academicJournals

Vol. 8(41), pp. 1954-1965, 9 November, 2013
DOI: 10.5897/IJPS2013.3809
ISSN 1992 - 1950 © 2013 Academic Journals
http://www.academicjournals.org/IJPS

International Journal of Physical Sciences

Full Length Research Paper

Theoretical and experimental studies for wool wax recovery by flotation

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Accepted 5 November, 2013

Theoretical and experimental studies have been performed to analyze the flotation processes for recovery of wool wax from an industrial effluent by air flotation. Batch flotation process was carried out at different experimental conditions and different air flow rates. The results obtained show that wool wax can be successfully recovered from the tested effluent using batch air flotation. The percentage of wax recovery increased with increasing the flotation time, that 90% wax recovery was achieved within 10 min. These high recoveries reduce extraction costs. Wax recovery was found to decrease with increasing the initial concentration of wax/water emulsion. The highest percentage recovery of wax was obtained at a column working height approximately eight times the diameter of the flotation column at an emulsion of pH value of 9.0, inlet flow rate 1560 ml/min at a wool-soaping concentration 0.5 g/L. An appropriate kinetic model was identified to represent the behavior of wool wax recovery from an industrial effluent by air flotation.

Key words: Wool wax, recovery, air flotation, industrial effluents.

INTRODUCTION

During recent years there is an increasing public awareness concern regarding environmental pollution. Pollution is the contamination of environment as a result of human activities. The term pollution refers preliminary to the fouling of air, water and land by wastes. One of the major sources which can affect the quality of seas, rivers and underground water is oil and greases. Low concentration of oil or grease in seas and rivers impart an unpleasant taste to fish. Heavy surface grease films interfere with the process of natural aeration and photosynthesis. Free wax or oil and emulsions in water sources may coat their surface and destroy algae and other planktons thereby removing a source of fish food (Bateup et al., 1996; Christoe, 1986).

Millions head of sheep, the total sacrifice and sacrifice for the pilgrimage season every year in Kingdom of Saudi Arabia. A recovery process for removal of valuable wool wax as a product having the specification of lanolin from wool grease was found in the liquor from the scouring of raw wool. The process, apart from removing wool grease/wool wax from wool scouring effluent (Christoe et al., 1976; Kolattukudy, 1976; Moldovan et al., 2002), avoids the production of pollutants. And in the case of the treatment of wet sludge containing wool grease produces a virtually grease- free solid product suitable for disposal as an environmentally safe soil.

The cost of treatment of wax water emulsion by known processes such as those processes which rely on acid or

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base treatment or solvent extraction is quite high and in general have not proved economically attractive (Dominguez et al., 2003). However, the discarding of valuable and useable materials often found in the emulsion is unfortunate waste and in most cases the disposal of the emulsion to sewage systems and rivers is environmentally unacceptable without treatment of some sort (Anna et al., 2012). It may be practically useful to make some research efforts towards improvement of the performance in wax removal by simple operations and economic procedures (Max and Klaus, 1980).

Flotation processes have been proved (Rubio et al., 2002; Bradeley, 1985) suitable to remove both suspended solids and waxes at a time from a great variety of liquid water such as the effluents of pulp industry, the textile and dying industry, the food industry, the tannery process, the petrochemical industries (Tsubouchi et al., 1985), waste water, oil production, and refining electroplating and battery industries as well as, municipal waste water (Rippon, 1992). Flotation process is applicable to a wide range of oil water emulsions, which were found difficult to be processed by the known methods. Representative of these emulsion are the wool grease in water emulsions resulting from wool scouring processes. Flotation constitutes, thus an alternative process, which cover the advantages of concentration and separation method in one operation (Genon et al., 1984; Mercz and Cord-Ruwisch, 1997; Eychene et al., 2001).

Flotation processes are classified with respect to the method by which the air bubbles are generated as dissolved air flotation, induced air flotation, and electro flotation (Van Ham et al., 1983; Nicholas and Byeseda, 1980; Rajinder and Masliyan, 1983). The aim of the present work is to recover wool wax from industrial wool scouring effluents by air flotation using a batch flotation process, which is carried out at different conditions as preliminary steps for further continuous operation works. To suggest appropriate kinetic model to represent the behavior of wool wax recovery from an industrial effluent by air flotation.

EXPERIMENTAL

In order to obtain oil/water (W_x/W_a) emulsion, wool is first cleaned and then scoured (Gibson et al., 1981; McCracken and Chaikin, 1978). The obtained emulsion is treated by a flotation process at different conditions, of initial concentration, airflow rate, and the amount of soap used during the scouring process. Besides, both batch and continuous flotation are done at different feeding points. Within the scope of the present investigation discussion will emphasis on the batch flotation process only.

Materials

a) Wool: Raw wool, a protein (keratin), contains glandular secretions (suint and wool grease) and feces from the sheep, plus dirt, straw, and vegetable matter. Residues of treatments applied for disease control or for identification of the animal may also be present. Being amphoteric, wool is damaged by caustic or acid solutions, so special care is taken in processing.
b) Synthetic detergent: It reduces surface tension, increases the wetting capacity of the solution, converts the impurities of the fiber into emulsion and stabilizes the colloidal system formed (Athukorala and Mazza, 2011). In this work Nestabone, a local dispersing agent was used.
c) Soda soap and diethanolamine soaps: are used as foaming agent to create froth while diethanolamine is a soaping agent added for wax emulsification.
d) Soda ash,Na_2CO_3 (1%): Partially softens the water by interacting with calcium salts, creating an active reaction of the media which is most favorable for the formation of stable emulsion, and increase fiber swelling thus assisting the release of impurities from the fiber.
e) Caustic soda and nitric acid (Analar 99%), are used to adjust pH value

Emulsion stability test

Five W_x/W_a stocks solutions were prepared using 0.5 g of wool wax per liter of water. For all samples, mixing was continued for 60 min at a rate of 800 rpm, concentrations of emulsifier used were 200, 300, 400, 500 and 600 ppm. The samples were left to stand for 24 h each in a separate bottle, samples were taken from the surface and the bottom of each bottle and the turbidity was measured for each sample. The optimum amount of emulsifier required to attain 24 h stability was found to be 500 ppm.

