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RECOVERY OF WOOL WAX FROM AN INDUSTRIAL EFFLUENT BY A BATCH AIR FLOTATION TECHNIQUE

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ABSTRACT

Recovery of wool wax from an industrial effluent by air flotation was experimentally investigated. Batch flotation process was carried out at different experimental conditions and different airflow rates were also tested. From the results obtained it was concluded that wool wax can be successfully recovered from the tested industrial effluent by using batch air flotation process. The percentage of wax recovery increased with increasing the flotation time, so that 90 % wax recovery was obtained within 10 minutes. 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 working column height eight times the diameter of the flotation column at a 9.0 pH value of the emulsion, inlet flow rate 1500 ml/min, at a soap concentration 0.5g/l.

KEYWORDS

Wool wax – recovery - air flotation- industrial effluents

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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).
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 (Christoe1976, 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 some known process such as those processes which rely on acid or base treatment or solvent extraction is quite high and in general has not proved to be aluable 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. 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 (Rubio et Al, 2002, Bradeley 1985) have been proved 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, Merez et Al, 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 et. Al, 1980, Rajinder and Masliyan 1983).

The aim of the present work is to recover wool wax from industrial tanning effluents by air flotation using a batch flotation process, which is carried out at different working heights, pH values, and airflow rates as preliminary steps for further continuous operation works.

EXPERIMENTAL WORK

In order to obtain oil/water (O/W) emulsion, wool is first cleaned and then scoured (Gibson et. Al, 1981, McCracken et. Al, 1978). The obtained emulsion is treated by flotation process at different conditions, such as 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 din, straw, and vegetable matter. Residues of treatments applied for disease control or for identification of the animal may also be present. Wool is normally insoluble in water, but above 250°F (121°C) some fractions dissolve. Wool fiber expands upon wetting, but contracts to its original size when dried. Being amphoteric, wool is damaged by caustic or acid solutions, so special care are taken when subjecting it to such treatments in processing.

b) Synthetic detergent: Synthetic detergent reduces surface tension, increases the wetting capacity of the solution, converts the impurities of the fiber into emulsion and stabilizes the colloidal system formed.

c) Soda soap: Soda soap is a foaming agent added to create froth.

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) Diethanolamine / Caustic soda and nitric acid (analar 99%), are used to adjust pH value.

Emulsion Stability Test

Five O/W 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 hours each in a separate bottle, samples were taken from the surface and the bottom of each bottle and the turbidity is measured for each sample. The optimum amount of emulsifier required to attain acceptable stability was found to be 500 ppm.

Emulsion Preparation

Ten wax / water emulsion concentrations were prepared by the following procedure (0.5 gram of wool wax is added to 0.5 gram 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 10 different concentrations of wax / water emulsions.

Flotation Column and Procedure

A column, 1m height, 0.05 m diameter (Fig. 1) with an opening for air inlet in the bottom was used, fitted with asperger 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 to receive and output the foam. Accessory elements for the column are: heater, pH meter, a condenser, a digital balance, a compressor, flow meters for air and liquids and a pump. Results were judged by a turbidity meter, measuring FTU for comparison.

The procedure includes three main processes: cleaning, scouring of wool and the flotation process. In the cleaning process 10 gram of grey wool is boiled with 1gram synthetic detergent in 200 ml tap water for 7 min. and then wool is removed from liquor. To that boiled wool sample 0.5 gram soda soap powder is added in addition to 3 ml diethanolamine, 1 gram 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 where that resulting, emulsion is treated by the following batch flotation process (Poole et. Al,1999, Poole et. al, 2004, Polole and Cord-Ruwish 2004).

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 O/W emulsion, working heights, inlet airflow rate, different pH values. In all the experimental runs, samples are taken at 2 minutes intervals from different sampling points to detect the best one.

