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EFFECT OF REACTION TEMPERATURE AND TIME ON THE RECOVERY OF GLYCEROL FROM SPENT LYE

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ABSTRACT

The effect of reaction temperature and time on the recovery of glycerol from spent lye was investigated using $Al_2(SO_4)_3$ as the precipitating agent and a coagulant followed by subsequent evaporation of the water of solution. Reaction temperatures of 60, 70, 80, 90 and 100°C with varying reaction times of 45, 60 and 90 minutes were investigated. Results show that with a rise in reaction temperature from 60 to 90°C, there is an increase yield of glycerol and a decrease as the temperature rises above 90°C. For all reaction time investigated, a maximum glycerol yield of 11.96% was obtained after treating the spent lye with concentrated HCl and $Al_2(SO_4)_3$ at a reaction temperature of 90°C with a reaction time of 90 minutes. The plots obtained can be used as correlations to study glycerol recovery from the treatment of spent lye at different saponification reaction temperatures and times other than the once used in this work

KEYWORDS: Glycerol Recovery, Spent Lye, Reaction Temperature, Reaction Time

INTRODUCTION

Spent lye (soap lye) is a by-product of soap production through the Saponification process (Kirk-Othmer, 1980; Daniel, 1981; Buchingham, 1982). In the process of soap manufacturing (Shreve, 1977; Danielson, 1973; Source Category Survey, 1980), fat is boiled with caustic soda solution and salt. The fatty acid constituents of the fat combine with soda to form soap, which is obtained as an upper layer and lower aqueous layer referred to as spent lye, contains glycerol, water, salt and unchanged caustic.

The chemicals most commonly used to remove impurities from spent lye are alum, ferric chloride, ferric sulphate, hydrochloric acid and caustic soda. The aluminium and iron salts are used as coagulants and precipitant and the others serve as precipitant and neutralizers. In the first stage of operation, spent lye is commonly boiled with fatty acids to reduce the content of free caustic and soda ash. After cooling the solid soap is skimmed off, and the spent lye is subjected to a two-stage treatment step.

The first stage is the addition of an acid and a coagulant followed by filtration, removing the bulk of the organic impurities. In the second stage caustic soda is added to the filtrate to precipitate and remove the balance of coagulants and to regulate pH of the liquor to a point at which it is least corrosive to subsequent process treatment (Kirk-Othmer, 1980).

Glycerol is completely soluble in water and alcohol but insoluble in ether, benzene, and chloroform. It is a clear, colourless syrupy liquid, odourless, hydroscopic and having a sweet taste. It has a low mammalian toxicity. Other physical properties of glycerol are given by Dean (1979). Glycerol plays an important role in nearly every industry (Shreve, 1977).

Although investigations on spent lye (Alaohuru,1995; Ooi et al, 2001 and Hedtke, 1996) has been reported, none on the effect of temperature and reaction time is known to be available in the open literature. This paper reports on the effect of reaction temperature and time on glycerol recovery from spent lye using $Al_2(SO_4)_3$ for the chemical treatment.

EXPERIMENTAL

MATERIALS

Crude Palm Kernel Oil (PKO) was obtained from RIVOC Port Harcourt with a clean jerican. All the reagents and chemicals (Potassium hydroxide, Concentrated HCl acid, Sodium Chloride, 95 - 97% Sulphuric Acid , Aluminium Sulphate($Al_2(SO_4)_3$) used were of analytical grade Constant temperature water bath, Beakers, Electric Heater Filter papers, Electric Stirrer, Separating Funnel, Thermometer, Weighing Scale.

ACID WASHING OF THE PALM KERNEL OIL (PKO)

Crude Palm Kernel Oil (PKO) was acid washed with dilute H_2SO_4 based on 1g 95 - 99% H_2SO_4 per 100g palm kernel oil. The equivalent 95 - 99% was diluted to 0.1 M H_2SO_4 before use for the acid washing. This process served as a pre-treatment of the crude PKO by partially bleaching the dark brown oil into a lighter one.