Emulsion preparation

Ten wax/water emulsion concentrations were prepared by the following procedure (0.5 g of wool wax are added to 0.5 g of emulsifier in one liter of water and the resultant mixture is stirred for one hour at 800 rpm, a 500 ppm wax/water emulsion is obtained). A stock solution (250 ml) is diluted to one liter to obtain 125 ppm of oil in wax water emulsion. The turbidity is measured and recorded and the steps are repeated to prepare and measure 10 different concentrations of wax/water emulsion.

WAX WATER EMULSION PREPARATION

The procedure includes three main processes cleaning, scouring of wool and the flotation process. In the cleaning process 10 g of grey wool are boiled with 1 g synthetic detergent in 200 ml tap water for 7 min and then wool is removed from liquor. To that boiled wool sample 0.5 g soda soap powder are added in addition to 3 ml diethanolamine, 1 g synthetic detergent, 1 ml Na_2CO_3 solution (1%), and 200 ml tap water and the mixture is boiled for one hour. The resultant wool is washed, squeezed and the resultant squeezing off solution is added to the original mixture. The resulting emulsion is treated by the flotation process (Poole et al., 1999, Poole, 2004; Poole and Cord-Ruwish, 2004).

Flotation column and procedure

A column, 1 m height, 0.05 m diameter (Figure 1) with an opening for air inlet in the bottom was used, fitted with a sparger of 1 mm diameter for distributing of air into small bubbles. Along the column height there is a sampling opening and near the top of the column there is an outlet the foam. Accessory elements for the column are; pH meter, compressor, air flow meter and a pump. Results were

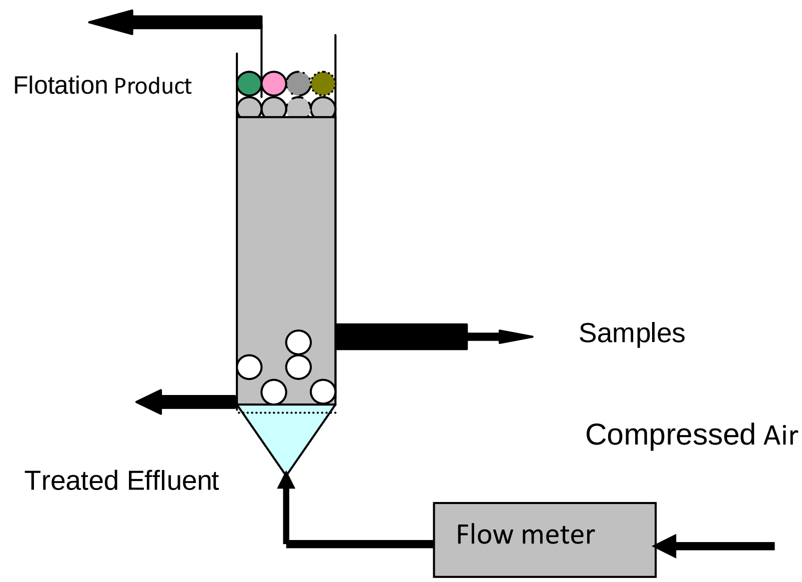

Figure 1. Sketch of flotation column.

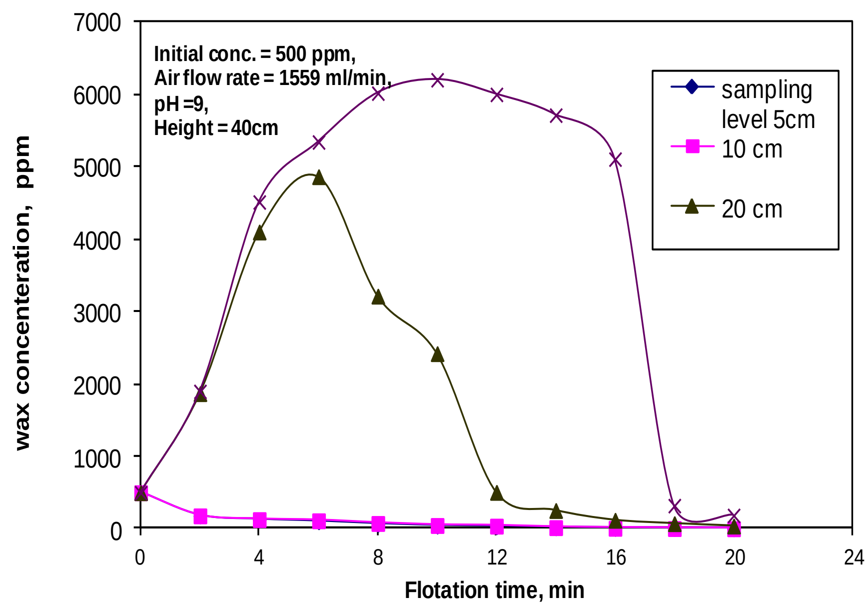

Figure 2. Effect of sampling level on wax concentration.

judged by a turbidity meter, measuring eFTU for comparison. The flotation column is washed thoroughly and is connected with all accessories required to begin an experiment. Turbidity and pH of the emulsion are measured before the flotation process. Different flotation experiments were carried out at different operating conditions such as initial concentrations of the W_x/W_a emulsion, working heights, inlet airflow rate, and different pH values. In all the experimental runs, samples are taken at 2 min intervals from different sampling points to measure the wax concentration.