RESULTS AND DISCUSSION

1. Effect of sampling point location

The effect of sampling point positions is clearly shown in Fig. (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 lower sampling point and this is attributed to that gas flotation process is 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 (Fig. 3) attach themselves to the wax droplets (Stewart 1988). The wax appears lighter because the density difference between the wax-air agglomerate 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 that 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 increases with time until 6min then with further increase in time the wax concentration decreases. This is attributed to that after nearly 6 min, all the wax droplets present in the column in the height below 20 cm is carried by air bubbles from the bottom of the column to the top of it and 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 and so the air bubbles starts to carry the oil droplets from height above 20 cm to the top of the column and so the wax concentration starts to reduce. The same is right with respect to the sampling point at height 40 cm where in 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 then air bubbles start to carry the wax droplets from the top it self and so the wax concentration at the top of the column is increased at the start of the flotation process till time 16 minutes then with increasing the time the wax concentration is decreased.

2. Effect of different amount of soap

Fig. (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 oil water emulsion is increased till the maximum value of 99.6 % at 0.5 g and time of flotation of 20 minutes.

This increase of the percent removal of the wax can be explained as follows when soap is acting as foaming agent the amount of 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.

3. Effect of air flow rates and flotation time on percent removal of wax

Fig. (5) shows the effect of flotation time on the percent of wax removed during the flotation process while Fig. 6 shows the effect of air flow rates. Both figures show that with increasing the inlet air flow rate from 538 ml / min to 1814 ml/min, the percent removal of wax is first increased by increasing the air flow rate till inlet air flow rate reaches a value of 1559 ml / min and then it decreases with no further increase in the inlet flow rate. This can be explained as follows: 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 ml/min to 1559 ml / min the number of air bubbles per unit volume is increased and so its ability to carry wax droplets from the O/W 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 1559 ml / min the percent removal of oil is decreased. This is not only due to air bubbles slugging, but also to the formation of eddies which hinder the smooth bubble / droplets rising which was clearly observed visually during the experimental work.

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. 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, 1995,1999).

Fig. 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 minutes. It was clear 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 ml /min to 1559 ml / min and this increase nearly constant after time 10 minutes for each inlet air flow rate.

4. Effect of initial concentration of the wax/ water emulsion

Fig. (8) shows the relation between the percent removal of wax and time at different initial concentration of wax/water emulsion. The figure clearly shows that with increasing the initial concentration of wax in the oil water emulsion the percent removal of wax is highly. The decrease for 500 ppm initial concentration compared to that for 250 ppm is somewhat undetectable. This means that the initial concentration of wax/water emulsion does not affect the process up to 500 ppm.

When the concentration of wax in oil/water emulsion is 250 ppm and the air flow rate is 1559 ml/min the produced bubbles have the ability to remove most of the wax droplets present in the oil /water emulsion and when the oil concentration is increased from 250 ppm to 500 ppm and the inlet air flow rate is still 1559 ml/min it appears that the produced air bubbles have the ability to remove the increased amount of wax droplets that result from increasing the wax percent in the O/W emulsion, but when the wax concentration in the W_x/W_a emulsion is increased from 500 ppm to 750 ppm at the same inlet air flow rate of 1559 ml/min, it is noticed that the percent removal of oil is dramatically decreases 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 O/W 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 can not carry such wax droplets so that gas flotation can not work effectively. (Arnold, 1998).

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.

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 iis 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. The kinetics of an oil flotation process can be expressed by the following relation:

$$- dC/dt = K\,C^n$$

where dC/dt is the collection rate and K is the distribution function of the rate constant.

5. Effect of pH values of the emulsion

Fig. (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 air flow rate, initial concentration and height of the batch charge. It is clear that the percent removal of wax from the emulsion increased with increasing pH, taking into consideration that the percent removal of wax is calculated with respect to the 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.

The addition of acid decreasing pH value of the wax water emulsion from 9 to 5 separating result is spring fatty acids from their soaps and can be easily separated, this process is commonly used in treating the oil/water emulsion produced from the scouring process (acid cracking) and this is clearly shown from the data where the turbidity of the O/W emulsion is decreased with decreasing the pH value of the oil water emulsion. Further removal of the oil 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.

6. Effect of working heights of emulsion on flotation process

Fig. (10) shows the relation between the percent removal of wax and time at different working heights of emulsion in the flotation column, while Fig. (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 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. This is since at low working height the chance of forming eddies and turbulence is high so that wax doesn't attach firmly with the air bubbles (diverse flotation process) by increasing the working height from 40 cm to 60 cm the percent removal is decreased. It seems that the longer upward travel of wax droplets causes some separation of wax by loosening its attachment to the air bubbles.

