Catalysed Saponification of Oils/ Fats Using *o*-Cresol, *m*-Cresol, 1-Naphthanol, 2,5-Dimethylphenol and 2-Nitrophenol

[1]B. KHALID, [1]L. LIAQUAT, [1]J. I. KHAN, AND [2]C. M. ASHRAF*
[1]*Applied Chemistry Research Centre, PCSIR Labs Lahore, Pakistan.*
[2]*Chemistry Department,*
Forman Christian College (A Chartered University), Lahore, Pakistan.

(Received 20th July, 2006, revised 16th September, 2006)

Summary: An easy and swift method for soap manufacture through the saponification reaction of oils and fats, in the presence of different organic compounds (o-cresol, 1-naphthol, 2,5-dimethylphenol), which act as catalyst, has been studied under ambient conditions of temperature. *o*-Cresol, *m*-cresol, 1-naphthol and 2,5-dimethylphenol showed 50-70 % reduction of reaction time for saponification, while 2-nitrophenol did not show any catalytic activity.

Introduction

The manufacturing of soap goes back to 2300 years and is one of the oldest industry[1]. The saponification is usually carried out with sodium hydroxide, or potassium hydroxide depending upon the requirement of the product. Varying amounts of caustic alkalies are used to produce soft soap, soap for creams, shaving creams and other allied products.

Generally, the industry uses three processes; cold, semiboiled and fully boiled processes to carry out saponification in the soap manufacturing. Cold process, which is slow and incomplete, is usually employed for producing laundry soap [2]; the semiboiled and fully boiled processes are carried out at elevated temperatures, which consume a lot of energy. It is found that 42687 x 10^3 BTU of heat energy is required for the saponification of 100 Lbs of fat charge. The process usually takes about 8h or more [3] and is neither spontaneous nor rapid. There is a demand for the development or discovery of catalysts, which can facilitate the soap manufacture and effectively reduce the cost.

Formerly, phenols (0.4-1.8 %) like thymol, carvacrol, *p*-chlorophenol, 2,4-dichlorophenol and 4-chloro-3,5-dimethylphenol were primarily tried for the catalytic hydrolysis of oil/ fat [4-7]. In order to extend the scope of phenolic compounds as catalysts to facilitate the saponification reactions, *o*-cresol, *m*-cresol, 1-naphthol, 2,5-dimethylphenol and 2-nitrophenol (0.5–1.0 %), have now been used at ambient temperature and ordinary pressure. These catalyzed the saponification reaction and reduced its duration. This is likely to decrease the effective cost of soap manufacture. The results obtained through present studies, are being presented in this paper.

Results and Discussion

The saponification of cottonseed oil, coconut-oil and tallow, blended with cotton seed oil (1:1), was carried out with and without catalyst and unsaponifiable obtained at different time intervals, are recorded in Tables 1, 2 and 3, respectively. It is inferred from these results that by the addition of *o*-cresol (0.5 %), the saponification reaction of cotton seed oil was accelerated and almost completed (97.5 %) in 120 minutes, while the duration of this reaction was 480 minutes when no catalyst was used. Coconut oil was saponified (98 %) within 60 minutes in the presence of *o*-cresol, while a blend of tallow and cotton seed oil (1 : 1 w/w) saponified in 80 minutes, when *o*-cresol was used as a catalyst. Cotton seed oil and tallow blend took 200 minutes and 240 minutes respectively without the addition of catalyst. Almost similar was the case with *m*-cresol with a little change in time. Use of 1-naphthol as the catalyst with coconut oil saponified it (97 %) in 80 minutes, cotton seed oil (96 %) in 120 minutes and tallow blended with cotton seed oil (95.3 %) in 100 minutes. 2,5-Dimethylphenol also catalyzed the reaction up to 99.5 %, 96.0 % and 96.5 % saponification of coconut oil, cotton seed oil and tallow blend with cotton seed oil in 40 minutes, 100 minutes and 80 minutes respectively. 2-Nitrophenol was also tried but it did not show encouraging results.

The results so obtained indicated that the saponification time depends upon the nature of oil

*To whom all correspondence should be addressed.

Table-1: Saponification of Coconut Oil with and Without Catalysts

Catalysts Used (0.5%)	Oil % Saponified After Minutes								
	20	40	60	80	100	120	140	160	180
Coconut oil without catalyst	49.9	59.2	62.8	67.2	68.2	70.3	71.8	73.1	75
Coconut oil + *o-cresol*	94.0	98.6	99.5	---	---	---	---	---	---
Coconut oil + *m-cresol*	93.75	98.3	99.2	99.5	---	---	---	---	---
Coconut oil+1-*naphthol*	91.02	92.33	94.38	97.0	---	---	---	---	---
Coconut oil + 2,5-*dimethylphenol*	94.9	99.0	99.5	---	---	---	---	---	---
Coconut oil + 2-*nitrophenol*	50.0	58.5	62.8	67.21	68.0	70.0	70.9	72.8	74.7