RESULTS AND DISCUSSION

Effect of sampling point location

The effect of sampling point positions is clearly shown in Figure 2, which shows the relation between the wax concentration and time at different sampling points. It is clear that lowest wax concentration is obtained at the

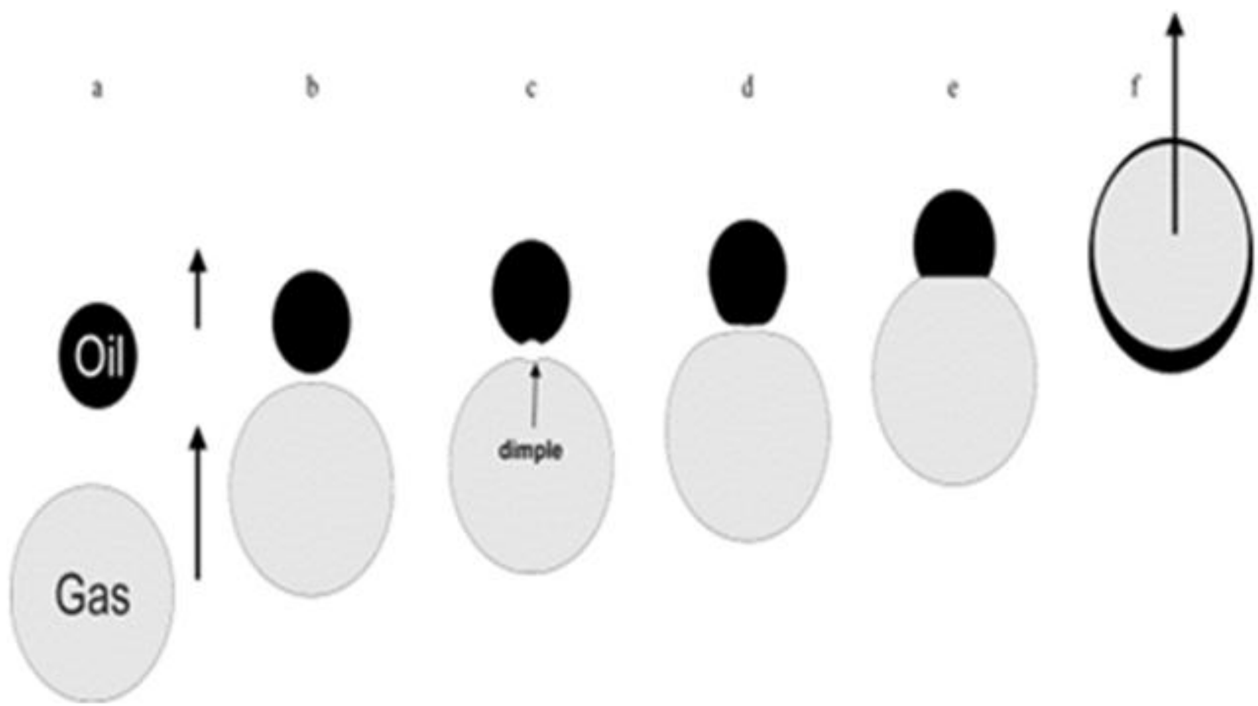

Figure 3. The attachment process. (a) Bubble and drop approach, (b) water film thinning, (c) thin film dimples due to interfacial tension gradients, (d) the dimples disappears as the film drains, (e) at a critical thickness film ruptures and if spreading conditions are present, the oil spread around the gas, (f) the conglomerate then continues to rise. If this processes have not occurred within the timeframe of approach, the bubble and drop do not attach but move away from each other.

lower sampling point and this is explained that gas flotation process may be an accelerated gravitational separation technique in which fine gas bubbles are injected into a water phase containing immiscible liquid droplets (wax) so that the gas bubbles (Figure 3) attach themselves to the wax droplets (Stewart, 1988a). The wax appears lighter because the density difference between the wax-air agglomerates and water is increased, consequently, the wax rises faster enabling a more rapid and effective separation from the aqueous phase. Air bubbles enter the flotation column near the lower sampling point where the bubbles are fresh and did not yet carry any wax so their ability to carry wax droplets is the highest along all the flotation column and so the wax concentration in the bottom of the column is the lowest one.

On the other hand at a height of 20 cm of the column top the wax concentration is increased with time until 6 min then with further increase in time the wax concentration is decreased. This is attributed to that after nearly 6 min all the wax droplets present in the column in the height below 20 cm are carried by air bubbles from the bottom of the column to the top. So the concentration of wax in the top is increased and with further increase in time there is no more wax droplets to be carried by the bubbles from the bottom. The air bubbles start to carry the oil droplets from height above 20 cm to the top of the column. Thus the wax concentration starts to reduce. The same holds with respect to the sampling point at height 40 cm at the start of the flotation process the air bubbles carried the oil droplets from the bottom of the column to the top of it till nearly most of the wax present in the bottom of the column is removed. The air bubbles start to carry the wax droplets from the top itself and so the wax concentration at the top of the column is increased at the start of the flotation process till time 16 min then with increasing the time the wax concentration is decreased.

Effect of different amount of soap

Figure 4 shows the effect of the different amount of soap used in the scouring process on the percent of wax removal during the flotation process. It is clear that as increasing the amount of soap the percent removal of the wax from the wax/water emulsion is increased till the maximum value of 99.6% at 0.5 g and time of flotation of 20 min. This increase of the percent removal of the wax can be explained as where soap is acting as soaping agent. The produced foam is increased with increasing the amount of soap and hence the efficiency of flotation process is increased and so the percent of wax removed is increased.