7. 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 it shown down. In the first period of flotation where the number of air bubbles per unit volume of the emulsion is high most of wax droplets are removed rapidly and the percent removal of wax is then high by further increase time of flotation the remaining part of wax droplets which is actually smaller than that removed in the first period gets removed at a slower rate..

Fig. (12) shows the relation between flotation time and percent recovery at the optimum flotation conditions.

Within the first four minutes nearly about 80 % of wax was recovered, while within in the following 4 minutes only 8 % recovery was achieved decreasing to only 4 minutes only 4 % recovery afterwards. It is clear that from the economic point of view that the optimum time is 8 minutes of flotation time which can be increased to 10 minutes for ensuring total gain of wax

CONCLUSIONS

In the batch flotation process achieved, the percent wax recovery was found to increase by increasing the flotation time, the optimum flotation time was 10 minutes and attain 90 % wax recovery. The optimum working height was found to be 40 cm and this is eight times the diameter of the flotation column. The highest percent recovery is obtained at pH value of 9.

The optimum inlet flow rate was found to be 1559 ml / min. The percent wax recovery was decreased with increasing the initial concentration of wax water emulsion; 500 ppm is the optimum initial concentration for recovery of wax. The optimum soap amount was found to be 0.5 g, which gives the highest percentage recovery of wax. 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.

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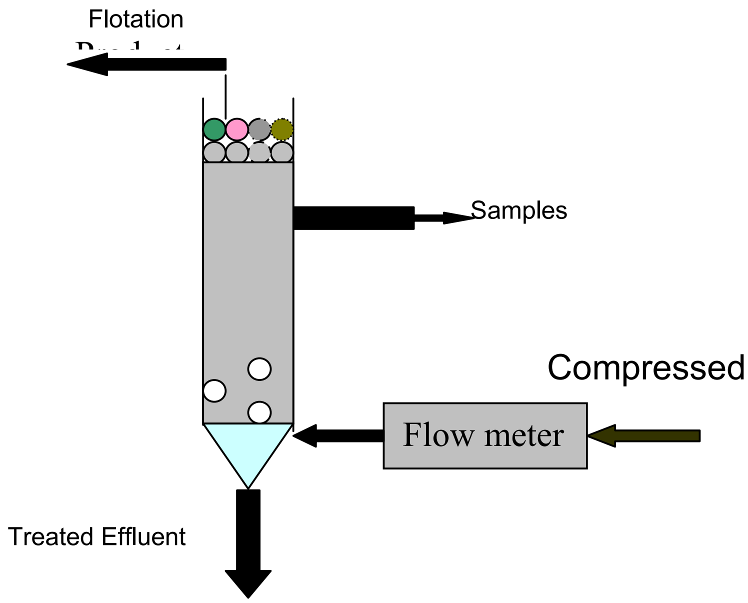

Fig. 1: Sketch of a Flotation Column

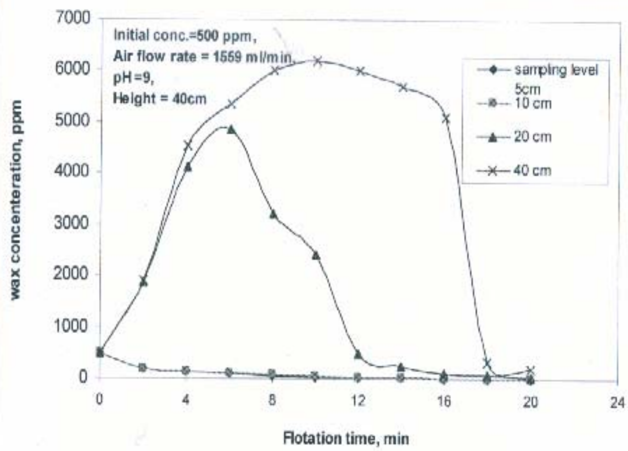

Fig. 2: Effect of sampling level on wax concenteration

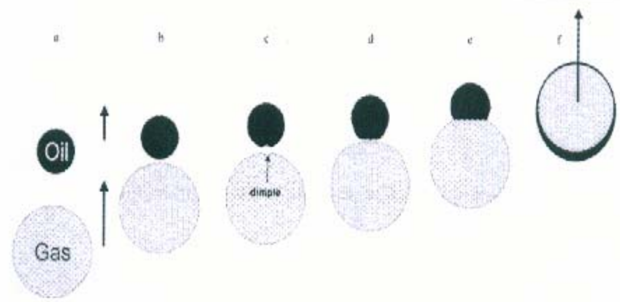

Fig. 3. The attachment process. (a) Bubble and drop approach; (b) water film thinning; (c) thin film dimples due to interfacial tension gradients; (d) the dimple disappears as film drains; (e) at a critical thickness film ruptures, and if spreading conditions are present, the oil spreads around the gas;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

