LIGHT COMPONENT EVAPORATION FROM DI-IZO-OCTYL-PHTHALATE IN FILM EVAPORATOR

By

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The di-izo-octyl-phthalate is produced in large quantities by esterification of phthalic acid anhydride with 2-ethyl-hexanol as PVC plasticizer. In the esterification reaction alcohol is used in an excess of 5 to 20 per cent, to be removed after reaction, together with residual water after washing "raw ester".

According to the present technology the alcohol and water – further on light component — evaporation takes place in a 10 to 16 m^3 steam heated vessel at 400 Torr pressure and from 150 to 160 °C temperature by batch process. Under these conditions the material treated is often discoloured, perhaps due to the thermal decomposition of ester, so the product does not meet quality requirements (colour, electrical property, evaporation loss, etc.).

Film devices are often used — because of their known advantages — when a small portion of the feed has to be removed and the pureness of the product is very important [1, 2].

Accordingly it seemed proper to examine circumstances to facilitate the light component evaporation from di-izo-octyl-phthalate in film evaporator.

Research was done in a cylindrical roller type film equipment designed and built in our department.

The technical data of film equipment:

length:	570 mm
diameter:	80 mm
surface:	0.14 m^2
heated surface:	0.13 m^2
speed of rotor:	700 rpm.

Description of the equipment

The scheme of the research equipment is seen in Fig. 1. The raw ester was fed from a stainless steel vessel (1) by means of a peristaltic pump (2) via preheater (3) to the film evaporator (4). The vapours condensed in a water

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cooled condenser (5), the condensate was gathered in a picking vessel. The film evaporator was heated by hot oil fed by an electrothermal unit in its three separate sections.

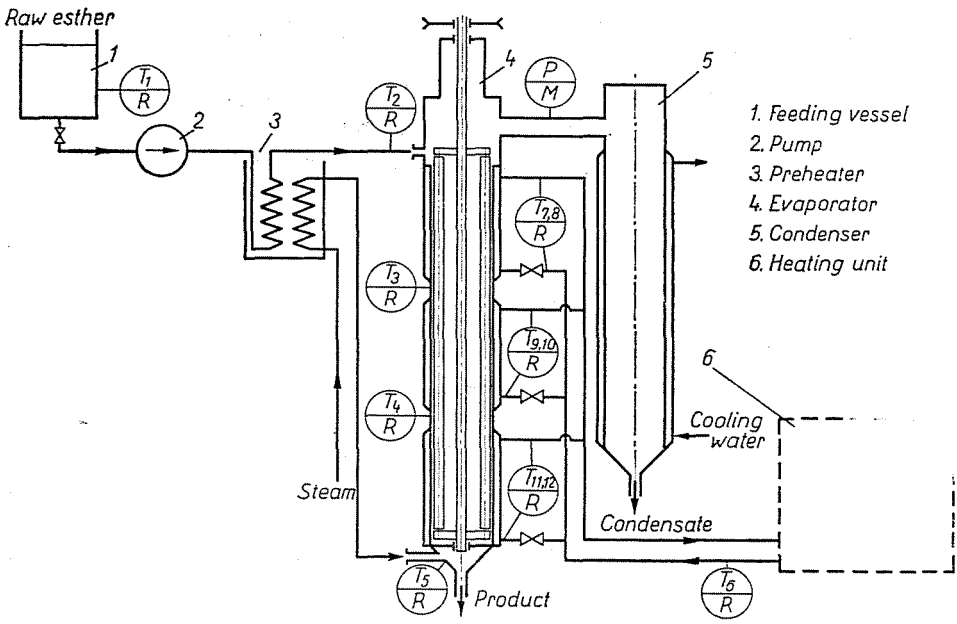

Fig. 1. The sketch of research apparatus

Description of the research work

Data of the raw ester:

density at 20 °C	0.965 g/cm³
ester number	267
acid number	0.01

The ester content, calculated from ester number is 92.8%. The raw ester contained 7.2% *i*-octyl-alcohol, water and a little active carbon.

The light component was removed in the film evaporator under vacuum with and without steam feeding. The research aimed at conditions of achieving the allowed 0.4% evaporation loss. The temperature had to be chosen from the aspect of thermal decomposition at a given temperature. Differential thermogravimetry (DTG) showed this temperature to be 165 °C where the thermal decomposition started.

Light component evaporation under vacuum

The light component evaporation without steam feeding was done at 50 to 90 Torr pressure. A known quantity of raw ester was fed to the film evaporator. As it was expected not the total amount of the light component has evaporated during once through operation, so the ester had to be recirculated several times. During test product and condensate were weighed, and the light component concentration in the product was determined. The results are seen in Table I.

The variation of light component concentration with the evaporator length is shown in Fig. 2.

Deviation of the 11 kg/h feed is due to temperature difference. Results show the evaporator length necessary to reach the allowed evaporation loss (0.4%) to increase with increasing feed.