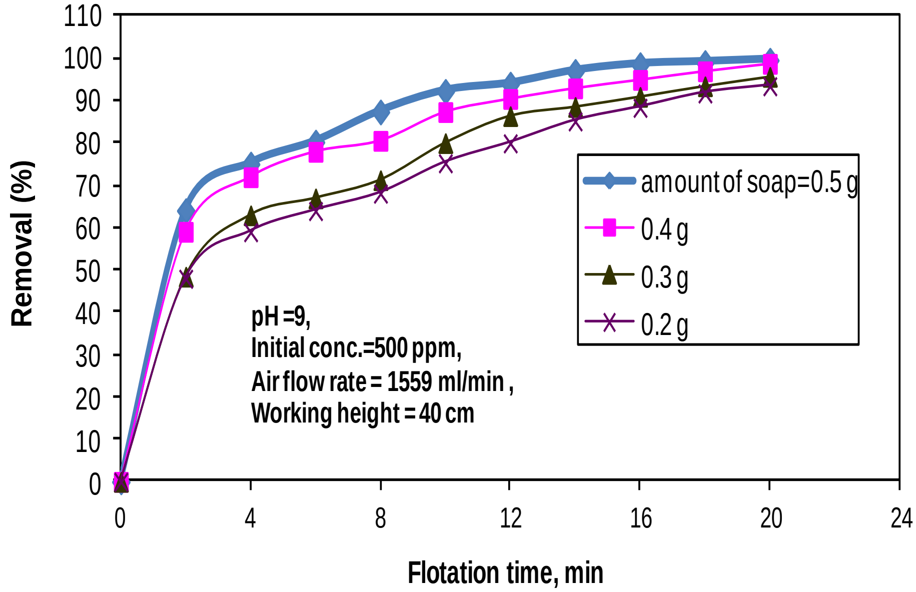

Figure 4. Effect of amount of soap in the scouring process on % removal of wax during flotation.

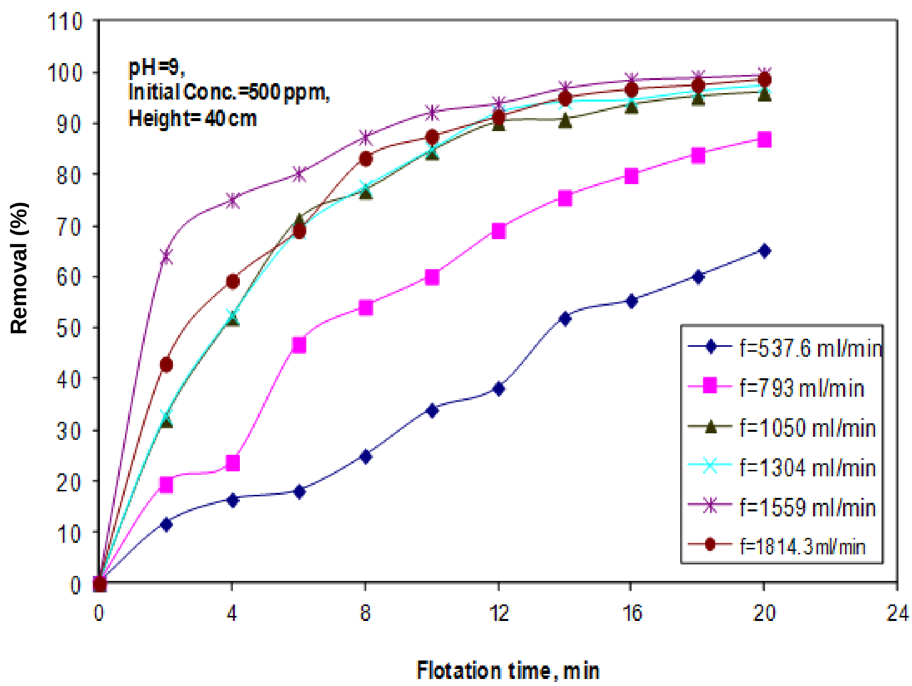

Figure 5. Effect of air flow rate on % removal.

Effect of air flow rate and flotation time

Figure 5 shows the effect of flotation time on the percent of wax removed during the flotation process while Figure 6 shows the effect of air flow rates. Both figures show that with increasing the inlet air flow rate from 538 to 1814 ml/min the percent removal of wax is first increased by increasing the air flow rate till inlet air flow rate