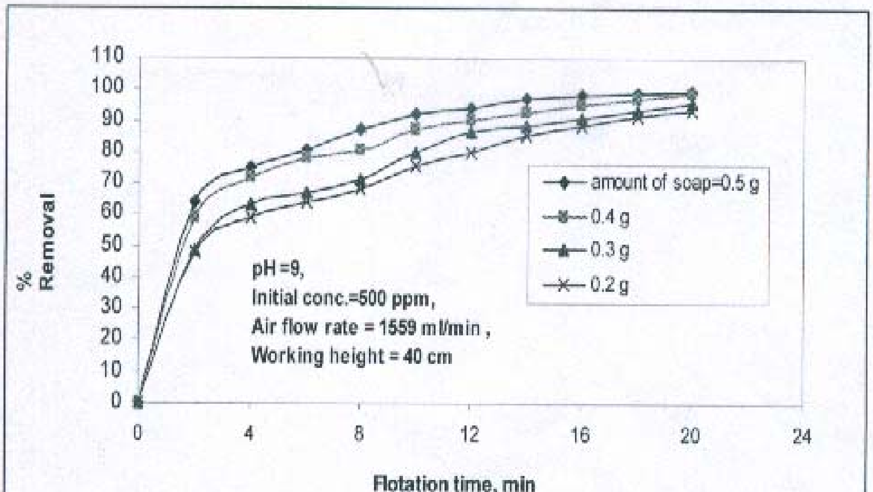

Fig. 4: Effect of amount of soap in the scouring process on % removal of wax during flotation

