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SAPONIFICATION CATALYSTS

Part-III. Catalytic activity of *O*-hydroxybenzaldehyde and 4-allyl-2-methoxy-phenol for the saponification of oils and fats

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A simple economical and quick method for the saponification of oils and fats under mild conditions, using O-hydroxybenzaldehyde and 4-allyl-2-methoxy phenol as catalyst has been investigated.

Key words: Saponification catalysts, Soaps, Hydroxybenzaldehyde, Allyl-methoxy phenol.

Introduction

In continuation of the previous work [1-2] some more organic compounds have been investigated which accomplish the saponification of oils and fats in short time under mild conditions and thus present a simple, cheaper and economical method of soap making. This can contribute certainly in lowering the production cost and make the soaps available to the consumer at competitive prices.

Experimental

Saponifications were carried out at room temperature (32°C). Coconut and cotton seed oils (100 ml each) were taken separately in two beakers (250 ml) and temperature was recorded. To each oil the catalyst (0.4/0.75 ml) was added and dispersed thoroughly. 34% Sodium hydroxide solution (50 ml) was added to the oil catalyst mixtures and stirred well.

For blank determination, 34% sodium hydroxide solution (50ml) was added to 100 ml of the oil in another beaker (250 ml) and agitated. The contents of the beakers were stirred from time to time. It was observed that the mixture which contained catalyst, thickened within ten minutes alongwith rise in temperature but no thickening was observed in the sample without catalyst.

Nearly 4-5g of the sample from reaction mixture were taken in a beaker, after intervals of every ten and twenty minutes from the beginning of the saponification reaction in order to determine the non saponified matter. Rise or fall in temperature was also recorded on each determination. The nonsaponified oil was extracted with *n*-hexane (B.P. 60-70°C), dried over anhydrous sodium sulphate and its percentage was determined as usual after recovering the dried solvent.

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Results and Discussion

Saponification of each oil i.e. coconut oil and cotton-seed oil by sodium hydroxide solution (34 % w/v) with or without the addition of catalysts was conducted at room temperature and the kinetics of these chemical reactions was studied and recorded as given in the Tables 1-4.

In the present studies catalytic activity of organic compounds particularly those with hydroxy groups directly attached with benzene was studied. It has been recorded that *o*-hydroxybenzaldehyde and 4-allyl-2-methoxyphenol act as a saponification catalysts.

The catalytic saponification of cotton seed oil and coconut oil, as examplified in the present studies suggested that the process was accelerated and completed in a much shorter time (60 min.) than usual (480 min.).

From the results, it was observed that by using *o*-hydroxybenzaldehyde (0.75% of the oil) as saponification catalyst 98.80% cottonseed oil was saponified in 140 min. and 97.56% coconut oil in 100 min. With 4-allyl-2-methoxy-phenol (0.6% of the oil) as catalyst 97.97% cottonseed oil was saponified in 140 min. and 95.52% coconut oil was saponified in 100 min. Without using the catalysts only 52.90%

TABLE 1. THE EXTENT OF SAPONIFICATION OF COCONUT OIL WITH AND WITHOUT CATALYST AT AMBIENT TEMPERATURE.

Oil/Catalyst used	% Of oil reacted at different time intervals (minutes)								
	20	40	60	80	100	120	140	160	180
Coconut oil alone	46.50	48.75	51.95	53.33	55.90	56.27	60.54	64.50	68.95
Coconut oil+ 0.75 & *O*-hydroxy benzaldehyde	71.05	79.76	83.64	89.96	97.56				
Coconut oil + 0.6 % 4 allyl-2-methoxy phenol	70.21	77.99	85.88	91.96	95.52				

cottonseed oil was saponified in 180 min. and 68.95% coconut oil was saponified in 180 min. These results showed that *o*-hydroxybenzaldehyde was more active than 4-allyl-2-methoxy-phenol as saponification catalyst.

Another interesting observation made from these studies was that oils containing saturated fatty acids were saponified rapidly in comparison to those containing unsaturated fatty acids. For example, coconut oil (containing higher amount of lauric acid) gave 97.56% saponification while cottonseed oil (with lesser saturated contents) provided 97.97% saponification in 100 and 140 min. respectivley.

It was inferred that saponification time depended upon the nature of both oil and the catalyst. Coconut oil was saponified in a shorter time than cottonseed oil.

Generally saponification is a slow process and is accomplished when fats react with alkalies at raised temperature (100°C) with specific concentration of lye (34%) and continuous agitation. It is possible that the catalysts act as stabilizers in the water-in-oil system and shorten or completely eliminate the first slow step of saponification [3]. Additionally, formation of phenolates which dissolve in the soap layer might accelerate the saponification [4]. A plausible representation of this idea is given below.

TABLE 2. THE EXTENT OF SAPONIFICATION OF COTTONSEED OIL WITH AND WITHOUT CATALYST AT AMBIENT TEMPERATURE.

Oil/Catalyst used	% Of oil reacted at different time intervals (minutes)								
	20	40	60	80	100	120	140	160	180
Cotton seed oil alone	35.52	38.42	46.27	50.40	51.30	51.65	51.76	52.01	52.90
Cotton seed oil + 0.75% *O*-hydroxy benzaldehyde	61.80	71.50	81.20	83.20	85.68	87.90	98.80		
Cottonseed oil +4-allyl -2-methoxy phenol	71.74	79.57	81.52	85.68	89.57	91.75	97.97		

TABLE 3. INCREASE OF TEMPERATURE DURING THE CATALYTIC SAPONIFICATION OF COTTONSEED OIL.