Table I

Number of runs	Feed rate, kg/h	Heating oil temperature, °C	Film temperature, °C	Pressure, Torr	Light component concentration, %	Evaporation rate, kg/m²h
1/1	10.9	165	158	75	0.88	6.05
1/2	11.8	165	158	75	0.22	6.60
2/1	11.3	169	162	88	1.29	6.76
2/2	10.7	171	162	88	0.39	0.90
2/3	11.1	171	162	87	0.13	0.29
3/1	22.6	165	158	78	3.02	9.04
3/2	22.0	165	162	74	1.31	3.30
3/3	21.8	165	163	76	0.61	1.40
4/1	22.2	165	157	76	4.67	5.16
4/2	20.9	166	157	74	2.25	4.93
4/3	20.9	165	158	75	1.20	1.97
4/4	21.0	165	159	75	0.68	1.40
4/5	20.7	165	159	74	0.36	0.66
5/1	35.7	166	154	75	5.24	7.15
5/2	35.5	166	155	72	3.08	6.37
5/3	36.0	166	155	72	1.86	3.58
5/4	36.1	166	157	68	1.25	2.02
5/5	36.4	166	158	72	0.85	1.35
6/1	44.1	170	160	77	5.70	6.46
6/2	45.8	171	160	76	4.00	6.69
6/3	46.0	171	161	74	3.00	4.19
6/4	45.9	171	166	75	1.70	5.46

While at the feed of 11 kg/h the desired (0.4%) evaporation loss was reached after one additional recirculation at the feed of 46 kg/h the 0.4% evaporation loss was not achieved even after five additional recirculations (about 3 m evaporator length).

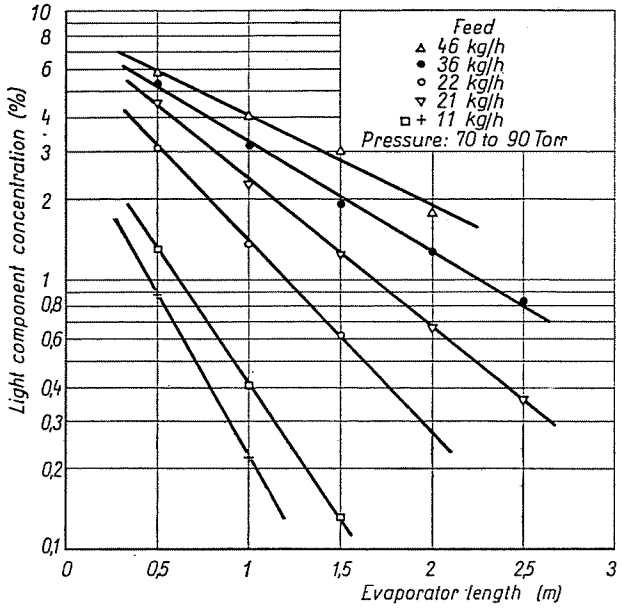

Fig. 2. The variation of light component concentration with evaporator length

Light component evaporation in vacuum with steam feed

Research covered varying raw ester feeds, steam to ester feed ratios and pressures between 75 to 300 Torr. The raw ester was recirculated three times with steam, thereafter twice without steam to remove water. The results of these runs are seen in Table II.

The variation of light component concentration with the evaporator length at different feeds is seen in Fig. 3.

Figure 3 shows the evaporation rate to be higher with the curve has steeper ascent than without steam. This difference is apparent from Fig. 4 showing the evaporation rate *vs.* average concentration of light component. During the research pressure and temperature were kept constant, the steam rate slightly changed.

The effect of pressure on evaporation rate is shown in Fig. 5. Decreasing the pressure by 100 Torr increased the evaporation rate by 20%. For higher light component concentrations the rate of evaporation is higher and its varia-

Table II

Number of runs	Feed rate, kg/h	Heating oil temperature, °C	Film temperature, °C	Pressure, Torr	Light component concentration, %	Evaporation rate, kg/m²h
1/1	35.4	162	154	200	2.18	12.46
1/2	35.7	162	152	200	0.95	3.81
1/3	35.3	162	155	200	0.35	1.92
1/4	35.1	163	158	200	0.27	0.27
1/5	34.9	164	159	200	0.23	0.14
2/1	44.3	166	140	100	2.43	11.02
2/2	45.0	167	146	100	1.28	4.38
2/3	44.2	166	151	100	0.40	3.52
2/4	44.4	165	155	100	0.34	0.26
2/5	44.6	165	158	100	0.30	0.17
2/6	44.2	165	158	100	0.28	0.09
3/1	49.3	164	155	200	2.31	0.68
3/2	50.0	163	150	200	1.12	5.13
3/3	50.6	165	155	200	0.53	2.72
3/4	49.2	165	159	200	0.45	0.38
3/5	47.8	165	158	200	0.39	0.29
3/6	49.5	166	156	200	0.35	0.26