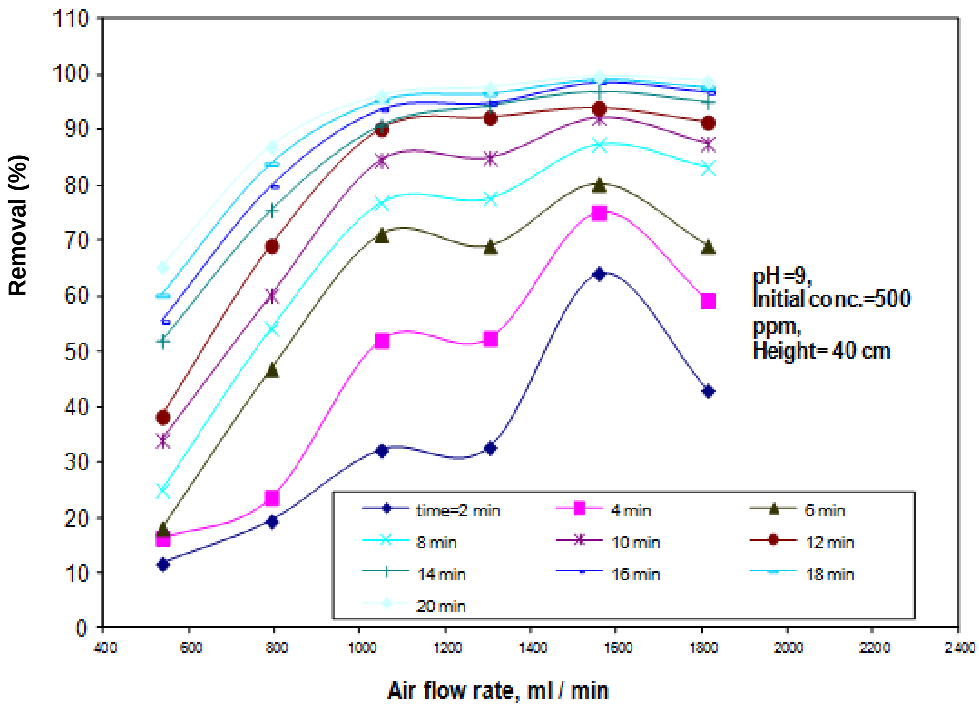

Figure 6. Effect of air flow rate on removal percent.

reaches a value of 1560 ml/min and then it decreases with no any further increase in the inlet flow rate this can be explained as where the efficiency of the flotation process is mainly dependant on the number of air bubbles which carry the oil droplets out of the flotation column. So, with increasing the air flow rate from 538 to 1560 ml/min the number of air bubbles per unit volume is increased and so its ability to carry wax droplets from the W_x/W_a emulsion is increased and thus, the percent of wax removed is increased. On the other hand if the inlet air flow rate is increased more than 1560 ml/min the percent removal of oil is decreased this is not only due to air bubbles slugging, but also due to the formation of eddies which hinder the smooth bubble/droplets rising which was clearly observed visually during the experimental work.

Figure 7 shows the effect of different inlet air flow rates on the wax concentration in the O/W emulsion in the column by using the highest sampling point (at height = 40 cm) and only for 12 min. It was clearly that the highest wax concentration in the column was at the highest sampling point as discussed above. Also it is clear that the wax concentration increased by increasing the air flow rate from 538 to 1560 ml/min and this increase nearly constant after time 10 min for each inlet air flow rate. As it was discussed before that in the flotation process gas bubbles and wax drops have to contact and then attach, but since wax and gas are both less dense than water, they will both rise if present in water. The longer the residence time of the gas bubbles in the flotation tanks, the greater the number of gas bubble-wax droplet collisions (contact efficiency), the greater also the quantity of the wax that ought to be removed (Oh et al., 2009). If the air flow rate is increased up to the optimum value this will give the best number of gas bubbles at the optimum residence time (Jones et al., 1995, Jones and Westmoreland, 1999).

Effect of initial concentration of the wax / water emulsion

Figure 8 shows the relation between the removal percent of wax and time at different initial concentrations of wax/water emulsion. The figure clearly shows that with increasing the initial concentration of wax in the wax water emulsion the removal percent of wax is high. The decrease for 500 ppm initial concentration than that for 250 ppm is somewhat undetectable. This means that the initial concentration of wax/water emulsion do not affect the process up to 500 ppm.

When the concentration of wax in W_x/W_a emulsion is 250 ppm and the air flow rate is 1560 ml/min the produced bubbles has the ability to remove most of the wax droplets present in the wax/water emulsion and when the oil concentration is increased from 250 to 500 ppm and the inlet air flow rate is still 1560 ml/min appear to the produced air bubbles have the ability to remove the

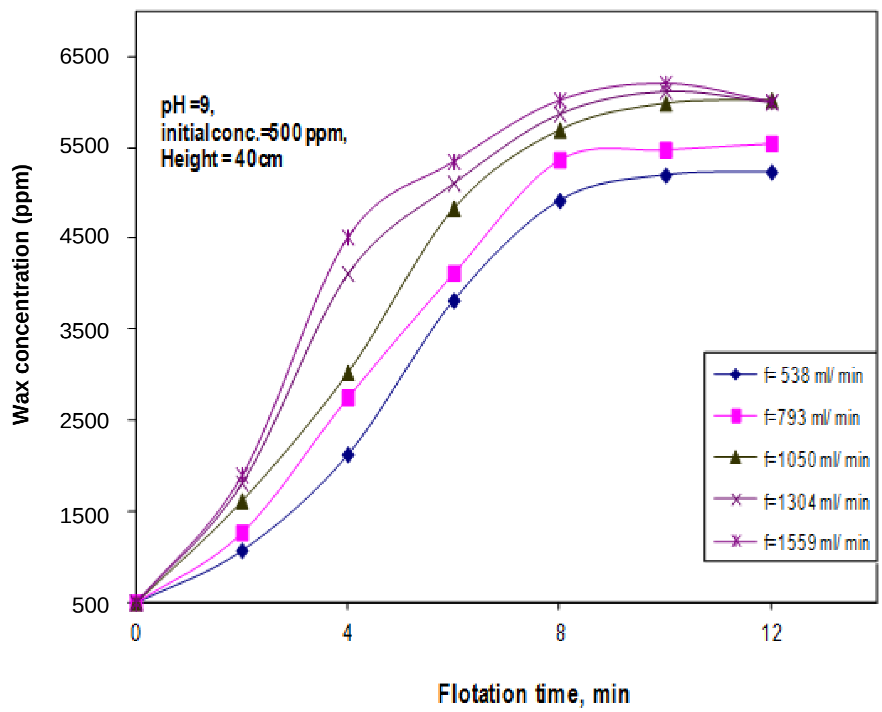

Figure 7. Effect of air flow rate at the heighest sampling point on wax concentration.