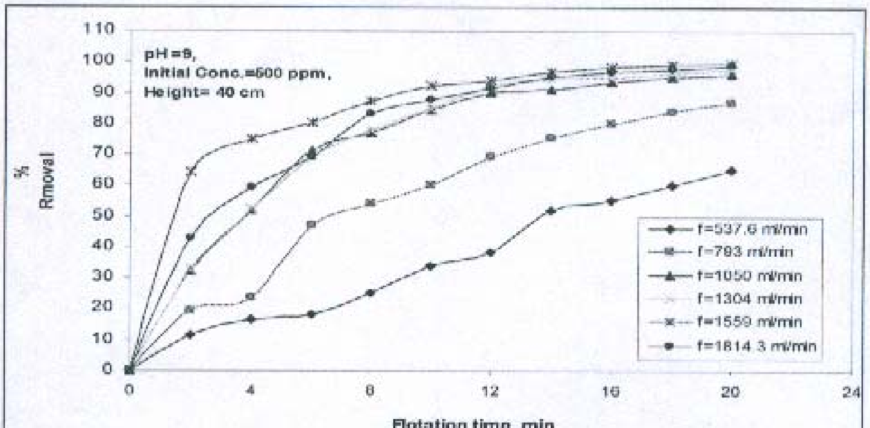

Fig. 5: Effect of air flow rate on % removal

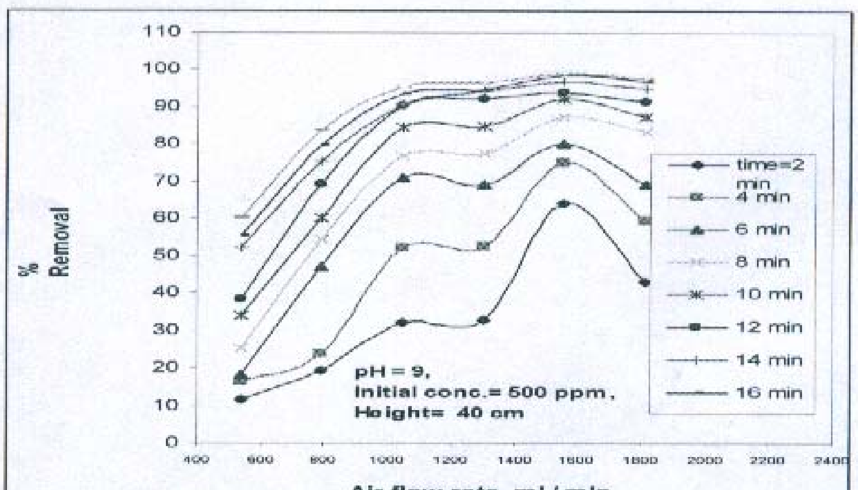

Fig.6: Effect of air flow rate on % removal

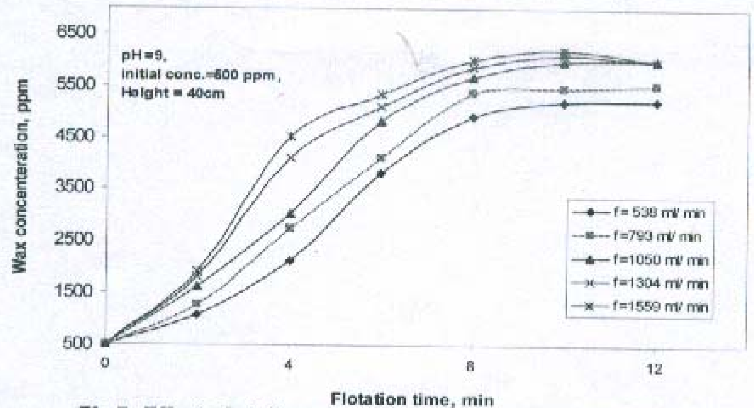

Fig.7: Effect of air flow rate at the heighest sampling point on wax concenteration

