

# Deinking of Black Toner Ink from Laser Printed Paper by Using Anionic Surfactant

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**Abstract:** The objective of this research was to synthesize natural anionic surfactant from renewable material and use of this surfactant for ink removal from printed paper via flotation deinking in paper recycling process. The work included the preparation of the castor seed raw material, extraction of castor oil, preparation of anionic surfactant and deinking flotation of waste printed paper. Foamability and foam stability, critical micelle concentration and hydrophilic lipophilic balance of the surfactant was characterized and used to evaluate its fundamental deinking ability properties for the purpose of deinking flotation. The hydrophilic-lipophilic balance (HLB) of the fatty acid soap of 19.48, average Foamability height of 49cm and critical micelle concentration (CMC) of 0.0065M was obtained. Lab scale flotation deinking process was done and the basic flotation parameters like flotation time, fatty acid soap dosage and pH were optimized for evaluating deinking efficiency. The flotation deinking efficiency was probed by measuring the residual ink concentration of the hand sheet via Perkin Elmer spectroscopy before and after deinking flotation. The individual and interaction effects between the basic parameters were studied by using design Expert Software 6.0.8. For the deinking flotation, the maximum ink removal efficiency was determined to be 78.02% at the flotation time of 90min, fatty acid soap dosage of 1.5% and pH of 9. Increasing flotation time from 30min up to 90 min and decreasing fatty acid dosage from 3% to 1.5% and increasing of pH from 3 up to 9 were found to have increased the ink removal efficiency.

**Keywords:** Anionic Surfactant, Waste Paper, Flotation Deinking, Hand Sheet, Effective Residual Ink Concentration

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## 1. Introduction

Paper is a sheet of fibers with a number of added chemicals that affect the properties and quality of the sheet. In Ethiopia both the local produced and the imported paper met the demand of the society. However, due to the existence of only one paper factory in the country, i.e. Ethiopian Pulp and Paper Factory located at Wonji, the majority of the supply is from import.

During 1998/99- 2004/05 E. C, the best supply offered by the domestic manufacturer was 12,685 tons while the yearly average consumption during the same period was 146,296 tons [1]. To avoid this shortage of paper in the country, it is necessary to recycle the waste paper by removing its ink and other dirties from the fiber.

The paper recycling is the process of wash, removing and bleaching unnecessary spots and other materials from printed paper therefore, this can be sold as an alternative to new

produced paper manufacturing from original raw material. Recycling of wastepaper can have different uses and applications with respect to environmental, operating costs and energy consumptions compared to the various papers that produced directly from pulp. It has been estimated that for every ton of paper, which is made from 100% recycled wastepaper, 24 trees is saved. One ton of pulp made from deinked and bleached wastepaper requires 60% less energy than to manufacture a ton of bleached virgin Kraft pulp [2].

As the consumptions of paper increased from time to time, the need for a cheap resources grows radically, and the recycling of waste paper increased. The first step for recycling waste paper is the removal of inks from the printed paper because it affects the quality and brightness of paper. The main technique for removal of ink is deinking flotation. This is a process to selectively separate the hydrophobic ink particles from the hydrophilic fiber particles.

## 2. Materials and Methods

### 2.1. Materials and Chemicals

The chemicals and analytical reagent grade used were sodium hydroxide, sodium sulfide, hydrochloric acid, distilled water, sulfuric acid, potassium hydroxide, N-hexane, phenolphthalein, Sodium chloride and ethanol. The equipments used during the experimentations includes laboratory autoclave, PerkinElmer spectrophotometer (AAU17 5839), aeration apparatus (Ch-EL254), Laboratory Pulper (EPP-3-01-5), Soxhlet extractor, Pulp disintegrator machine (EPP-3-01-6), Pressing machine (EPP-3-01-10), water bath(SBS40), sheet forming machine (EPP-3-01-9), and beating machine (EPP-3-01-05).

### 2.2. Methods

#### 2.2.1. Preparation of Anionic Surfactant

Sodium stearate of the molecular formula  $\text{CH}_3(\text{CH}_2)_{16}\text{COO}^-\text{Na}^+$  is anionic surfactant used for flotation deinking purpose. To manufacture this surfactants: a sample of the extracted refined oil was weighted into a 1000 mL glass beaker, and then 1000mL of ethanol water mixture (1:1) was added to the same beaker followed by 25.4g of sodium hydroxide. The beaker was then fit to a boiling water bath with a temperature of 60°C for 30 min with occasional shaking.

