UTILISATION OF COTTONSEED OIL FOR THE PREPARATION OF OLEO-RESINOUS VARNISHES

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Tack-free coatings are prepared from cottonseed oil by open-kettle cooking process by the judicious control of several factors: (1) the cooking temperature should not be less than 300°C.; (2) a minimum of 1.0% PbO and 0.5% MnO_2 by weight of oil should be used as catalysts; (3) cooking should be to the point when the rate of change in viscosity shows a sudden rise and (4) the proportion of the resin. These coating compositions are limited to an oil length of 18 gallons, and compare favourably with those prepared from linseed oil.

Introduction

Cottonseed oil has so far been used in the paint industry only to a limited extent as an adulterant for the usual drying oils. It can be combined to a maximum of 20-25% with linseed oil and about 30-33% with tung oil for the preparation of oleo-resinous varnishes without impairing the quality of films produced. Any greater proportion of cottonseed oil will produce a tacky film. A process for the production of cottonseed oil based oleo-resinous varnishes[1] was developed at these Laboratories and the results of the study, which led to the successful utilisation of cottonseed oil for such compositions, are presented in this paper.

Experimental

Cottonseed oil used for the present study had an acid value of 0.20-0.30, and an iodine value (Hanus) of 105-108. The rosin had an acid value of 168.0 and a melting point of 76°C.

Different qualities of varnishes were prepared and are classified as follows:—

Class A: wherein the acidity of rosin is reduced by calcium oxide alone or by a mixture of calcium and zine oxides.

Class B: wherein the acidity of rosin is reduced partially by calcium and zinc oxides and partially by esterification with glycerol.

Class C: wherein the acidity of rosin is reduced by esterification with glycerol and calcium and zinc oxide are added in small quantities to catalyse the rosin-glycerol reaction.

Quality A.—Rosin is melted in an open beaker and heated to 200°C. A portion of cottonseed oil (usually 10% of the total quantity of the oil) is added and the temperature raised to 240°C. The oxides are then gradually introduced in the following order: calcium oxide, zine oxide, lead monoxide, manganese dioxide. After the addition of oxides, the balance of the oil is added to the mass and the temperature raised to and maintained at 310°C. for the desired period. Agitation of the mass is maintained throughout by means of a mechanical stirrer and samples are drawn at regular intervals for examination.

Quality C.—Rosin is melted in an open beaker and heated to 200°C. A portion of cottonseed oil is added as above and the temperature raised to 270°C. Zinc oxide (catalyst) is introduced and glycerol is added in instalments while maintaining the mass at 270-280°C. for about one hour. The balance of the oil is added gradually while maintaining the same temperature. Finally lead monoxide and manganese dioxide are added and the temperature of the mass is raised to 310°C. The cooking is continued at this temperature and samples are drawn at regular intervals for examination.

Quality B.—The method for quality B is the same as for quality A and C except that, calcium oxide and zinc oxide are added prior to the incorporation of glycerol and lead monoxide and manganese dioxide after the esterification of rosin with glycerol.

The varnishes were evaluated as follows: (1) acid value by dissolving the sample in benzene and titrating against standard alkali, (2) viscosity by bubble tube method, (3) drying properties by touching with finger at regular intervals after application and noting the time taken for the film to dry to touch, hard dry and tack-free, (4) scratch resistance as the load in grams required for rupturing the films of the varnishes on a standard scratch test apparatus, and (5) water resistance by dipping the panels in water. Tests (4) and (5) were carried out seven days after the application of the film.

Results and Discussion

It was found that under no circumstances could more than 1.5 parts of oil be used with 1.0 part rosin, to get tack-free finishes. The following composition has been chosen to show the influence of the many variables on the properties of the varnishes:

Cottonseed oil	1000 g.
Rosin	1000 g.
Zinc oxide	1.0% by weight of rosin
Glycerol	12.5% by weight of rosin
Lead monoxide	1.0% by weight of oil
Manganese dioxide	0.5% by weight of oil

Thinner used for dilution was composed of 9 parts mineral turpentine and one part gum turpentine.

Influence of Degree of Polymerisation.—Samples were drawn during cooking at different intervals and examined for (1) viscosity after dilution with 33% of thinner—a measure of degree of polymerisation and (2) drying properties. The results are shown in Table 1.