BLENDING OF ALKALI AND FAT

193.50 ml of 1.0 M KOH was mixed with 50 ml of PKO prior to saponification, based on a saponification value of 245g. This is consistent with the work of Okeke et al (1989).

SAPONIFICATION

The blended KOH and oil was placed in a constant temperature water bath at different reaction temperatures of 60°C, 70°C, 80°C, 90°C and 100°C. The solutions were vigorously and continuously stirred and allowed for different reaction times of 45, 60 and 90 minutes respectively. Sodium chloride crystals were added to the samples to salt out the soap. The spent lye, which contains glycerol, water, salt, unchanged caustic and fatty acids absorbs the sodium chloride and caused phase separation between the lye and the soap. The upper layer is the soap while the lower aqueous layer is the spent lye solution.

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TREATMENT OF SPENT LYE TO REMOVE IMPURITIES

On addition of 20g of sodium chloride into the soap lye solution, the entire mass was poured into a separating funnel, where there was a phase separation. The lower aqueous layer (spent lye) was run off and the mass noted by weighing. The spent lye was treated in the following way for the recovery of glycerol

5mls of conc. HCl was added to the spent lye solution and put into a constant temperature water bath at about 60^{0}C for 10 minutes. The solution was continuously stirred. $Al_2(SO_4)_3$ was added on a basis of 4g $Al_2(SO_4)_3$ per 1000g spent lye and after 2 minutes the solution was filtered and weighed. $Al_2(SO_4)_3$ was chosen as the reagent for the treatment because, the sodium sulphate (Na_2SO_4) formed is less soluble in the aqueous solution saturated with NaCl (Helmold,1993; Thomas,1983) and would crystallized out for easy recovery by subsequent evaporation and decanting, considerably reducing the dissolved salt in the crude glycerol (Ooi et al., 2001).

FURTHER TREATMENT

The crude glycerol was subjected to further treatment by way of heating noting that the boiling point of glycerol is 290^{0}C (Dean,1979). The water of the solution was evaporated to obtain a concentrated glycerol. The process of evaporation was discontinued when constant weight was obtained.

RESULT AND DISCUSSION

RESULTS

Tables 1 – 5 shows the results on the treatment of spent lye recovered as a by-product during the soap making process by saponification of different reaction temperature with varying reaction time.

Table 1: RESULT OF SPENT LYE TREATMENT FOR SAPONIFICATION REACTION AT 60^{0}C

t (min)	Mass of Spent lye recovered (g)	Mass of crude glycerol (g)	Mass of Conc. Glycerol (g)	Mass $Al_2(SO_4)_3$ added (g)
45	210.50	204.60	12.46	0.842
60	253.25	249.17	17.92	1.013
90	199.99	199.57	15.37	0.800

Table 2: RESULT OF SPENT LYE TREATMENT FOR SAPONIFICATION REACTION AT 70^{0}C

t (min)	Mass of Spent lye recovered (g)	Mass of crude glycerol (g)	Mass of Conc. Glycerol (g)	Mass $Al_2(SO_4)_3$ added (g)
45	247.93	242.63	19.27	0.992
60	228.12	224.59	21.49	0.913
90	199.70	199.30	20.41	0.799

Table 3: RESULT OF SPENT LYE TREATMENT FOR SAPONIFICATION REACTION AT 80^{0}C

t (min)	Mass of Spent lye recovered (g)	Mass of crude glycerol (g)	Mass of Conc. Glycerol (g)	Mass $Al_2(SO_4)_3$ added (g)
45	247.91	243.20	22.45	0.992
60	228.07	225.10	23.75	0.912
90	199.66	199.27	22.86	0.799

Table.4: RESULT OF SPENT LYE TREATMENT FOR SAPONIFICATION REACTION AT 90^{0}C

T (min)	Mass of Spent lye recovered (g)	Mass of crude glycerol (g)	Mass of Conc. Glycerol (g)	Mass $Al_2(SO_4)_3$ Added (g)
45	228.42	224.31	21.11	0.914
60	203.50	201.05	22.72	0.814
90	199.72	199.36	23.84	0.799

