Sebacic Acid and 2-Octanol from Castor Oil[1]

A.K. Vasishtha*, R.K. Trivedi and G. Das
Harcourt Butler Technological Institute, Kanpur 208 002, India

Alkali pyrolysis of castor oil was carried out in the presence of white mineral oil. The operating parameters were optimized to obtain high yields of 2-octanol and sebacic acid. Alkali pyrolysis at 280 ± 2°C in the presence of 1% red lead catalyst yielded 70.1% 2-octanol and 72.5% sebacic acid on the basis of their respective theoretical yields.

Castor oil is unique in having three reactive functional groups, i.e., double bond, hydroxyl group and ester linkage. Pyrolysis of castor oil in the presence of alkali yields 2-octanol and sebacic acid. These oleochemicals are precursors for industrially important plasticizers, surface coatings and perfumery chemicals (1–3).

The normally expected theoretical yields of 2-octanol and sebacic acid from alkali pyrolysis of castor oil containing 84% ricinoleic acid are 35.7 and 43.6%, respectively. The yields of these oleochemicals in earlier attempts, reported as percent of theoretical yields from castor oil and methyl ricinoleate, have been low, and far from satisfactory. Adams and Marvel (4) obtained 2-octanol in 23–41% of the theoretical yield by alkali fusion of sodium soap of castor oil fatty acids. Verma and Agarwal (5) reported 53% of the theoretical yield of sebacic acid by alkali fusion of castor oil with 30% sodium hydroxide in 8 hrs at a temperature of 260°C and pressure of 49.3 kg/cm^2. Grimberg (6) obtained basic yields of 32–40% of sebacic acid and 18–25% of 2-octanol by alkali pyrolysis of castor oil in presence of superheated steam and cadmium sulfate as catalyst. Diamond *et al.* (7) obtained 70% of the theoretical yield of sebacic acid by heating methyl ricinoleate at 250°C in the presence of 48% sodium hydroxide and 5% red lead as catalyst. Nanavati *et al.* (8) reported 63.19% of the theoretical yields of sebacic acid and 57.14% of the same of 2-octanol by alkali fusion of castor oil in the presence of white mineral oil.

This research work was undertaken with a view to develop a simple process for alkali pyrolysis of castor oil while improving the yields of the desired oleochemicals.

EXPERIMENTAL

Materials. Raw grade castor oil was obtained from the local market. L.R. grade cadmium oxide, calcium oxide, aluminium oxide and red lead, all used as pyrolytic catalysts, were obtained from Lab-Chem. Industry (Bombay, India). L.R. grade sodium hydroxide used for alkali pyrolysis was obtained from Sarabhai M. Chemicals (Vadodara, India). White mineral oil (bp 400 ± 20°C), used as diluent in the alkali pyrolysis, was obtained from Techno Pharma Pvt. Ltd. (Kanpur, India). It was purified by heating to 380°C by distilling off its low boiling fraction.

Castor oil (150 g), pre-saponified with sodium hydroxide (20 g), was used in some of the pyrolysis experiments of this study.

Characteristics of castor oil. The physicochemical characteristics of castor oil were determined by the standard AOCS procedures (9).

The fatty acid composition of the castor oil was determined by gas-liquid chromatography of the methyl esters prepared according to the method of Jameison and Reid (10) on Model CIC (Chromatography & Instruments Company, Baroda, India) having a flame ionization detector. The chromotography was done on a 3.2 mm × 2 m stainless steel column, packed with 15% DEGS supported on Chromosorb W 60/80, isothermally at 190°C using nitrogen as carrier gas at a flow rate of 30 ml/min. The fatty acid composition was calculated on the computer Model BBC Acron DFS (Basic) attached to the gas chromatograph.

Pyrolysis of raw and saponified castor oil. A mild steel cylindrical reactor (38.1 cm H × 10.2 cm D) equipped with arrangements for stirring, electrical heating, temperature control, feed inlet and outlet and arrangements for condensation of emanated products was used for the pyrolysis reaction.