After being heated for about 30 min, the odor of alcohol was disappeared, indicating the completion of the saponification reaction. A stock mass containing a mixture of the soap, glycerol, and excess sodium hydroxide was obtained.

To precipitate or "salt out" the soap, 150 mL of a saturated sodium chloride solution was added to the soap mixture while stirring vigorously. This process increases the density of the aqueous solution; therefore, soap was floated out from the aqueous solution and after this time it was put on filter paper to dry up.

#### 2.2.2. Experimental Flotation Deinking Process

The pulp was charged into column and floated in the presence of surfactants. The ink particles were attached to the air bubbles due to their relatively high hydrophobicity and were floated to the surface of suspension, and the hydrophilic fibers were remain in the water phase. A scraper was used for foam removal over the course of flotation. The removed foam was collected in a reject tank. The yield of the flotation was calculated once the reject was dried and was deducted from the original floated slurry weight [3].

#### 2.2.3. Pulp Hand Sheet Preparation

138g of oven dry pulp was mixed with 23 liters of water to make pulp slurry with consistency of 0.6%. Pulp slurry was added in to a beating machine and beat up by circulating it. Freeness of slurry was checked out at each ten minutes beating interval. This was done by taking 333ml of slurry from the beater (this contains 2gm of moisture free fiber) and then dilute to 1000ml with distilled water and measured

freeness value. When the freeness of pulp is 30CSF, 800ml sample was taken from beater and diluted to 2liters of water (0.17% consistency) and disintegrated at 1500rpm for five minutes. The beated and disintegrated pulp suspension was taken from the disintegrator and diluted to 4liters of water and agitated well by hand [4].

404ml from the diluted suspension was taken 60gm/m<sup>2</sup> was prepared by sheet forming machine. Ones the sheets are prepared two stage pressing was followed by applying 0.47MPa pressure for four minutes by pressing machine. Then the stocks are removed from the press and attached to the drying plates in order to dry by oven at 130°C for 45 minutes. The prepared sheets were then tested for the residual ink concentration.

#### 2.2.4. Evaluation of Deinked Pulp

The comparison of the blank hand sheet with those from the flotation runs allowed for determining the percentage of deinking efficiency. Deinking efficiency can be evaluated by measuring Effective Residual Ink Concentration (ERIC) of hand sheets made from the recycled paper before and after deinking. The hand sheets (6gsm) were prepared according to accepted procedure (TAPPI Standard) at Ethiopian pulp and paper industry located at Wonji. Hand sheets were prepared for measuring ink concentration of sheet.

Select the 950 nm filter position or modified spectral equivalent. Place the hand sheet on the instrument and read and record the reflectance of the sheet ( $R_\infty$ ). Place the sheet on the instrument and back it with the black cavity, Read and record its reflectance ( $R_0$ ). The scattering coefficient (s) of the sample at 950 nm was calculated by:

$$S = \left[ \frac{R_\infty}{W(1-R_\infty^2)} \right] \quad (1)$$

Where w = grammage (g/m<sup>2</sup>) and where  $R_0$  and  $R_\infty$  are expressed as decimals. Their ratio between  $K_{paper}$  and  $K_{ink}$  is defined as effective residual ink concentration (ERIC), with a unit of ppm.

$$K = s \frac{(1-R_\infty)^2}{2R_\infty} \quad (2)$$

$$\text{ERIC} = \frac{K_{\text{sheet}}}{K_{\text{ink}}} \times 10^6 \text{ (ppm)} \quad (3)$$

Black inks have been shown to have an absorption coefficient of about 10,000 m<sup>2</sup>/kg [5]. This was considered to be a default value for this method (i.e.  $k_{ink} = 10,000\text{m}^2/\text{kg}$ ). If an insitu means is available for determining the absorption coefficient for the ink, the value used should be reported [5].