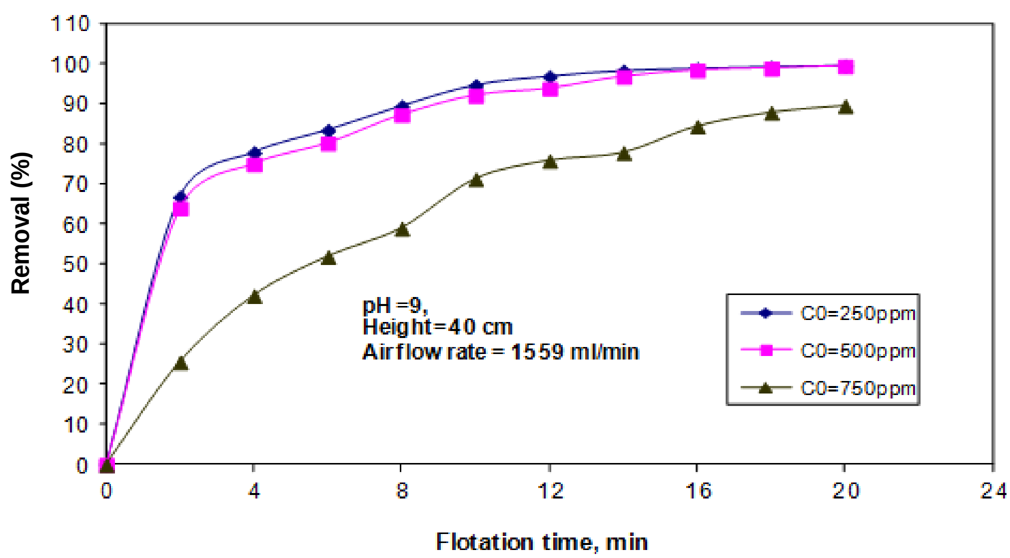

Figure 8. Effect of initial concentration of wax on % removal.

increased amount of wax droplets that result from increasing the wax percent in the wax/W emulsion but when the wax concentration in the W_x/W_a emulsion is increased from 500 to 750 ppm at the same inlet air flow rate of 1560 ml/min, it is noticed that the percent removal of wax is dramatically decreased and this can be attributed to that the number of produced bubbles are not enough to carry all the wax droplets.

If the wax concentration in W_x/W_a emulsion is increased to such a limit that the diameter of wax droplet is more than 200 µm, a primary separator must precede the flotation unit. This is because the gas bubbles cannot

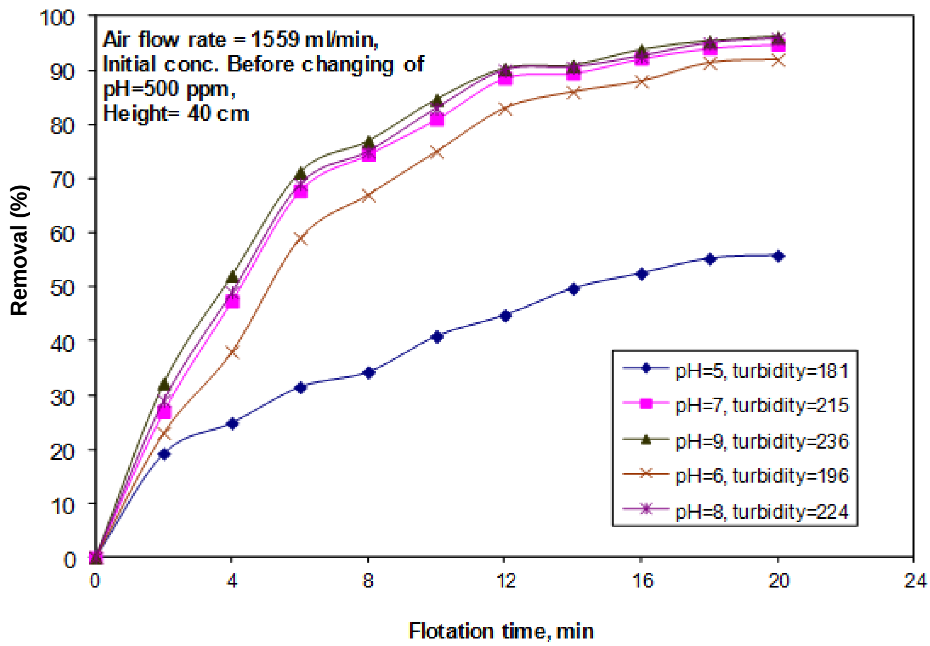

Figure 9. Effect of pH on % removal.

carry such wax droplets so that gas flotation cannot work effectively. Arnold and Stewart (1998) found that when the oil concentration exceeds 1000 mg/L with droplets larger than 200 µm in diameter, a primary separator must precede any flotation unit. This is because the gas bubbles cannot float such large oil droplets, so that gas flotation work effectively. Xuq and Chiang (1999) implied that the oil particle flotation differs from solid particle flotation in that oil–particle attachment may play an important role as bubble–particle attachment at high concentration.

Effect of pH values of the emulsion

Figure 9 shows the relation between the percent removal of wax at different values of pH of the wax/water emulsion at the fixed conditions of airflow rate, initial concentration and height of the batch charge. It is clear that the removal percent of wax from the emulsion increased with increasing pH, taking into consideration that the percent removal of wax is calculated with respect to the equivalent turbidity of the oil/water emulsion at the specified pH.

If the used process for wool scouring is the saponification and emulsification of fatty acid, the process occurs by the use of sodium, triethanolamine soaps by addition of an alkali and a synthetic detergent. The scouring solution penetrates inside the fabric structure allowing for penetration of the impurities at the same time. The alkali contained in the wetting liquor neutralizes free fatty acids on the fabric and forms soaps. Most fatty substances contaminated with the fabric are thus emulsified and eliminated as emulsion (Toro et al., 2012).