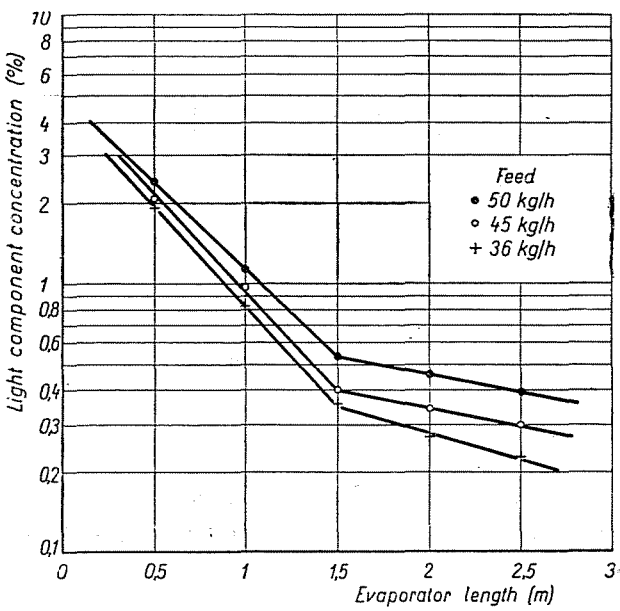

Fig. 3. The variation of light component concentration with evaporator length

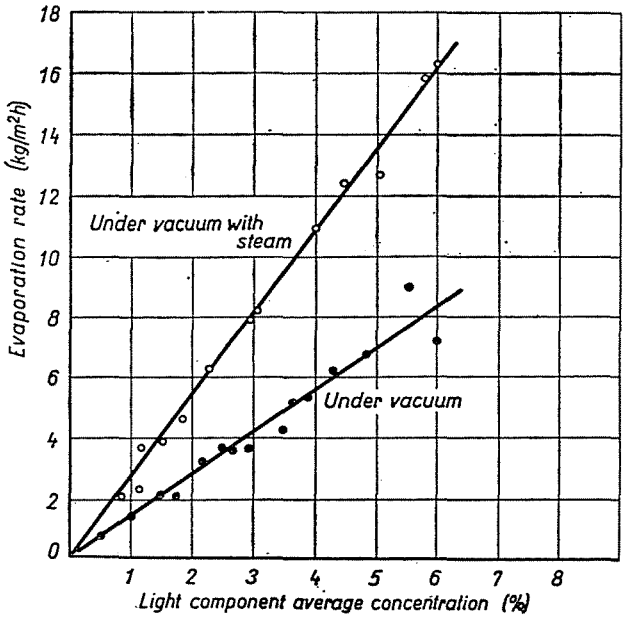

Fig. 4. The evaporation rate as a function of light component average concentration

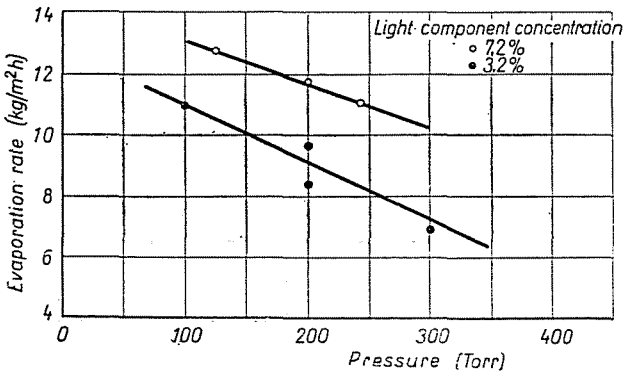

Fig. 5. The variation of evaporation rate with pressure

tion is less dependent on changes of the pressure. (The two points at 200 Torr do not coincide because of the different steam feeds.) The material loss was less than 1%.

Evaluation of the results

Our results permit to state that the light component can be evaporated from raw ester in a film evaporator.

Since the evaporation rate under steam feed is twice as high as the steamless one, it is advisable to apply steam feed.

According to our results 2.5 m heated evaporator length is necessary for the specified (0.4%) evaporation loss. Over the 2/3-rd portion of the 2.5 m evaporator length the alcohol is practically removed and the lower 1/3-rd portion is sufficient to evaporate the remaining water.

0.1 to 0.15 kg steam per 1 kg raw ester is needed to evaporate all the light component at 200 Torr pressure.

Summary

The light component evaporation from di-izo-octyl-phthalate in cylindrical roller type film evaporator was studied.

The light component evaporation was done either under vacuum or with additional steam feed. In both cases the evaporation rate was determined. The evaporation rate is twice as high in the case of steam feeding as in steamless runs.

According to our results, processing 50 kg/h ester in an 80 mm diameter film evaporator at 160 °C film temperature, the specified evaporation loss (0.4%) is reached on 2.5 m evaporator length. For 1 kg ester feed 0.1 to 0.15 kg steam is needed.

References

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