Deinking efficiency of the paper hand sheet could be calculated as follow:

$$\text{IE\%} = \frac{\text{ERIC}_{\text{UP}} - \text{ERIC}_{\text{DP}}}{\text{ERIC}_{\text{UP}} - \text{ERIC}_{\text{UNP}}} \times 100\% \quad (4)$$

Where,  $\text{ERIC}_{\text{UP}}$  and  $\text{ERIC}_{\text{DP}}$  are the residual ink concentration for un deinked hand sheet (pulp) and deinked hand sheet (pulp) and  $\text{ERIC}_{\text{UNP}}$  is unprinted paper.

### 3. Results and Discussion

#### 3.1. Characterizing of Anionic Surfactants

The Critical micelle concentration was 0.0065. And hydrophilic-lipophilic balance values describe how the surfactant interacts with water soluble and water repellent substances. In deinking flotation it has been suggested that surfactants with an HLB value of 14-20 are optimal for deinking of paper and magazine mixtures [22]. Foamability is the foam volume immediately after the generation of the foam, and the liquid content of the foam.

#### 3.2. Effect of Deinking Flotation Process Variables

##### 3.2.1. Effects of Flotation Time on Deinking Flotation

As shown in Figure 1 below the ink removal efficiency

was significantly affected by square flotation time. It can be seen from the figure that with increasing flotation time the ink removal efficiency increases up to it reaches 60min and after this point reversing its trend because the pulp brightness can actually decrease somewhat with further increase of flotation time. Additionally, if the flotation time is increasing beyond its maximum point it can lead to lower fiber yield and removal of its whiteness. This long flotation time has an effect on darkening the fiber by removing the bright fillers and pigments. Besides this, when the flotation time is too low, the foam generated would not stable enough and they need enough time to be stable and able attach with air rather they would return back in to the fiber. This low flotation time has a diverse effect, since more ink needing to be collected means that an optimum flotation time was required to reach the target pulp brightness.

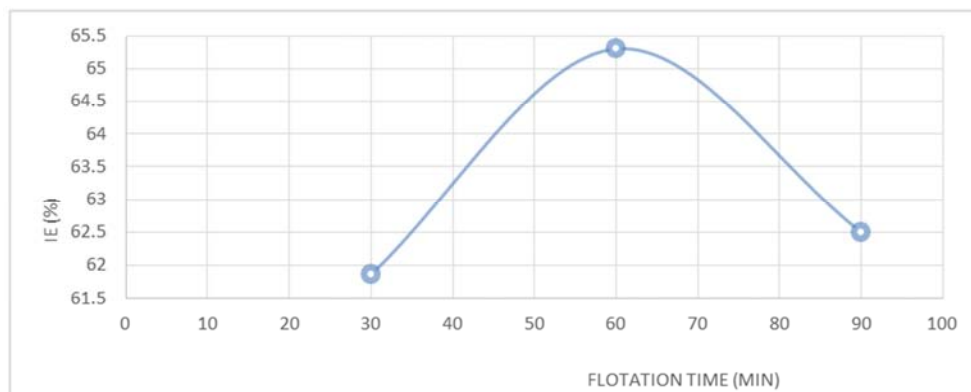


Figure 1. ink removal efficiency Vs flotation time.

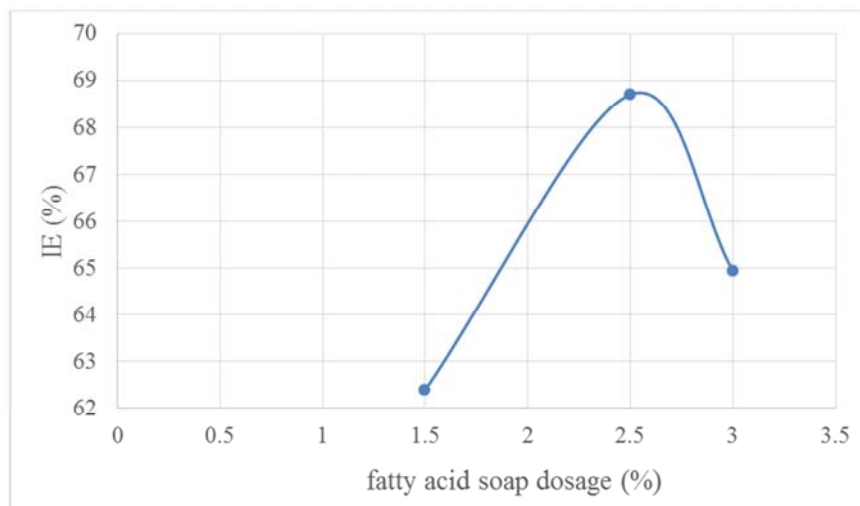


Figure 2. Fatty acid soap Vs ink removal efficiency.