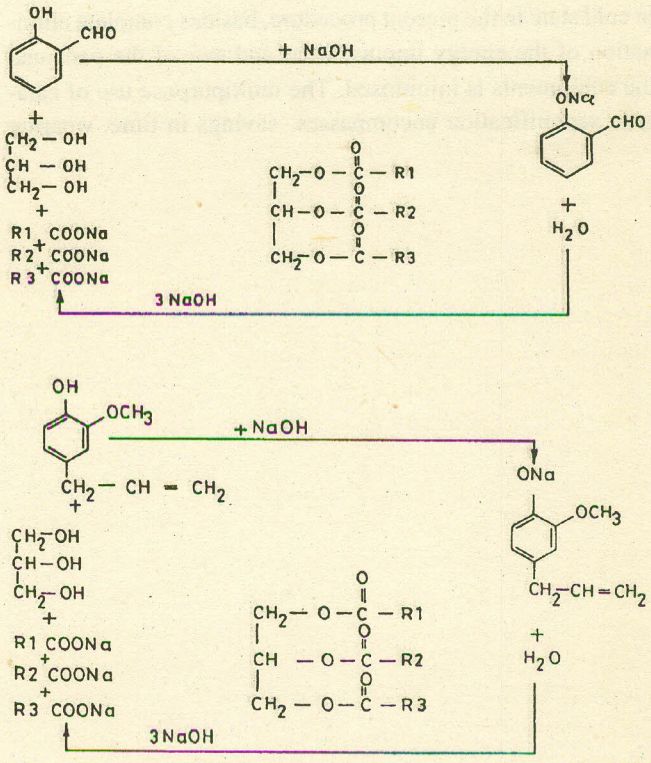
+ 0.75 % O-hydroxy Benzaldehyde			+0.6% 4-allyl-2-methoxy-phenol	
	Time (min.)	Temp.°C	Time (min.)	Temp.°C
After	0	30.0	After 0	30.0
	10	33.0	10	32.0
	20	33.5	20	33.5
	30	35.0	30	35.0
	40	35.5	40	36.5
	50	36.0	50	37.0
	60	36.5	60	36.0
	70	36.0	70	35.5
	80	35.0	80	34.0
	90	34.5	90	33.5
	100	33.0	100	32.0

Temperature of cotton seed oil before adding catalyst = 30°C.

TABLE 4. INCREASE OF TEMPERATURE DURING THE CATALYTIC SAPONIFICATION OF COCONUT OIL IS SHOWN BELOW.

+ 0.75 % O-hydroxy Benzaldehyde			+0.6%4-allyl-2-methoxy-phenol	
	Time (min.)	Temp.° C	Time (min.)	Temp.°C
After	0	30	After 0	30.0
	10	34	10	33.0
	20	39	20	38.0
	30	42	30	41.5
	40	45	40	46.0
	50	49	50	49.0
	60	51	60	49.0
	70	51	70	46.0
	80	46	80	42.5
	90	41	90	37.0
	100	36	100	35.5

Temperature of coconut seed oil before adding catalyst = 30° C.



It is observed that *o*-hydroxybenzaldehyde, 4-allyl-2-methoxy-phenol proved to be excellent saponification catalyst. Many other phenolic substances such as hydroquinone, indophenol, 4-aminophenol, nitroso-naphthol *m*-acetoxyphenol, *O*-amino-phenol, resorcinol, catachol, dinitro phenol, when studied did not show any catalytic activity. As these compounds did not thicken the emulsion within 10 min. of admixture, no phenolate like formation was perhaps possible with these compounds. This argument is based on the fact

that the compounds which raise the temperature and thicken the caustic solution and oil emulsion within a short time (10 min.) show greater catalysis.

Industrially the manufacturing of soaps is carried out by different methods, i.e. (i) cold process (ii) semi boiled and (iii) full boiled processes. The cold process which is carried out at normal temperature is used for the production of inferior quality soaps and does not result in complete saponification [5]. Semi and full boiled processes which are carried out at elevated temperatures are time taking and consume a substantial amount of heat energy, thus affecting the wearing life of soap pans and other processing equipmrent. The economics of soap manufacturing not only requires the availability of raw materials at cheaper rates, but also saving of time, wearing strength, labour, fuel consumption and other factors contributing to the production cost of the soaps. In the recent years the production costs have shown an upward trend, consequently the prices of the soaps have gone up.

As the saponification of oils and fats is carried out in cold state in the present procedure, besides complete elimination of the energy imputs, wear and tear of the pans and the equipments is minimised. The multipurpose use of catalytic saponification encompasses savings in time, wearing strength, labour and fuel economy. All these factors contribute substantially in lowering the production cost of the soap thus providing incentive both to the industry and the consumers alike. Moreover, catalysts studied are easily available organic compounds. Catalytic saponification is thus a simple, cheaper and economical method for soap making. If applied on a commercial scale, it expected to provide socio-economic dividends.

References

1. Rafi Ahmad, Khalid Mahmood, Tufail M. Qureshi and S. A. Khan, Pak. j. sci. ind. res., **31** (9), 671 (1988).
2. Rafi Ahmad, Muhammad Tariq, Muhammad Tufail Qureshi, Ghulam Rabbani Kahn and M. Shahid Sethi, Proc. 2nd All Pakistan Sci. Conf., Dec. 26-30 (1983), pp.116-117.
3. J. Davidson, *Soap Manufacture* (Interscience Publishers Inc. New York, 1953), Vol.1, pp.36.
4. B. A. Rashkogvan, and V. M. Lebedeva, Chemical Abstract, **44,** 5619 (1950).
5. Daniel Swern, *Bailey's Industrial Oil and Fat Products* (Interscience Publishers Inc., New York, 1964), pp.988.