Table.5: RESULT OF SPENT LYE TREATMENT FOR SAPONIFICATION REACTION AT 100^{0}C

T (min)	Mass of Spent lye recovered (g)	Mass of crude glycerol (g)	Mass of Conc. Glycerol (g)	Mass $Al_2(SO_4)_3$ Added (g)
45	228.31	224.80	20.66	0.913
60	203.04	201.13	20.82	0.812
90	199.53	199.19	21.87	0.798

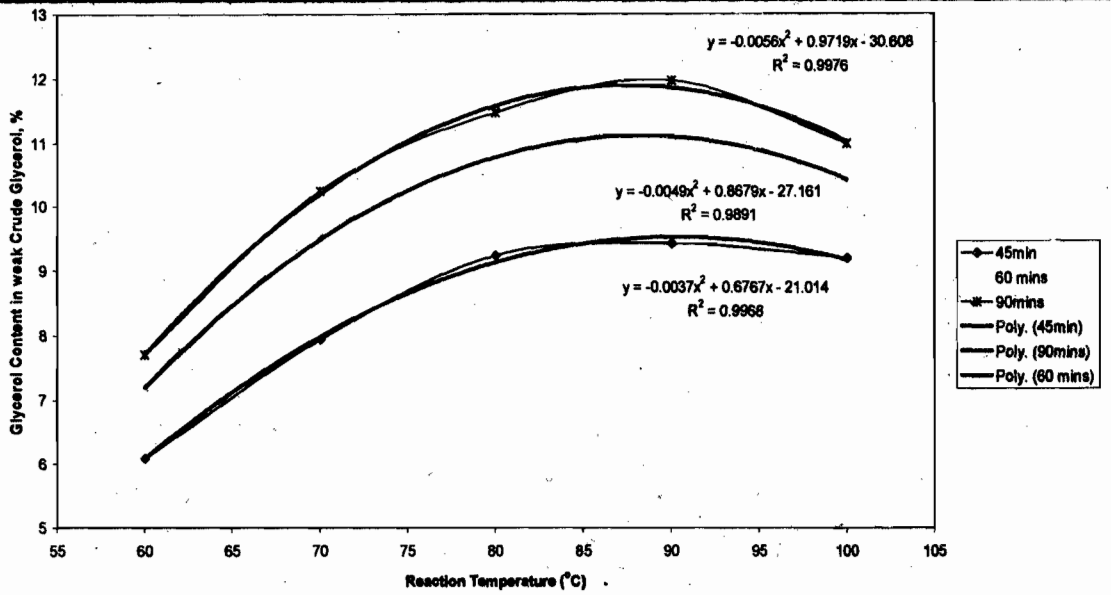

Figure 1: Glycerol Content in Weak Glycerol, % Against Reaction Temperature, °C

DISCUSSION OF RESULTS

RECOVERY OF GLYCEROL

Fig. 1 shows the plot of glycerol content in weak crude glycerol (%) with reaction temperature (°C), reaction time being a parameter. With increase in temperature from 60°C - 90°C, there is a rise in glycerol content, and a fall in the same from 90 -100°C. The glycerol content rose from 6.09% (at 60°C) to 9.41% (at 90°C) and fell to 9.19% (at 100°C) for a reaction time of 45 minutes. A maximum glycerol yield of 11.96% was obtained at 90°C and 90 minutes for reaction temperature and time respectively.
This is good enough since spent lye resulting from soap making processes generally contain from 8-15% glycerol (Kirk-Othmer,1980).

CONCLUSION

The effect of reaction temperature and time on the recovery of glycerol from spent lye has been investigated using $Al_2(SO_4)_3$ as both precipitant and a coagulant at different reaction temperatures and times. Results show that a maximum glycerol yield of 11.96% was obtained at a reaction temperature of 90°C with a reaction time of 90 minutes. Plots which can be used as correlations to study the effect of $Al_2(SO_4)$ as both precipitant and coagulant in glycerol recovery have been proposed.

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