##### 3.2.2. Effects of Fatty Acid Soap Dosage on Flotation Deinking

As shown in figure 2 the ink removal efficiency was increased with fatty acid soap dosage but when it reaches 2.5% (based on ODF) changes its slope downward and this was due to the reasons of increasing their coagulation speed and absorb on the surface of the fiber, this leads to increase the

hydrophobicity property of the fiber and This leads to the result of exactly stability of the ink particles and thus a reduced amount of ink attached to the air bubbles in flotation. Therefore the graph concludes excess dosage of fatty acid soap would affect the deinking flotation process. On the other hand, a lower limit for the dosage of fatty acid soap also seems to exist. Optimum fatty acid soap per dry fiber looks to be most favorable in this study.

### 3.2.3. Effects of pH on Flotation Deinking

In deinking flotation, the pH of the pulp slurry was important because it helps to determine the extent of ionization, hydrolysis and the charge of surfactant on the surface of the fiber slurry. This in turn influences the possibility of the collector/fatty acid soap to attach on the fiber surface. Therefore, at the various ionized solid/liquid interfaces, pH can either helps or hinders the adsorption of the surfactant and contributing to greater or lesser selectivity of deinking flotation. Acidic medium deinking flotation has a

risk to damage the equipment due to their more negative charges imposed on the contaminant and substrate to repulsive each other. The removal efficiency increased with increasing pH up to 9, which means there was higher concentration of sodium hydroxide that was used to swelling up the fiber and helps to detach the ink from the fiber easily. And low pH levels also convert acids at the fiber surfaces to their unionized foams, making the fibers more hydrophobic and hence, more difficult to flotation.

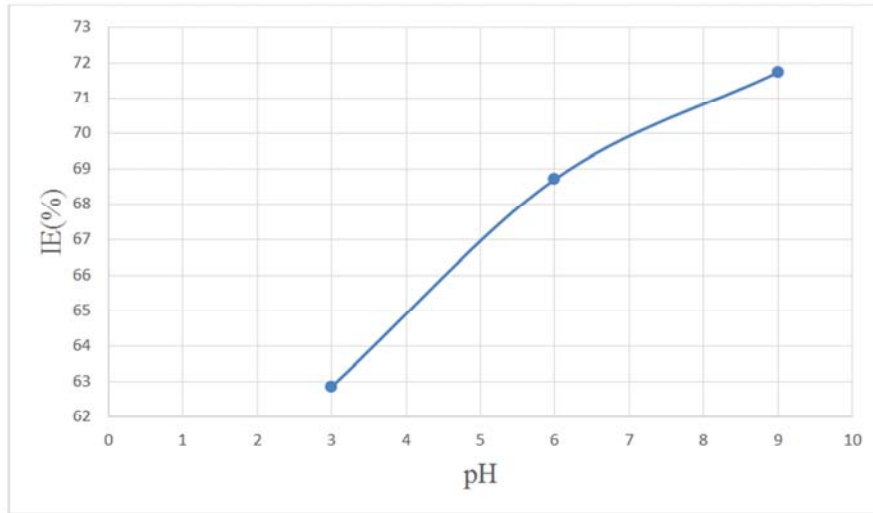


Figure 3. pH Vs ink removal efficiency.

### 3.2.4. Interaction Effects of Flotation Time and Fatty Acid Soap Dosage

The Figure 4 and 5 Below shows the 3D response surface and counter plots for the ink removal efficiency generated by the predicted model respectively. in the counter plot, ink removal efficiency increased until the flotation time reached 60min, and then it decreased. And also, increasing the fatty

acid soap dosage from 1.5% to 2.5% led to increase in the ink removal efficiency and begins to decrease when the fatty acid soap dosage was further increasing from 2.5% to 3%. Based on the below graphs (Figure 4 and 5), optimal conditions for ink removal efficiency was 66.5% with a desirability of 0.5311 at 2.5% (based on ODF) and 60min flotation time with constant pH of 6.