Lead Monoxide and Manganese Dioxide.—The influence of the quantities of lead monoxide and manganese dioxide and the properties of the varnishes cooked to the same degree of polymerisation, as judged by measurement of viscosity (600 centipoises after dilution with 33% thinner) has been presented in Table 2.

It is obvious that a proper control of the various factors is essential for getting tack-free finishes, and, since these results are obtainable only by open cooking, it is clear that use of oxygen is indispensable. These conditions for the different qualities have been established and are presented in Table 3. The degree of polymerisations to which these compositions must be cooked to get tack-free finishes is expressed as volatile matter

TABLE 1.—INFLUENCE OF DEGREE OF POLYMERISATION ON DRYING PROPERTIES OF VARNISH.

Period of cooking (310°C.) Hrs. Min.	Viscosity at 30°C. (centipoises)	Time (hrs.) for the film to		
		dry to touch	hard dry	be/tack-free
1— 0	40	—	—	—
1—30	60	> 24	—	—
2— 0	100	20—24	—	—
2—30	160	> 10 < 24	36	—
2—45	310	6—8	>12<24	72
3— 0	700	2—3	8	24

TABLE 2.—INFLUENCE OF CHANGE IN THE PROPORTION OF LITHARGE AND MANGANESE DIOXIDE ON THE DRYING PROPERTIES OF THE VARNISH.

Oxides % by weight of oil	Time taken to attain viscosity of 600 centipoises (hrs.)	Time (hrs.) for film to		
		dry to touch	hard dry	be/tack-free
PbO (MnO_2 0.5% fixed)				
1.0	2—55	2—3	8	24
0.8	3—30	2—3	8—10	72
0.6	4—55	3—4	12—24	—
MnO_2 (PbO 1.0% fixed)				
0.25	3.25	10—12	24—36	—
0.40	3.0	4—6	12—14	96
0.50	2.55	2—3	8	24

TABLE 3.—COMPOSITIONS FOR TACK-FREE VARNISHES.

Varnish reference No.	Oil length and quality	CaO% of rosin	ZnO% of rosin	PbO% of oil	MnO2% of oil	Glycerol % of rosin	Volatile content of varnish adjusted to 300 centistokes at 30°C.
1.	18A	7.0	—				
		4.0	3.0	0.9	0.33	—	50-56%
2.	18B	3.0	1.0	1.0	0.5	8.0	46-50%
3.	18C	1.0	0.5	1.25	0.5	12.5	44-48%
4.	12A	7.0	—				
		4.0	3.0	0.75	0.33	—	44-50%
5.	12B	3.0	1.0	0.90	0.50	8.0	40-43%
6.	12C	1.0	0.5	1.0	0.5	12.5	36-40%

TABLE 4.

Varnish No.	Oil length and quality	Load in g.	
		Cottonseed oil varnish	Linseed oil varnish
1.	18A	165-190	160-180
2.	18B	200-225	200-220
3.	18C	260-280	275-295
4.	12A	160-180	150-160
5.	12B	190-210	180-200
6.	12C	235-255	250-275

TABLE 5.—WATER RESISTANCE OF COTTONSEED OIL BASED OLEO-RESINOUS VARNISHES.

Varnish No.	Oil length and quality	Time for disappearance of haze after					
		1 hr. dip	2 hrs. dip	3 hrs. dip	4 hrs. dip	24 hrs. dip	48 hrs. dip
1.	18A	Unaffected	5 mins.	8-10 mins.	10-12 mins.	8 hrs.	24 hrs.
2.	18B	,,	Unaffected	Unaffected	Unaffected	2-2½ hrs.	10-12 hrs.
3.	18C	,,	,,	,,	,,	30 mins.	3-4 hrs.
4.	12A	,,	5 mins.	10-15 mins.	20 mins.	12 hrs.	Persists for 24 hrs.
5.	12B	,,	Unaffected	Unaffected	Unaffected	3-4 hrs.	24 hrs.
6.	12C	,,	,,	,,	,,	2-3 hrs.	8-10 hrs.

