RECOVERY OF GLYCERINE FROM SOAP-LYE

By P C. Guha and Ajoy Gupta

As the result of a request from the Director, Industrial Research Bureau, Delhi, dated 2nd October 1937, enquiring whether it will be possible to undertake research on the recovery of glycerine from soap-lye, in our laboratories, the problem was undertaken for investigation At the time of receipt of this request there was no factory in India to recover glycerine from soap-lye Recently, however, one or two factories *e g*, Tata Oil Mills, Swastik Oil Mills, have started manufacturing glycerine in this country

Uses and Applications of Glycerine

According to the import figures given in the Indian Sea Borne and Trade Journal, India imported 4189 cwt .of glycerine (price Rs 197520) in the year 1938 This figure indicates only the glycerine used as such, *e g.* in pharmaceutical and toilet purposes Glycerine is used extensively in the manufacture of explosives, as a moistening and sweetening agent for tobacco industry, and as an antifreeze agent for automobile radiators In the publication of the American Chemical Society Monograph series (1928) on glycerine, it is described that the world consumption of glycerine for explosives is 80 million pounds, for tobacco industry 25 million pounds, and for antifreeze machines 13 million lbs

Here below is given a list of the products of every day use in the manufacture of which glycerine forms one of the chief ingredients Tooth paste, toilet soap, toilet cream, water colour paints antiphlogistines, photography, lead cement, pharmaceuticals, hydraulic jack, wines of all kinds, confectionary, linolium, glycerine glue, solvent for aniline dyes, solvents for perfumes, cordials, textile, leather industry, paper industry, lubricants, rubber goods, preservatives, plastics duplicating compounds, inks, liquid soap, printers roll, gas meter, ha manufacture, synthetic resins

To give some further idea about the varied kinds of uses c glycerine, a list of chemicals derived from it is given below glycery

esters of phosphoric, boric, tartaric, succinic, malic, maleic, fumaric, citric, butyric, acetic, and formic acids, ethers of glycerine (*e.g.*, methyl, ethyl, naphthyl, dixylyl, phenyl), glycerine-mono-chlorohydrine, glycerol-dichlorohydrine, epichlorohydrine, allyl alcohol, starting material for synthesis of quinoline compounds, glycerol-β-acetyl sulphonate, acrolein, 1 3-butylene glycol, etc, and are used as explosives, gelatinizing agents, leather industry, textile, dyeing, dyestuffs, ceramic industry, as solvent for decorating colours, in electric coating compositions, solvents, cellulose products, cellulose product plasticizer, soap substitutes, wetting agents, synthetic perfumes, metallic colloids, poisonous gas preparations, disinfectants

The Quantity of Soap Produced and the Corresponding Amount of Soap-Lye Available Annually in India which could be utilised for Glycerine Manufacture

If the soap molecule is regarded roughly as $C_{17}H_{35}COONa$, and on the basis that 1 molecule of glycerine $C_3H_5(OH)_3$ corresponds to 3 molecules of soap, for the production of every 10 parts of soap there should be liberated one part of glycerine The total soap production in India (according to a report published in *Hindu* dated 15th April 1941) is about 75,000 tons per year Allowing 15 per cent as moisture content of the soap, the weight of the dry soap should be 63,750 tons according to which 6375 tons of glycerine liberated in the process of soap-making, worth about Rs 71,00,000 calculating at the rate of Rs 56/- per cwt * is being wasted.

Operations Involved

Soap-lye as it comes from the soap factories looks very dirty and contains various impurities, such as, dissolved and undissolved soaps, unsaponifiables, products of rancidity, albuminus matters, salts, excess of alkali, etc The following operations were carried out to recover glycerine from the soap-lye procured from Mysore Government Soap Factory containing, as analysis showed, about 7 45

* It is to be noted that the retail price of glycerine is about Re 1/- per lb

l of glycerine (a) Clarification-conversion of soap-lye into a itable for concentration, (b) concentration of the lye to crude ıe by removal of water in vacuum evaporators, and the ıeous removal of salt and other inorganic matters, (c) distill- f the crude glycerine with super heated steam, (d) final tion and concentration giving rise to pale straw coloured ıe Bi-distilled glycerine was obtained by repeating the last ıations (*viz*, c and d) each once

Experimental

Two samples of soap-lye were obtained from Mysore Govern- Soap Factory and analysed for its glycerine content, by both nate oxidation method (Glycerol and the Glycols, Lawrie,) as well as by the acetin method (Glycerol and the Glycols, ', p 241) Following are the percentages of glycerine as ed from two samples of deep yellow dirty looking alkaline on analysis

o I sample—Dichromate method	7 3871 and 7 3942	per cent
Acetin method	7 4208 and 7 4010	,,
o II sample—Dichromate method	7 2301 and 7 2875	,,
Acetin method	7 3102 and 7 3002	,,

ic gravities of the above two samples were found to be 1 10 099, respectively. Refractive indices were found to be 1 37 3695 respectively, at 30°C

The laboratory experiment—2000 G of the lye on treatment ial with commercial hydrochloric acid and alum, followed by ion gave 1980 g of a clear solution, which was then evaporated - vacuum, yielding crude glycerine (182 g), the weight of the ated salt being 180 g The crude glycerine on analysis was l to contain 75 per cent of glycerine.