DESIGN-EXPERT Plot

IE

X = A: Flotation time

Y = B: Fatty acid soap

Actual Factor

C: pH = 6.00

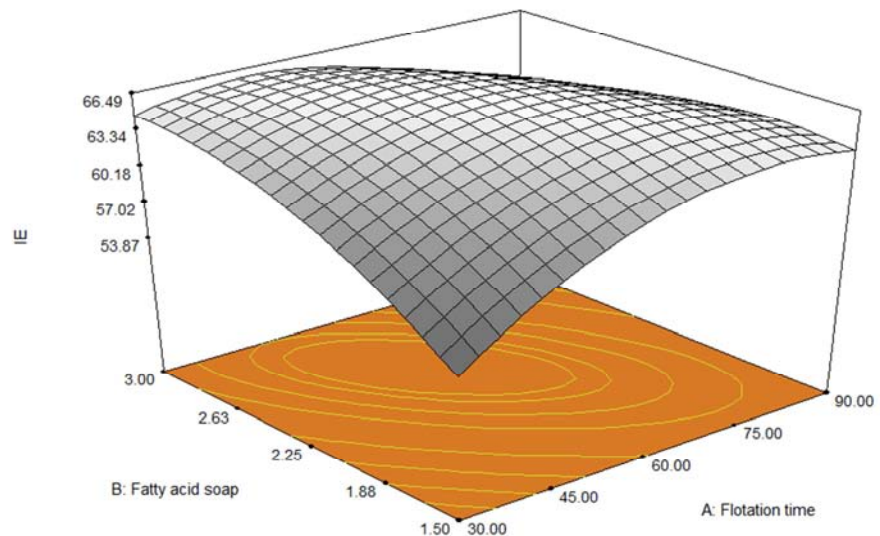


Figure 4. Surface plot of the interaction effect of flotation time and fatty acid soap on ink removal efficiency.

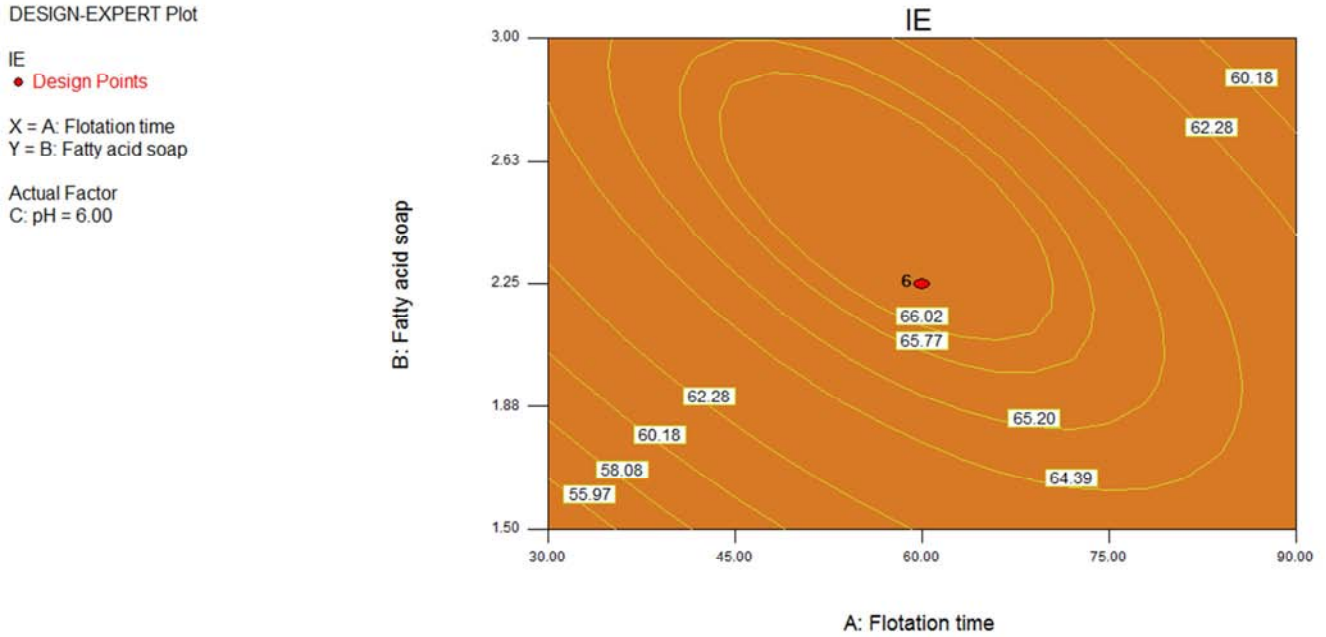


Figure 5. Contour Surface plot of the interaction effect of flotation time and fatty acid soap on ink removal efficiency.