White mineral oil in 100–450 g quantities was taken in the reactor, to which 50% aqueous solution of 60–160 g sodium hydroxide was slowly added while heating and stirring the whole mass to obtain uniform dispersion of sodium hydroxide in mineral oil. After the removal of water, the temperature of white mineral oil and alkali mixture was raised to 240–320°C for the pyrolysis reaction. Thereafter, raw castor oil in 150 g quantities with or without 0.5–5.0% (w/w) catalyst was slowly added to the reactor over a period of 1 hr and the pyrolysis was further continued for 2–8 hrs.

The volatile products of pyrolysis along with water vapors emanating from the reactor were condensed and collected. After separating 2-octanol, the condensate was returned to the reactor through the feed system. The traces of moisture from 2-octanol were removed by drying over anhydrous sodium sulfate.

The pyrolyzed residue from the reactor was diluted with 2 l of hot water in a glass container and acidified to pH 6.0 with dilute hydrochloric acid. An oily layer of white mineral oil floating at the top was separated from the aqueous layer. The latter was further acidified to pH 2.0 and cooled to 20°C. The sebacic acid was recovered from it by filtration as a white fluffy mass, which was then dried.

Some sets of experiments were also conducted using pre-saponified castor oil instead of raw castor oil as a starting material.

The oleochemicals were characterized and assessed for purity by spectroscopic methods. The identity of 2-octanol as an alcohol was confirmed by ceric ammonium nitrate and sodium metal tests (11), and its purity was further assessed by ester value and bisulfite method (12).

The purity of sebacic acid was assessed by its neutralization number (12) and melting point (11).

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*To whom correspondence should be addressed.

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The identity of the aforementioned oleochemicals was further confirmed by IR and NMR spectroscopy of the purified samples.

RESULTS AND DISCUSSION

The physicochemical characteristics and fatty acid composition of castor oil used in this study are given in Table 1. It is apparent from its normal values that it was a genuine sample.

Pyrolysis of raw castor oil. The effect of the proportion of white mineral oil, used as diluent, on the yields of sebacic acid and 2-octanol in alkali pyrolysis of castor oil is shown in Table 2. The yields of oleochemicals from 150 g castor oil with 80 g sodium hydroxide increased with the increase in quantity of the diluent up to an alkali/diluent ratio of 8:30. This may have been due to better dispersion of alkali in the diluent, which should result in a better contact between the reactants, i.e., oil and alkali. A further increase in the quantity of the diluent did not improve the yields of the products. These results suggested an optimum alkali/diluent ratio to lie somewhere between 8:25 and 8:30, and oil/diluent ratio between 3:5 to 3:6.

TABLE 1

Physicochemical Characteristics and Fatty Acid Composition of Castor Oil[a]

Physicochemical characteristics:	
Specific gravity (at 30°C)	0.959
Color, Lovibond (Y+5R in 2.54 cm cell)	15.0
Refractive index (at 30°C)	1.4660
Optical rotation (in 10 cm tube)	+4°15′
Acid value	5.7
Hydroxyl value	154.6
Iodine value (Wijs)	85.9
Saponification value	179.4
Unsaponifiable matter, %	0.7
Fatty acid composition: (% wt)	
Palmitic acid	1.7
Stearic acid	2.4
Arachidic acid	0.3
Oleic acid	4.2
Ricinoleic acid	83.8
Linoleic acid	6.7
Linolenic acid	0.9

[a] Average of two determinations.

The results presented in Table 3 show the effect of the proportion of sodium hydroxide to the raw oil on the yields of desired oleochemicals during pyrolysis in presence of 400 g white mineral oil diluent. The yields of sebacic acid and 2-octanol from 150 g castor oil increased by increasing the proportions of sodium hydroxide up to 120 g (sodium hydroxide/castor oil ratio of 12:15), beyond which the yields were little improved. Therefore, the ratio of sodium hydroxide to castor oil at 12:15 (which provided more or less the maximum yields of the products) was considered to be the optimum. At this optimum proportion of sodium hydroxide, the ratio of alkali to diluent worked out to be roughly 8:27.

The effect of temperature of alkali pyrolysis of raw castor oil with optimized proportion of sodium hydroxide in diluent on yields of the oleochemicals is illustrated in Table 4. The yields of sebacic acid and 2-octanol increased with an increase in reaction temperature up to 300°C, at which the yields were 64.2% and 65.2%, respectively. At 320°C, the yields of the oleochemicals were low, perhaps because of polymerization of the oil and formation of more side-reaction products.