Table-2: Saponification of Cotton Seed Oil with and Without Catalysts

Catalysts Used (0.5%)	Oil % Saponified After Minutes								
	20	40	60	80	100	120	140	160	180
Cotton Seed Oil without catalyst	42.2	46.7	49.7	52.42	54.5	55.61	56.2	58.2	60.0
Cotton Seed Oil + *o-cresol*	93.0	93.9	94.8	95.4	96.7	98.0	---	---	---
Cotton Seed Oil + *m-cresol*	92.67	93.7	94.7	97.3	98.2	98.23	---	---	---
Cotton Seed Oil + 1-*naphthol*	80.4	84.8	89.0	94.7	96.0	---	---	---	---
Cotton Seed Oil + 2,5-*dimethylphenol*	88.25	90.15	92.2	94.3	95.4	96.0	---	---	---
Cotton Seed Oil + 2-*nitrophenol*	---	---	---	---	---	---	---	---	---

Table-3: Saponification of Cotton Seed Oil–Tallow Blend (50 %) with and Without Catalysts

Catalysts Used (0.5%)	Oil % Saponified After Minutes								
	20	40	60	80	100	120	140	160	180
Blended oil without catalyst	59.65	60.3	61.2	62.7	64.2	65.9	67.0	69.1	70
Blended oil +*o-cresol*	93.4	95.41	96.75	97.5	---	---	---	---	---
Blended oil +*m-cresol*	93.8	96.2	97.3	97.4	---	---	---	---	---
Blended oil+*1-naphthol*	91.8	94.5	95.3	97.1	---	---	---	---	---
Blended oil + *2,5-dimethylphenol*	94.4	95.9	96.1	96.5	---	---	---	---	---
Blended oil + *2-nitrophenol*	---	---	---	---	---	---	---	---	---

and the catalyst. Coconut oil and tallow blended with cotton seed oil, were saponified in shorter period of time than cotton seed oil. Catalysts might form phenolates, which dissolve in the soap layer and accelerate the saponification reaction [6].

2-Nitrophenol was also studied but it did not show any catalytic activity. The reason may be that phenol bearing an electron-withdrawing group is more acidic and is likely to reduce the effective concentration of corresponding phenoxide ions. This has nothing to do with its steric hindrance as suggested in an earlier report [8]. This suggestion is based on the fact that compounds, which raise the temperature and promote oil emulsion within a short time proved to be effective catalysts. The saponification reaction generally takes place on the oil-water interface. Therefore, the substance capable of becoming part of interface will demonstrate greater catalytic activity and thus reduce the saponification time (3 to 7 times). In conventional cold process of soap making, the saponification reaction is not completed even after 70 hrs. Classical hot process requires 8 hours and consumes large amount of energy [4].

It may be concluded that phenolic compounds, like *o-cresol*, *m-cresol*, 1-*naphthol* and 2,5-*dimethylphenol*, used in the present study resulted in the enhancement of rate and extent of saponification reaction with the exception of 2-nitrophenol. This catalytic saponification may prove to be a simple and rapid technique for soap industry.

Experimental

Cottonseed oil (100 g) was taken in 250 mL capacity beaker to which *o*-cresol catalyst (0.5 g) was added with stirring, followed by the addition of sodium hydroxide solution (34 %). The reaction was carried out at room temperature (35 °C). Blank experiment without the use of catalyst was also carried out. The rate of saponification reaction was determined by withdrawing samples (5 g), from the reaction mixture at intervals of 20 minutes. The unsaponified oil was extracted with petroleum ether (B.P. 60-70 °C) and different extracts obtained at different intervals were dried over anhydrous sodium sulphate. The solvents were recovered by distillation and the materials obtained were dried in weighed beakers in an electric oven at 100 °C for 1hr and then weighed to obtain percentage of unsaponifiable oil.

Same procedure was repeated for coconut oil and a blend of tallow and cottonseed oil (1:1 w/w) using *m-cresol*, 2,5-*dimethylphenol*, 1-*naphthol*, 2-*nitrophenol* as catalysts. Results are shown in Tables 1, 2 and 3, respectively.

References

1. J. Davidsohn, E. J. Better and A. Davidsohn, Soap Manufacture, Interscience Publishers, Inc. New York, **1**, P. 1 (1953).
2. Daniel Swern, Bailey's Industrial Oil and Fat Products (Interscience Publishers, Inc. New York) 3rd Ed. P. 988 (1964).
3. Daniel Swern, Bailey's Industrial Oil and Fat Products (Interscience Publishers, Inc. New York) 3rd Ed. P. 980 (1964).
4. J. Davidsohn, Soap Manufacture (Interscience Publishers, Inc. New York), Vol. 1, P. 36 (1953).
5. B. A. Rashkovan and V. M Lebedeva. Zhur. Obshchex Khim, *J. Gen. Chem.*, **20**, 253 (1950).
6. R. Ahmad, A. Shafiq, K. M. Khan and T. M. Qureshi, *Pak. J. Sci. and Ind. Res.*, **31**(9), 671 1988.
7. R. Ahmad, M. Tariq, M. T. Qureshi, G. R. Khan, M. S. Sethi, Proc. 2nd All Pak. Sci. Conf. Atchison College, Lahore ISSN 1013-5316; CODED:SINTE 5.(1993).
8. F. E. London Herbert Meiseich, Introduction to Organic Industry, Holt Rinchart & Winston, Inc., New York (P 616) (1961).