### 3.2.5. Interaction Effects of Flotation Time and pH

The effects of the deinking flotation parameters on the ink removal efficiency were plotted in Figure 6 in 3-D surface plot and in figure 7 in 2-D contour plots.

These figures shows the effect of the flotation time and the pH on the ink removal efficiency maintaining the fatty acid soap dosage constant at optimum value (i.e. 2.25%). As can be seen, the interaction effect between the variables (flotation time and pH) was obvious from both the 3-D response

surface and the contour-line plots. The increase of flotation time from 30min to 72.30 min leads to an increase of the ink removal efficiency from 61.09% to 72.4% and it begun to decreases when the flotation time was further increasing from 72.31min to 90 min to 56.1%. On the other hand, the effect of the pH was stronger at higher pH. Moreover, the increase of the pH from 3 to 9, increases the ink removal efficiency from 56.71% to 72.4%.

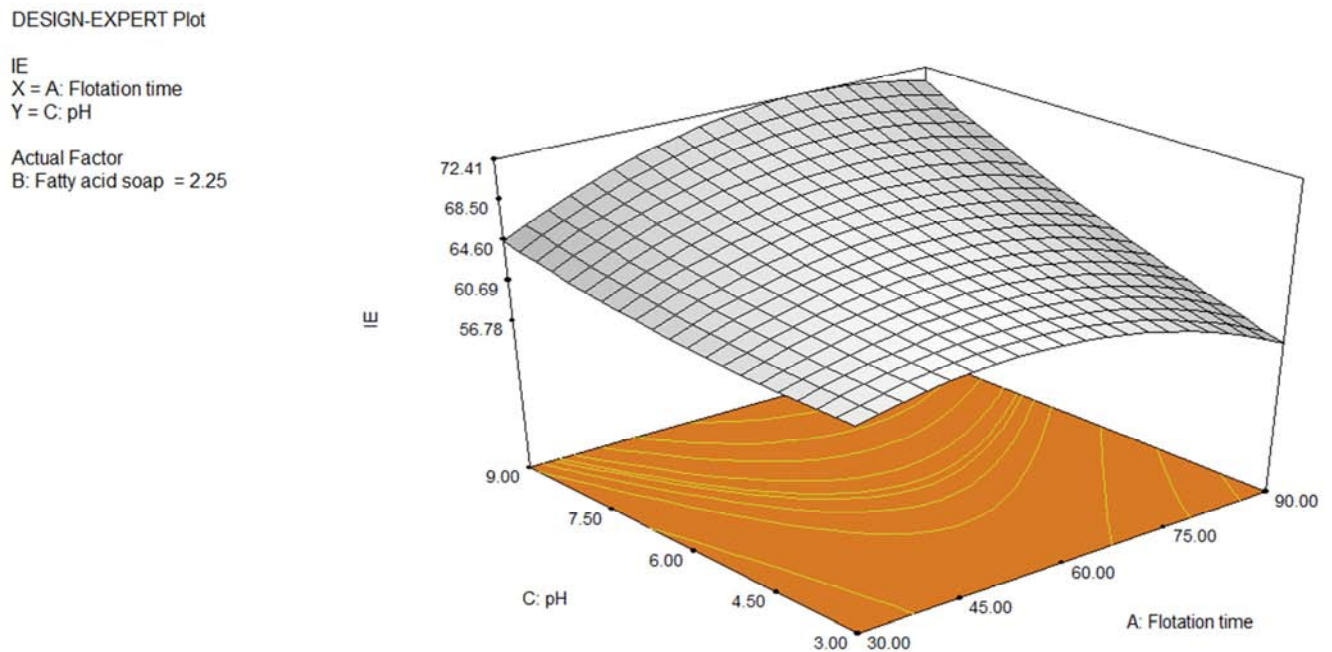


Figure 6. Surface plot of the interaction effect of flotation time and pH on ink removal efficiency.