Table 5 shows the effect of reaction time at 300°C ± 2°C on the yields of the oleochemicals during alkali pyrolysis of castor oil using alkali/oil ratio of 12:15 and alkali/diluent ratio of 8:27. The yields of sebacic acid and 2-octanol increased with an increase in reaction time up to 5 hrs. A further increase in reaction time resulted in little improvement in the yields of 2-octanol, but marked decreased the yield of sebacic acid, presumably because of its decomposition. Therefore, the reaction time of 5 hrs at 300°C ± 2°C was taken as the optimum.

Certain basic adjuncts claimed earlier by other researchers (4,5,13,14) to enhance the yields of sebacic acid and 2-octanol in alkali pyrolysis of castor oil were tried with the aforementioned optimized conditions of the reaction. The results presented in Table 6 show the adverse effect of calcium oxide and aluminium oxide on the yields of sebacic acid and 2-octanol. Sodium

TABLE 2

Effect of Proportion of Diluent[a] **on Yields of Oleochemicals**[b] **During Alkali Pyrolysis of Raw Castor Oil**[c,d]

White mineral oil (diluent) (g)	Oil/diluent ratio	Alkali/diluent ratio	Experimental yields (% of the theoretical yield)	
			2-Octanol	Sebacic acid
100	3:2	8:10	21.7	15.7
150	3:3	8:15	24.0	17.7
200	3:4	8:20	24.8	20.7
250	3:5	8:25	29.2	23.0
300	3:6	8:30	30.4	23.4
350	3:7	8:35	30.2	23.3
400	3:8	8:40	30.2	23.3
450	3:9	8:45	30.2	23.3

[a] White mineral oil (bp 400 ± 20°C)

[b] Average of two runs at 260 ± 2°C for 5 hrs.

[c] Quantity of oil taken for pyrolysis was 150 g.

[d] Quantity of sodium hydroxide added to the diluent in the reactor was 80 g.

TABLE 3

Effect of Proportion of Sodium Hydroxide in Diluent[a] on Yields of Oleochemicals[b] During Alkali Pyrolysis of Raw Castor Oil[c]

Sodium hydroxide in the diluent (g)	Alkali/oil ratio	Experimental yields (% of the theoretical yield)	
		2-Octanol	Sebacic acid
60	6:15	16.0	15.1
80	8:15	30.2	23.1
100	10:15	45.8	34.9
120	12:15	56.4	56.9
140	14:15	56.3	57.0
160	16:15	56.7	56.9

[a] 400 g white mineral oil (bp 400 ± 20°C) was added as diluent in the reactor.
[b] Average of two runs at 260 ± 2°C for 5 hrs.
[c] Quantity of oil taken for pyrolysis was 150 g.

TABLE 4

Effect of Temperature on Yields of Oleochemicals[a] During Alkali Pyrolysis of Raw Castor Oil[b,c]

Temperature[d] (°C)	Experimental yields (% of the theoretical yield)	
	2-Octanol	Sebacic acid
240	28.2	20.0
260	50.1	53.5
280	62.2	58.2
300	65.1	64.2
320	54.8	60.6

[a] Average of two runs after 5 hrs reaction.
[b] Quantity of oil taken for pyrolysis was 150 g.
[c] 120 g sodium hydroxide dispersed in 400 g white mineral oil (bp 400 ± 20°C) was first added to the reactor.
[d] Reaction temperature was maintained within ± 2°C.