182 G of 75 per cent crude glycerine on being subjected to lation with super-heated steam at about 180° gave 195 g of er cent glycerine, the dark brownish residue weighed 14 g

About 2 0 g of animal charcoal was added to 195 g of 67

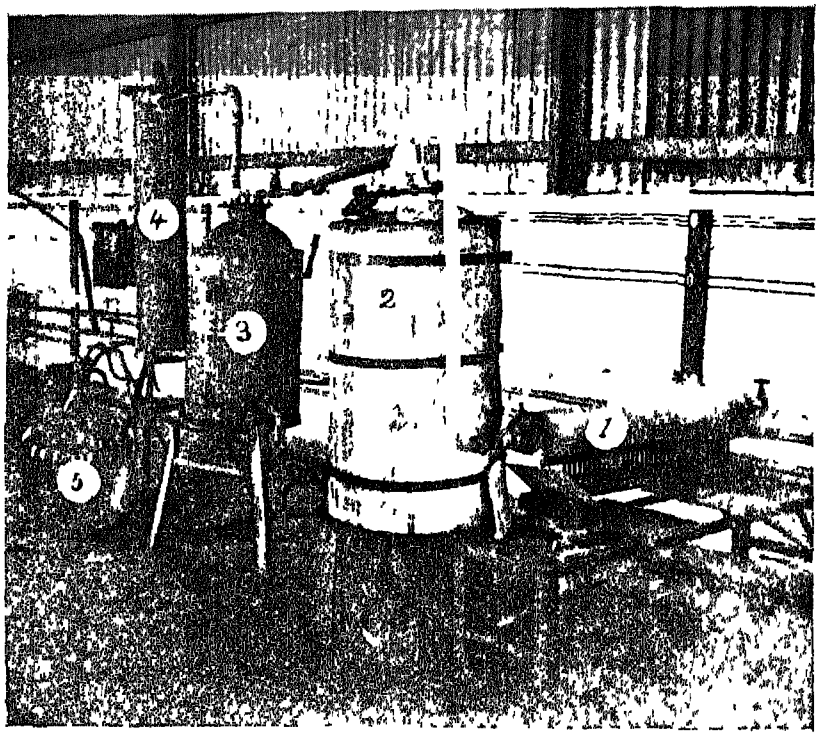
per cent glycerine and the mixture heated under stirring at 100° for about 2½ hours The clear filtered solution concentrated to 98 5 per cent pure glycerine (124 g) at 100° under reduced pressure This amounts to 6 2 per cent of glycerine, on the weight of the soap-lye

SEMI-LARGE SCALE EXPERIMENTS

(*a*) *Preliminary treatment*—200 lbs of the lye from the factory was first allowed to settle in a lead lined wooden tank and the suspended matters filtered off, made just acidic by commercial hydrochloric acid, whereby all the dissolved and undissolved soaps get broken up, then 1 lb (0 5%) alum was added to the lye and thoroughly agitated for a long time, whereby all the free fatty acids settled down as alumina soap together with the albuminus and mucilegenus matters The lye was then filtered through filter press, made slightly alkaline and again filtered, when a clear white liquid (196 lbs) was obtained

(*b*) *Concentration*—The next operation consists in the concentration of the treated lye to crude glycerine There are various types of evaporators which are used for this purpose, but we adopted the following procedure with satisfactory results 196 pounds of the treated lye was concentrated in an open steam jacketted evaporator of about 40 gallons capacity, with a salt catching arrangement at the bottom After the lye had been concentrated for some time, the salts began to separate out, which was removed from time to time When the glycerine content comes to about 50 per cent the rate of evaporation became very slow The liquid, at this stage, was transferred to a steam jacketted still of 25 gallons capacity and fitted with a vacuum and salt catching arrangement The glycerine was concentrated to about 75 per cent strength in this still, the resulting glycerine (19 lbs.) (sp gr 1 298, refractive index 1·4705 at 30°C) being dark brown in colour and sweet in taste The salts separated (17 lbs) can be used again for 'cutting' the soaps in the soap kettles

(*c*) *Super-heated steam distillation*—This is the most difficult operation in the whole series of operations involved in the manu-



SUPER HEATED STEAM DISTILLATION OF GLYCERINE

1 Super heater

2 Distillation still with oil bath

3 Glycerine receiver

4 Condenser

5 Sweet water receiver

facture of glycerine There are various types of plants known to be used for this operation, but they are very costly and require large quantities of crude glycerine for running A suitable plant was, therefore, designed, constructed and operated for the distillation of glycerine with super-heated steam.