DESIGN-EXPERT Plot

IE

● Design Points

X = A: Flotation time

Y = C: pH

Actual Factor

B: Fatty acid soap = 2.25

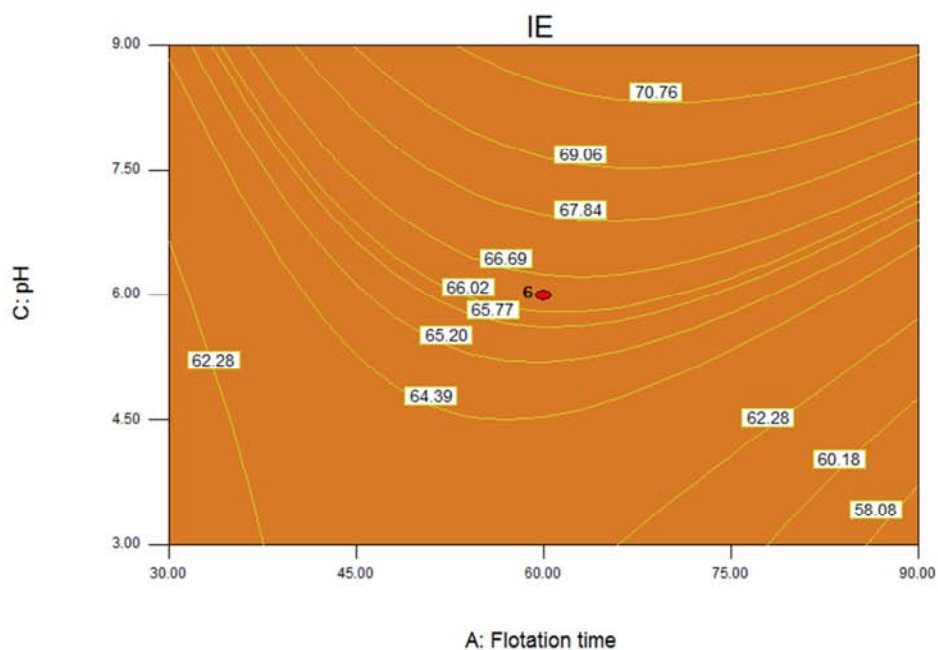


Figure 7. contour Surface plot of the interaction effect of flotation time and pH on ink removal efficiency.

### 3.2.6. Interaction Effects of Fatty Acid Soap Dosage and pH

The ink removal efficiency was increasing when the fatty acid soap dosage increases from 1.5% to 2.65% then begins to decrease when the fatty acid soap dosage increases further

from 2.65% to 3%. the ink removal efficiency was increasing with increasing the pH from 3 to 9. Therefore the optimum deinking flotation efficiency was at pH of 9 and fatty acid dosage 2.65% (based on ODF) at constant 60min flotation time.