TABLE 5

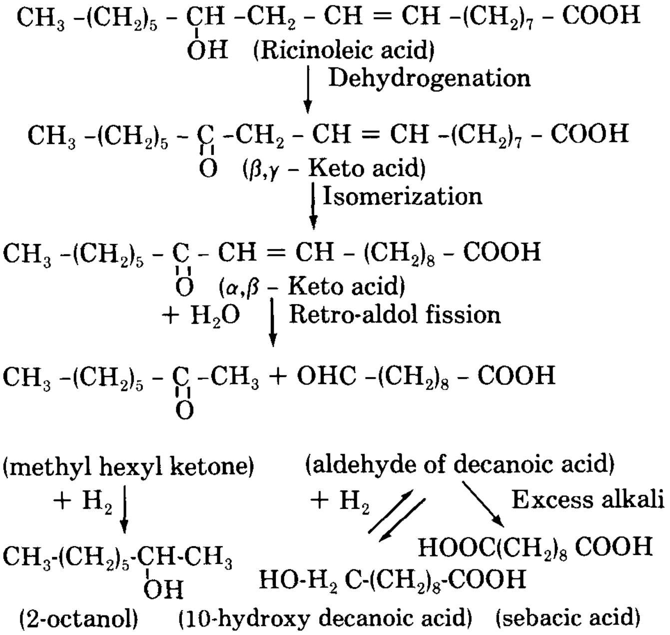
Effect of Reaction Time on Yields of Oleochemicals[a] During Alkali Pyrolysis of Raw Castor Oil[b,c]

Reaction time (h)	Experimental yields (% of the theoretical yield)	
	2-Octanol	Sebacic acid
2	25.6	30.1
3	48.5	47.9
4	61.3	60.2
5	65.2	64.2
6	65.9	64.1
7	66.6	61.8
8	66.8	52.2

[a] Average of two runs at 300 ± 2°C.
[b] Quantity of oil taken for pyrolysis was 150 g.
[c] 120 g sodium hydroxide dispersed in 400 g white mineral oil (bp 400 ± 20°C) was first added to the reactor.

carbonate and cadmium oxide were also ineffective for the purpose. Red lead, Pb_3O_4 improved the yields of the oleochemicals to some extent; the optimum level of use being 1% red lead by weight in the oil. The role of adjuncts in enhancing the yields of the desired products is, however, not very explicit in the literature.

However, Dynthan and Weedon (15) have given the following mechanism for the formation of sebacic acid and 2-octanol by the pyrolysis of ricinoleic acid in presence of alkali:



$$CH_3\text{-}(CH_2)_5\text{-}\underset{OH}{CH}\text{-}CH_2\text{-}CH=CH\text{-}(CH_2)_7\text{-}COOH \quad \text{(Ricinoleic acid)}$$

$$\downarrow \text{Dehydrogenation}$$

$$CH_3\text{-}(CH_2)_5\text{-}\underset{O}{\overset{}{C}}\text{-}CH_2\text{-}CH=CH\text{-}(CH_2)_7\text{-}COOH \quad (\beta,\gamma\text{ - Keto acid})$$

$$\downarrow \text{Isomerization}$$

$$CH_3\text{-}(CH_2)_5\text{-}\underset{O}{C}\text{-}CH=CH\text{-}(CH_2)_8\text{-}COOH \quad (\alpha,\beta\text{ - Keto acid})$$

$$+ H_2O \downarrow \text{Retro-aldol fission}$$

$$CH_3\text{-}(CH_2)_5\text{-}\underset{O}{C}\text{-}CH_3 + OHC\text{-}(CH_2)_8\text{-}COOH$$

$$\text{(methyl hexyl ketone)} \qquad \text{(aldehyde of decanoic acid)}$$

$$+H_2 \downarrow \qquad + H_2 \swarrow\!\!\nearrow \qquad \searrow \text{Excess alkali}$$

$$CH_3\text{-}(CH_2)_5\text{-}\underset{OH}{CH}\text{-}CH_3 \qquad HO\text{-}H_2C\text{-}(CH_2)_8\text{-}COOH \qquad HOOC(CH_2)_8COOH$$

$$\text{(2-octanol)} \qquad \text{(10-hydroxy decanoic acid)} \qquad \text{(sebacic acid)}$$