Description and working of the plant —A copper still of 14" diameter and 24" height was fitted (a) with a goose neck and (b) with an open steam coil fitted inside, the mouth of the goose neck having been provided with a baffle to protect any froth or entraint passing over The still is immersed to the neck in an oil-bath at a temperature between 180°–200°C The steam coming from the boiler was passed through a series of concentric tubes heated directly by burners which raised the temperature of the steam to about 180–190° before entering the still through the open steam coil, and the glycerine distilled over along with steam The first cooler is kept at about 90°C, so the glycerine is left behind in it and the steam passes through other coolers and condensers, and ultimately collects as sweet water, the whole system is kept at a vacuum of about 15 inches The resulting glycerine is dull yellow in colour and is of 67 per cent strength containing only water as impurity 18 5 lbs. of crude glycerine (75% strength) yielded 19 5 lbs of 67 per cent glycerine, sp gr, 1 22, refractive index 1 462 at 30°C , residue 1½ lbs , time required about 2 hours (about 10 lbs per hour)

(*d*) *Clarification and concentration* —The glycerine obtained in the previous operation was treated with animal charcoal in proportions of 1 part in 1000 parts of glycerine, in a vessel fitted with a mechanical stirrer arrangement, and kept at a temperature of 80°C After about 6 hours it is filtered, and concentrated in an evaporator under vacuum, whereby all the water is driven out from glycerine. 19 5 lbs of distilled glycerine of 67 per cent strength yielded 12 25 lbs of 98 50 per cent glycerine, sp gr, 1 26, refractive index, 1 467 at 30°C The 98 5 per cent odourless glycerine, thus obtained is known in the market as pale straw coloured dynamite glycerine

Bidistilled glycerine —For the production of bidistilled glycer-

ine, the 98 5 per cent pale straw coloured glycerine was once more distilled and concentrated under vacuum, this on analysis showed glycerine content, 99 6 per cent, sp gr 1 261, refractive index, 1 968 at 30°

Crude glycerine (75 4 %) of 40 lbs per batch from the Tata Oil Mills Co, Ltd, Tatapuram, on being subjected to the superheated steam distillation in our plant yielded 67 per cent glycerine (42 25 lbs), which on clarification with animal charcoal and concentration yielded glycerine (26 5 lbs) of 98 5 per cent strength

In the following table are given the percentages of glycerine at different stages, with their specific gravities, refractive indices and ash contents

No		Glycerine Content	Ash Content	Sp gr at 20°	Ref Index at 30°
1	Soap-lye	7 3871%		1·10	1 3695
2	Crude glycerine	75 4%		1 298	1 4705
3	Dilute pure glycerine (just after super-heated steam distillation)	67 0%		1 22	1 462
4	Pure pale straw colour glycerine	98·5%	0 150%	1 260	1·467
5	Chemically pure glycerine	99 6%	0 0210%	1 261	1 468
6	Standard chemically pure glycerine	99 5–100%		1 26	1 468
7	Standard pale straw colour glycerine	98 5%		1 26	1 465

Conclusion

Soap-lye containing about 6,375 tons of glycerine is liberated annually in India in its soap factories about 1000 in number Amongst the Indian soap factories there are many in which soap is manufactured in rather small quantities, and it is not possible for them to

purchase from abroad and install elaborate glycerine recovery plants The capacity of the semi-large scale plant (production capacity 10 lbs of 67% glycerine per hour) that has been used here for our operations can be used as such, and the units may be multiplied in number as required, and if necessary, the plants can be conveniently enlarged and the experimental conditions modified in such a way that the yield can be raised from 10 lbs to 20 lbs, without involving very much of technical intricacies in the operations and great engineering skill

Without claiming to be too accurate, it can be said, however, that the cost of making all the necessary equipments and appliances (for the production of about 70 lbs of pure glycerine per day) may not exceed Rs 1,000/- There is no doubt whatsoever that the cost of production of glycerine by such small scale plants will be much lower than the market price Another very important factor in favour of the small unit plants will be that any money spent in constructing them will be retained in India

Our thanks are due to the Mysore Government Soap Factory, Bangalore, for kindly supplying the soap-lye, and to the Tata Oil Mills Co, Ltd, Tatapuram, for kindly supplying the crude glycerine for this investigation

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