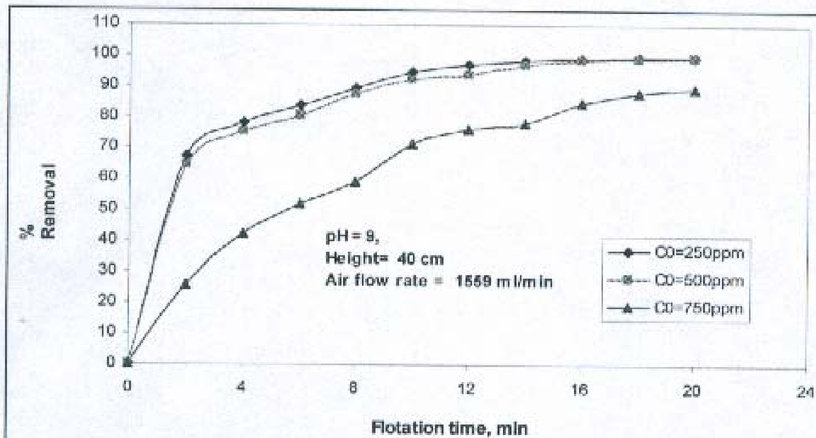

Fig. 8: Effect of initial concenteration of wax on % removal

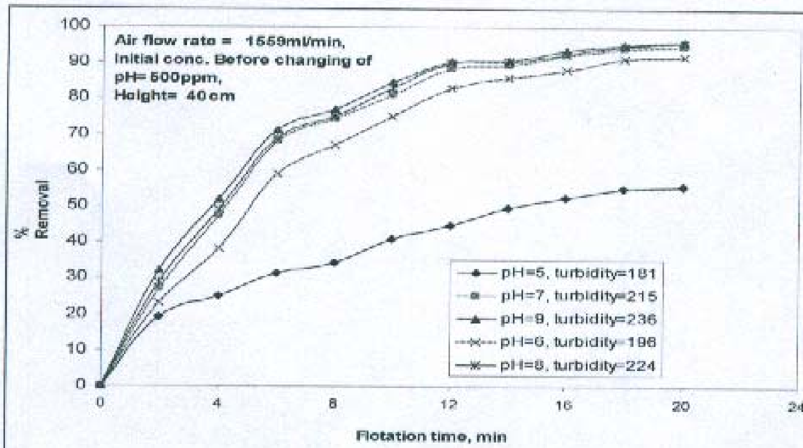

Fig. 9: Effect of pH on % removal

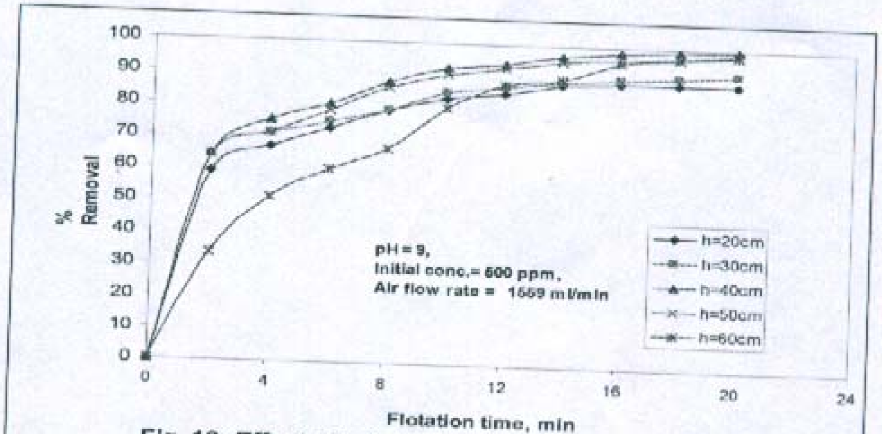

Fig. 10: Effect of height of waxy water in the column on % removal

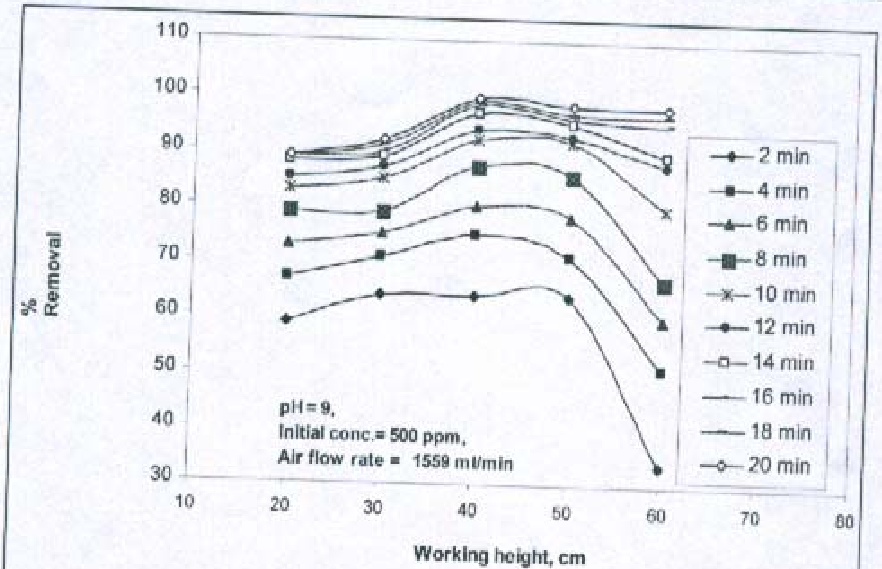

Fig. 11: Effect of height of waxy water on % removal

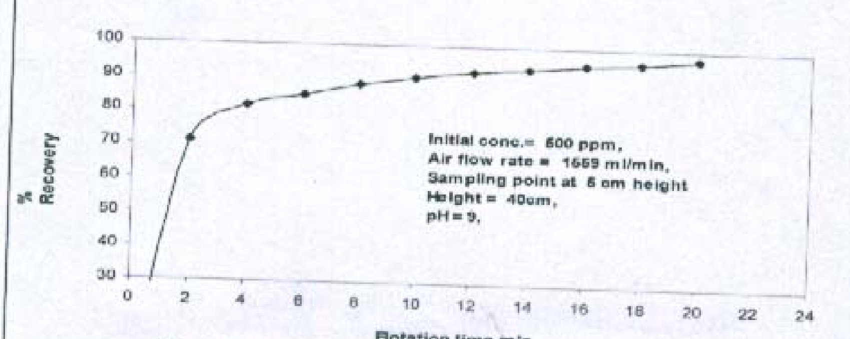

Fig.12 : Effect of time of flotation on % recovery