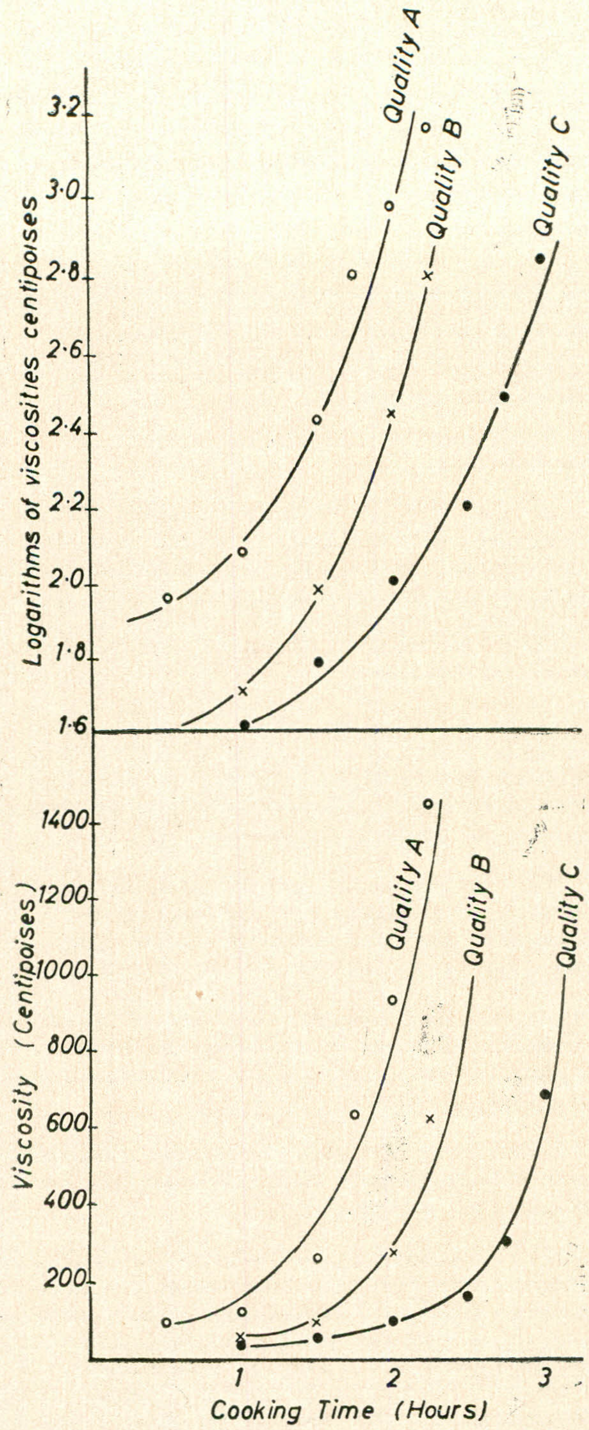

Fig. 1.—Polymerisation rates of cottonseed oil based varnishes.

required for dilution to adjust the viscosity of the varnish to 300 c.s. at 30°C.

The polymerisation rates of some of these compositions are shown in Fig. 1.

The loads for rupturing the films of the above varnishes on a standard scratch test apparatus were determind and compared with corresponding linseed oil based varnishes. The values are given in Table 4.

Water Resistance.—The varnishes were applied to glass at 0.002″ thickness, air dried for 6 days and then immersed in water. The panels were examined at intervals of 1, 2, 3, 4, 24 and 48 hours and time required for the haze to disappear after withdrawal was determined. The results are presented in Table 6. The water resistance of linseed oil based varnishes was found to be slightly better than the corresponding cottonseed oil based varnishes.

Conclusions

Cottonseed oil has for the first time been successfully used for the preparation of tack-free coating compositions by a simple open-kettle cooking process. The various factors which have to be suitably and simultaneously controlled in order to achieve the desired resuts are: (i) quantity of catalysts— a minimum of 1.0% litharge and 0.5% of manganese dioxide by weight of the oil. (2) degree of polymerisation—the point where the viscosity-cooking time curve shows a steep rise; (3) quantity of rosin—these varnishes being limited to an oil length of 18 gallons (oil length signifies gallons of oil per 100 lbs. of resin). Different qualities of varnishes can be prepared and these compare favourably with similar varnishes prepared from linseed oil. Of the various types, those in which the acidity of rosin is reduced almost entirely by glycerol (Class C) are most satisfactory. Weathering tests have shown that the paints made from cottonseed oil based varnishes are superior in ageing characteristics as compared with linseed oil varnishes.

References

1. Siddiqui and M. Aslam, Pakistan Patent 108246; Australian Patent 218, 208; British Patent 813,955; Canadian Patent 592,615; Indian Patent 60,100.