DESIGN-EXPERT Plot

IE

X = B: Fatty acid soap

Y = C: pH

Actual Factor

A: Flotation time = 60.00

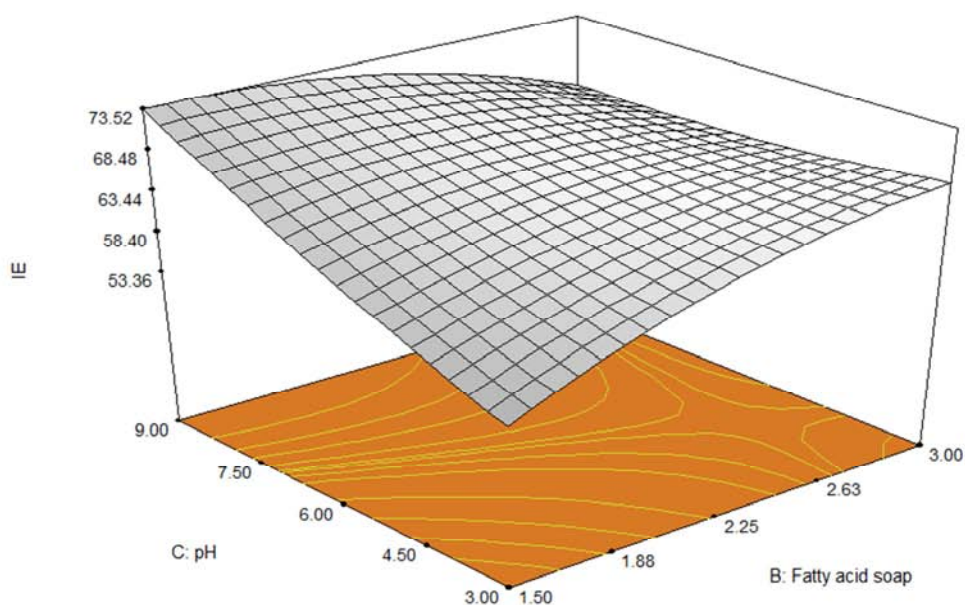


Figure 8. Surface plot of the interaction effect of fatty acid soap and pH on ink removal efficiency.

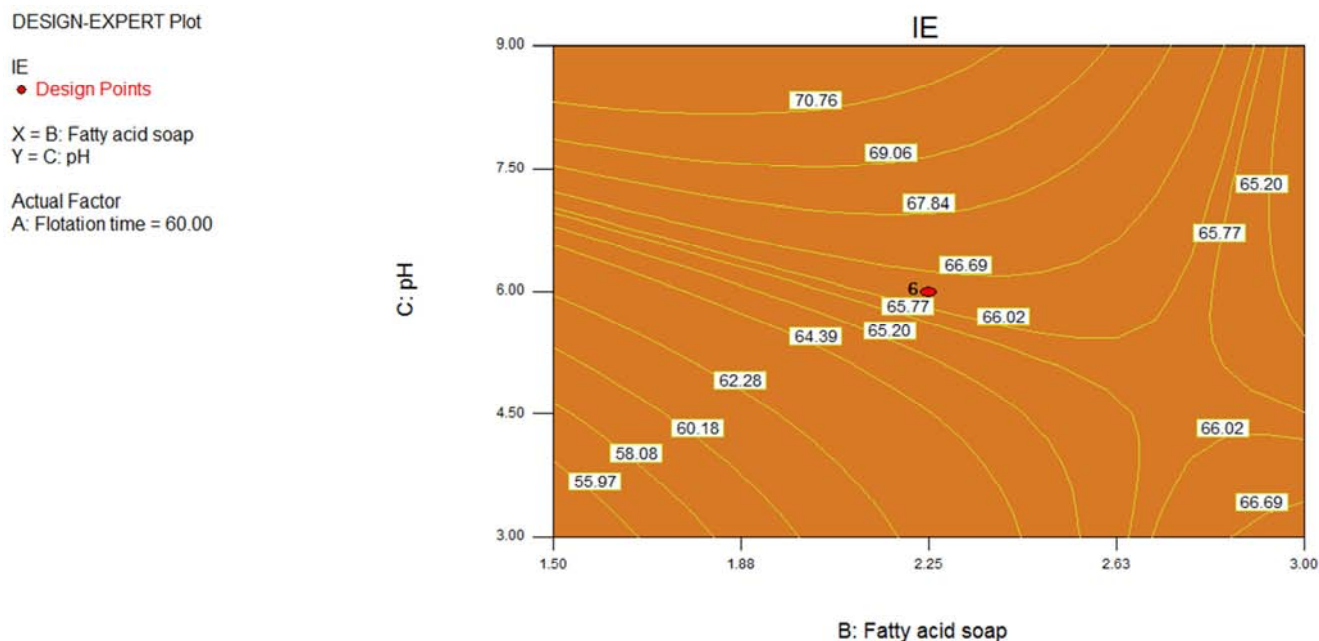


Figure 9. Counter plot of the interaction effect of fatty acid soap and pH on ink removal efficiency.

## 4. Conclusion

The three deinking flotation process parameters affecting the ink removal efficiency which are flotation time, fatty acid soap dosage and pH has been studied. A flotation time of 90min, amount of fatty soap of 1.5% (based on ODF) and 9pH results an optimal value of 78.0219% removal efficiency. The physicochemical properties determined for the fatty acid soap are HLB, CMC, foaming power & foam stability. This work investigated deinking efficiency of two steps (pulping /flotation) using flotation cell equipped with aeration apparatus.

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