The above mechanism shows the dehydrogenation of ricinoleic acid as the first step of pyrolysis in presence of alkali resulting in the formation of β,γ-keto acid which isomerizes to α,β-keto acid in the presence of alkali. This keto acid undergoes a retroaldol fission to yield methyl hexyl ketone and the aldehyde of decanoic acid in the presence of water. The methyl hexyl ketone takes hydrogen from the first step of dehydrogenation to form 2-octanol. On the other hand, two possible reactions may take place with the aldehyde of decanoic acid. One reaction is a reversible reaction of hydrogenation (the hydrogen being made available from the first step of dehydrogenation) to form 10-hydroxy decanoic acid, while the other reaction is the oxidation of aldehyde of decanoic acid to yield sebacic acid in the presence of alkali. This is where Pb_3O_4 may play an important role in increasing the yield of sebacic acid by oxidation of aldehyde of decanoic acid, thereby making more hydrogen available for the hydrogenation of methyl hexyl ketone to yield 2-octanol. The oxygen donating effect of Pb_3O_4 to yield larger quantities of sebacic acid suppresses the other forward reaction of hydrogenation of the aldehyde of decanoic acid to form 10-hydroxy decanoic acid, thus making more hydrogen available as required for the formation of 2-octanol. Since only one mole of hydrogen is available *in situ* after dehydrogenation of ricinoleic acid, and the same is likely to get distributed for the subsequent hydrogenation of methyl hexyl ketone and aldehyde of decanoic acid, the presence of Pb_3O_4 favors the oxidation reaction of aldehyde of decanoic acid. This suppresses the hydrogenation of the aldehyde and diverts more hydrogen required for the formation of 2-octanol. Thus, the presence of Pb_3O_4 helps in improving the yield of the desired reaction for both products.

Pyrolysis of pre-saponified castor oil. Table 7 shows

TABLE 6

Effect of Basic Adjuncts on Yields of Oleochemicals[a] During Alkali Pyrolysis of Raw Castor Oil[b,c]

Adjunct	Proportion of adjunct in oil (%, w/w)	Experimental yields (% of the theoretical yield) 2-Octanol	Sebacic acid
Calcium oxide	1.0	60.8	55.6
Sodium carbonate	1.0	64.6	64.1
Aluminium oxide	1.0	62.6	59.1
Cadmium oxide	1.0	65.0	64.2
	3.0	65.1	64.0
Red lead	0.5	65.7	66.1
	1.0	68.0	67.2
	3.0	68.0	67.3
	5.0	67.9	67.4

[a] Average of two runs at 300 ± 2°C for 5 hrs.
[b] Quantity of oil taken for the pyrolysis was 150 g.
[c] 120 g sodium hydroxide dispersed in 400 g white mineral oil (bp 400 ± 2°C) was first added to the reactor.

TABLE 7

Effect of Proportion of Free Sodium Hydroxide in Diluent[a] on Yields of Oleochemicals[b] During Alkali Pyrolysis of Pre-Saponified Castor Oil[c]

Free sodium hydroxide in diluent (g)	Experimental yields (% of the theoretical yield) 2-Octanol	Sebacic acid
40	46.8	32.0
60	52.4	46.3
80	59.1	58.7
100	67.9	66.1
120	68.1	66.1

[a] 400 g white mineral oil (bp 400 ± 20°C) was added as diluent in the reactor.
[b] Average of two runs at 300 ± 2°C for 5 hrs.
[c] 150 g castor oil pre-saponified with 20 g sodium hydroxide and diluted with water to 450 g was taken for pyrolysis.

TABLE 8

Effect of Temperature on Yields of Oleochemicals[a] During Alkali Pyrolysis of Pre-Saponified Castor Oil[b,c]

Temperature[d] (°C)	Experimental yields (% of the theoretical yield) 2-Octanol	Sebacic acid
240	43.0	31.6
260	56.4	56.9
280	69.9	68.7
300	67.9	66.1
320	60.0	59.2

[a] Average of two runs after 5 hrs of reaction.
[b] 150 g castor oil pre-saponified with 20 g sodium hydroxide and diluted with water to 450 g was taken for pyrolysis.
[c] 100 g sodium hydroxide dispersed in 400 g white mineral oil (bp 400 ± 20°C) was first added to the reactor.
[d] Reaction temperature was maintained within ± 2°C.

TABLE 9

Effect of Reaction Time on Yields of Oleochemicals[a] During Alkali Pyrolysis of Pre-Saponified Castor Oil[b,c]

Reaction time (hr)	Experimental yields (% of the theoretical yield) 2-Octanol	Sebacic acid
3	47.9	52.1
4	59.8	67.4
5	69.9	68.7
6	69.9	61.9

[a] Average of two runs at 280 ± 2°C.
[b] 150 g castor oil pre-saponified with 20 g sodium hydroxide and diluted with water to 450 g was taken for pyrolysis.
[c] 100 g sodium hydroxide dispersed in 400 g white mineral oil (bp 400 ± 20°C) was first added to the reactor.

