (30 sec\ml), (50 sec\ml), and (70 sec\ml) was studied. (Fig. 3) shows that the value of the degree of extraction %E rises slightly as the pump speed increases between (30sec\ml and 50s\ml), after that the values of %E decreases as the rate of pump speed increases; The reason is due to the low flow rate of the pump which allows the movement of bubbles to take up a large area for the extraction process from the organic layer; On the contrary, the high speed of the pump does not allow to complete the bubble extraction process to successively increase the organic layer of the peristaltic pump. For further study, a flow rate of 3 50sec/mL sec was chosen.

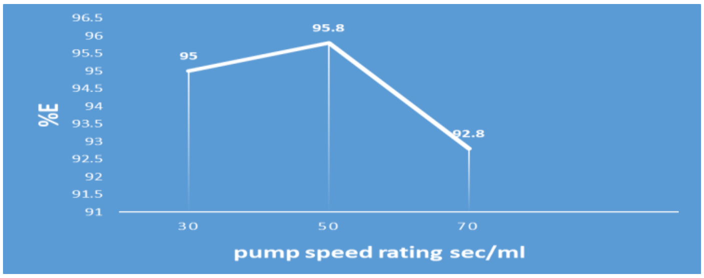

Figure 3. Effect of change the rate of pump speed

Study the effect of equal volumes

In this study, four volumes of dithizone solution were injected at an amount of (10,15,20, and 25mL) into a solution of copper chloride (II) specified in the same volume respectively for each stage of the extraction process during the study. When the: pump velocity is at (50sec/mL), the size of the bubbles is at(2.6L\min), and the shaking time is at (15 min). shows the results of the study as follows: The results of the study show that the volume (10mL), which is constant between the two solutions, gives a higher result for the values of E% and the distribution coefficient D, because the total volume between the two solutions (20mL), which is appropriate for the size of the bubbles, helped to generate stable and stable bubbles within the remaining space of the glass bottle, which gives better scope for the extraction process. As for the results of the other fixed volumes, they show low values for D and E%, they are close due to the small space remaining from the bottle that in turn, leads to disruption of the generated bubbles, which affects the extraction process as shown in the figure 4.

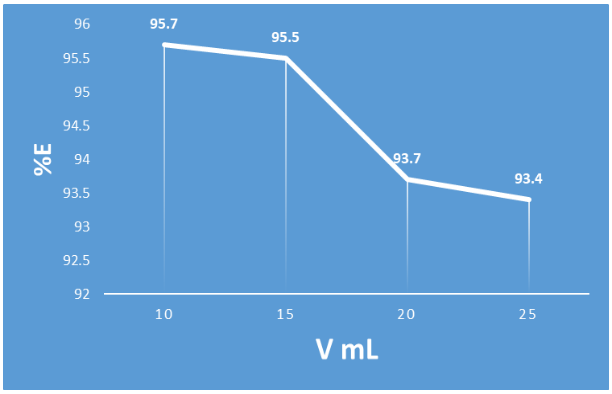

Figure 4. Study the effect of equal volumes

Studying the effect of different volumes

In this study, four different volumes of dithizone solution were injected at an amount of(10, 15, 20mL) sequentially to a constant volume of copper (II) chloride solution in the amount of (10mL)For each stage of the extraction process, when: the pump speed at(50sec\mL), bubbles size (2.6 L\min)and the time of shaking at(15min) Table 6. shows the results of the study.

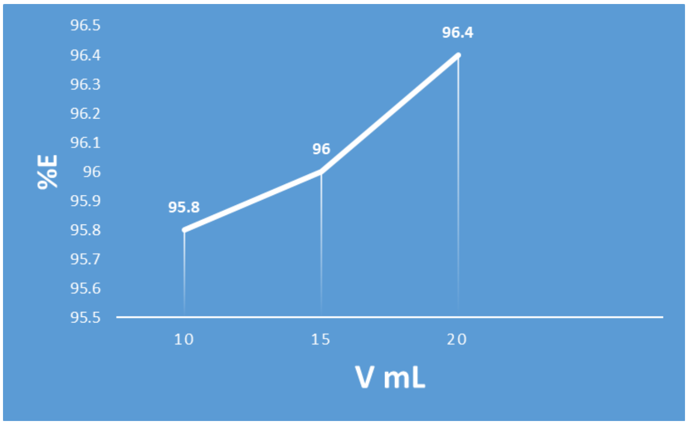

Figure 5. Studying the effect of different volumes

Accuracy of the bubbles extraction method

To evaluate the innovative extraction method, the bubble phenomenon was compared to the traditional method described in [20] which uses suppressive separation in the dithizone extraction process, and an analytical sample was prepared from a copper (II) solution at a concentration (1000 ppm), and analyzed by both methods. Using the optimal conditions that were studied in bubbles extraction: pump speed (50sec/ mL), bubbles size (2.6 L/min), vibration time (15min), temperature (25-27 °C), and Dithizone concentration (0.2 M), the extraction degree was (91.1±0.2) which was higher than that found by us with the traditional method. The results are shown in Table 7.

Table 7
Comparison between the results of the vortex extraction and the traditional method

Extraction method	Cu(II) concentration (ppm)	Dithizone concentration (M)	shake time (min)	D	E%
bubbles	1000	0.2	15	6.1	91.1
Traditional[20]	1000	0.2	30	1.73	78.5

Conclusions

The conclusion as resulting from (Table 2) indicates that the innovative bubbles extraction method can be used for its ease and speed of work, reduced use of materials and solvents, and low pollution that introduces them into the uses of green chemistry. The bubbles is extracted by a single process unlike multiple extraction, which needs longer time in addition to physical effort by using a separating funnel .

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