The addition of acid decreasing pH value of the wax water emulsion from 9 to 5 separates fatty acids from their soap. This process is commonly used in treating the wax/water emulsion produced from the scouring process (acid cracking) and this is clearly shown from the data where the turbidity of the W_x/W_a emulsion is decreased with decreasing the pH value of the wax water emulsion. Further removal of the wax from the resultant emulsion decrease the efficiency of the flotation processes with decreasing the pH value and this mainly due to the acid cracking. The soaps are dissociated and so the foaming agent which increases the efficiency of the flotation process is decreased. This was noticed visually during the experimental work, which is in agreement with the data in the literature which recommended a flotation after acid cracking to increase the pH value again (Stewart, 1988b).

Effect of working heights of emulsion

Figure 10 shows the relation between the percent removal of wax and time at different working heights of emulsion in the flotation column, while Figure 11 shows the relation between the percent removal of wax and height of wax water emulsion in the flotation column at different flotation times at the fixed conditions of initial concentration, pH and inlet air flow rate. These figures

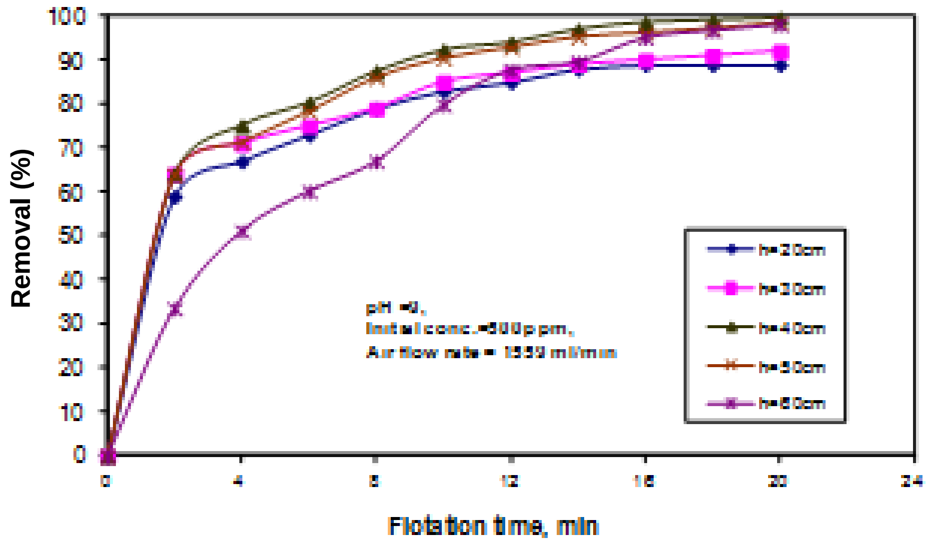

Figure 10. Effect of height of waxy water in the column on % removal.

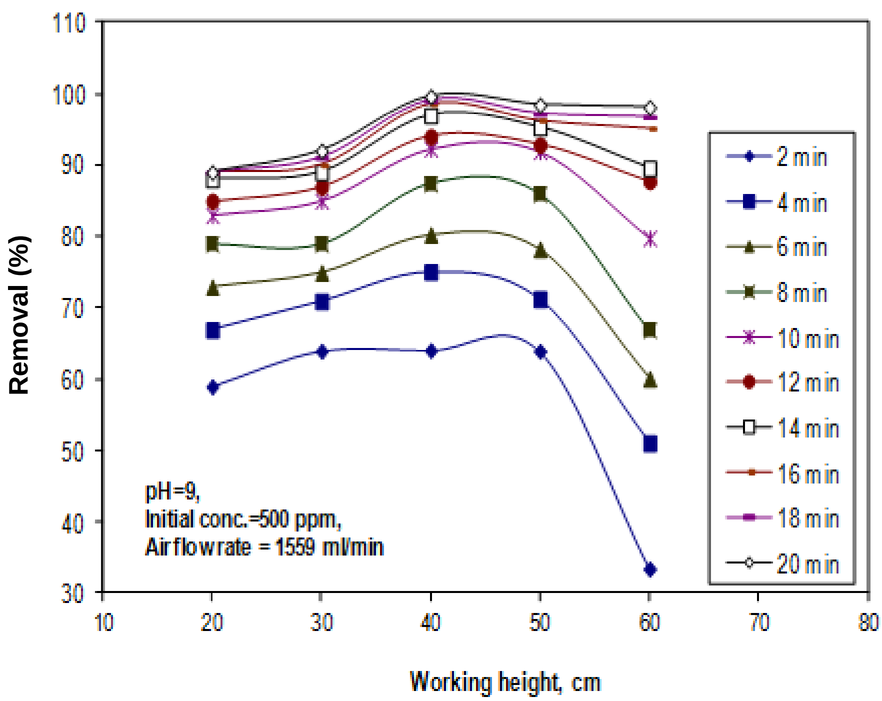

Figure 11. Effect of height of waxy water on % removal.

show that by increasing the working height of the wax water emulsion in the flotation column, the percent removal of wax is increased till 40 cm height then any further increase in the working height decreases the percent removal. By increasing the working height of wax/water emulsion in the flotation column from 20 to 40 cm the percent removal is increased because at the low working height the chance of forming eddies and turbulence is high so that wax does not attached firmly with the air bubbles (diverse flotation process) by increasing the working height from 40 to 60 cm the percent removal is decreased. It seems that the longer