TABLE 10

Effect of Basic Adjuncts on Yields of Oleochemicals[a] During Alkali Pyroysis of Pre-Saponified Castor Oil[b,c]

Adjunct	Prooportion of adjunct in oil (% w/w)	Experimental yields (% of the theoretical yield) 2-Octanol	Sebacic acid
Cadmium oxide	1.0	68.3	69.0
Calcium oxide	1.0	60.4	63.1
Aluminium oxide	1.0	66.8	67.8
Sodium carbonate	1.0	68.5	66.4
Red lead	0.5	69.4	68.8
	1.0	72.5	70.1
	3.0	72.4	70.1
	5.0	72.6	70.1

[a] Average of two runs at 280 ± 2°C for 5 hrs.
[b] 150 g castor oil pre-saponified with 20 g sodium hydroxide and diluted with water to 450 g was taken for pyrolysis.
[c] 100 g sodium hydroxide dispersed in 400 g white mineral oil (bp 400 ± 20°C) was first added to the reactor.

the results of experiments carried out to optimize the proportion of free sodium hydroxide in diluent during the alkali pyrolysis of pre-saponified castor oil. Considering the total quantities of sodium hydroxide used for pre-saponifying the castor oil and that taken in the diluent, the optimum proportion of alkali to oil was again found to be the same as 12:15.

The effect of temperature of pyrolysis of pre-saponified castor oil is shown in Table 8. The results indicate near maximum yields of the oleochemicals at 280°C. Therefore, the above temperature was taken to be the optimum for pyrolysis of pre-saponified castor oil.

Table 9 shows the effect of reaction time on the yields of the oleochemicals during pyrolysis of pre-saponified castor oil under the optimized reaction parameters. It was observed that the maximum yields of the oleochemicals were obtained in 5 hrs which was, therefore, taken as the optimum reaction time.

Table 10 shows the effect of different basic adjuncts used for the pyrolysis of pre-saponified castor oil under the optimized reaction conditions. Among all the basic adjuncts used, none, except the red lead Pb_3O_4, was found to be effective. Red lead at the level of 1.0% by weight of oil gave the maximum yields of the oleochemicals which was, therefore, the optimum level.

Thus, the optimized reaction parameters for alkali pyrolysis of raw and pre-saponified castor oil differed only in respect of temperature, which was 300°C for the raw oil and 280°C for the pre-saponified oil. Besides the temperature, the use of presaponified castor oil as a substrate in the pyrolytic reaction resulted in better yields of the oleochemicals and avoided the problem of excessive foaming in the reactor, which was encountered during pyrolysis of raw castor oil.

The purity of 2-octanol assessed by ester value and bisulfite method was found to be 90.0% and 96.0%, respectively. The presence of ketonic compounds, e.g., methyl hexyl ketone (16), in 2-octanol fraction obtained from alkali pyrolysis was confirmed by 2:4 dinitrophenyl hydrazine and semi-carbazide methods. The ketone content was found to be 10.0% by hydroxylamine hydrochloride method. The high purity of 2-octanol indicated by the bisulfite method was perhaps a consequence of the presence of ketones (which showed up in the analysis) in the product.

The purity of sebacic acid assessed by its neutralization number was 97.3%. The high purity of this oleochemical was confirmed by its melting point (133°C) and by gas-liquid chromatography of its methyl esters which indicated 99% purity of the compound.

The respective IR and NMR spectra confirmed the identity of the products as 2-octanol and sebacic acid.

The results of this study suggest that alkali pyrolysis of pre-saponified castor oil, containing 1% (w/w) red lead in a cylindrical reactor (38.1 cm H × 10.2 cm D) at 280 ± 2°C for 5 hrs using an overall ratio of alkali to diluent (white mineral oil) of about 8:27, yields as much as 72.5% 2-octanol and 70.1% sebacic acid (on the basis of theoretical yields) as of average 96% and 99% purity, respectively.

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