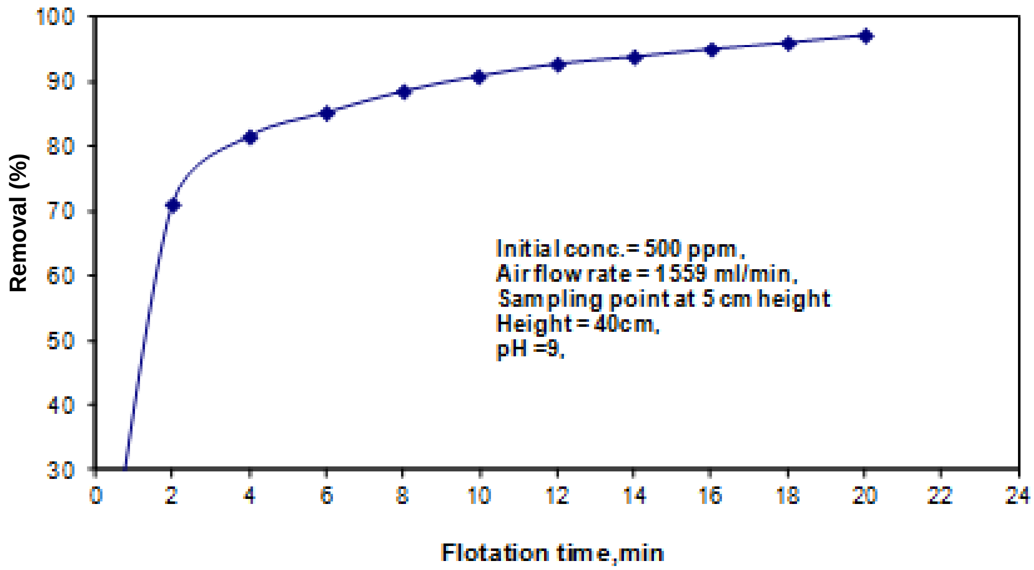

Figure 12. Effect of time of flotation on % recovery.

upward travel of wax droplets makes some separation of wax by loosening its attachment to the air bubbles.

Effect of flotation time on wax recovery

From previous experiments, it is clear that the percent of wax recovery is increased with increasing the flotation time. This increase in percent removal of wax is at high rate in the first period of the flotation process and by time this increase descents. In the first period of flotation where the number of air bubbles per unit volume of the emulsion is high so most of wax droplets are removed rapidly and the removal percent of wax is then high. By further increase the time of flotation the remaining part of wax droplets which is actually smaller than that removed in the first period is also removed but at slower rate.

Figure 12 shows the relation between flotation time and recovery percent at the optimum flotation conditions. The figure shows that the percent of wax recovery is rapidly increased in the first time of flotation, then the recovery rate is decreased. Within the first four minutes nearly about 80% of wax was recovered, while within the following 4 min only 8% recovery was achieved decreasing to only 4 min only 4% recovery is achieved. It is very clear from the economic point of view that the optimum time is 8 min, it can be increased to 10 min for ensuring total gain of wax.

Flotation model

The flotation model is a mathematical description of the separation process and can be used as a benchmarking tool and for optimizing the separation process. Given the type of particle and its size, an engineer can determine the best slurry flow rate, turbulence level and bubble size, so that the particles not only collide with the bubbles but also attach selectively during collision. The particle size determines how quickly and efficiently they collide and attach to the bubbles. Very fine particles collide with the bubbles infrequently and at a much slower rate than large particles, which collide easily but may also detach more readily because of the instability of the bubble/particle union. Among flotation models (Klimpel, 1995; Manqiu, 1998; Polata and Chander, 2000), the most acceptable model is the kinetic model which uses the first order reaction equation as the starting point. It is based on rules of mass trans-port from one phase to another.

In typical first order particle flotation, the collection rate (-dC/ dt) is considered to be proportional to particle concentration. Since the probability of collision among a group of particles is exponentially related to the number of particles, the particle-particle interaction is not important in first order kinetics. However, the interaction among the oil droplets seems to play a significant role in their collection. It can be easily imagined that this interaction increases with the increase in gas velocity and wax concentration. Therefore, if a wax flotation process is expressed in the below form, the kinetic order (n) will decrease with increasing oil concentration as can be summarized from literature data (Chopin et al., 2011; Dominguez et al., 2002; Orbán et al., 2009). The kinetics of an oil flotation process can be expressed by - dC/dt = K Cn, where dC / dt, is the collection rate, C, concentration of wax at any time, K is the rate constant and n, order of the reaction. This is in agreement with the data in the literature (Chipfunhu et al., 2010; Muganda et al., 2010).

It is based on an observation that was made in the earliest experimental kinetic studies of flotation that stated not all particles will be recovered by flotation no matter how much residence time they have in the flotation environment. Work in progress to develop a flotation model to increase the recovery and quality of wax from industrial effluents.

Conclusions

The flotation process proved to be useful for wax recovery from wool scouring effluent in batch flotation processes. The percent of wax recovery was found to increase by increasing the flotation time; the optimum flotation time was 10 min to attain 90% wax recovery. The optimum working height was found to be about eight times the diameter of flotation column. Higher percent recovery is obtained at ph value of 9.

The optimum inlet flow rate was found to be 1560 ml/min. The wax recovery percent was decreased with increasing the initial concentration of wax water emulsion; 500 ppm is the optimum initial concentration for recovery of wax. High wax recovery was obtained by higher soap concentration in the wool scouring liquor. Higher working heights or air flow rates as well as much lower column heights or air flow rates are not sufficient conditions for effective flotation. Moreover, lower pH values and lower soap amounts in the scouring process deteriorate the efficiency of wax recovery process. The kinetics of an oil flotation process can be expressed by first order particle flotation. The rate constant is useful for comparative evaluations of various operating parameters affecting flotation processes. Work in progress to develop a new flotation model to increase the recovery and quality of wax from industrial effluents

ACKNOWLEDGMENT

The authors express their sincere gratitude to Minia university, chemical engineering department for the facilities used in doing the experimental work in the their labs.

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