

UNITED STATES PATENT OFFICE

2,547,396

STABILIZED PARAFFIN WAX

Miles A. Joanen, Houston, Tex., assignor to Sinclair Refining Company, New York, N. Y., a corporation of Maine

No Drawing. Application October 17, 1946,
Serial No: 703,707

1 Claim. (Cl. 106—270)

1

My invention concerns a method for inhibiting the oxidation of paraffin wax and embraces, as a composition of matter, paraffin wax to which has been added the anti-oxidant or stabilizer employed in the practice of the method.

Paraffin waxes are in extensive use by paper and container manufacturers as a coating material in the production of moisture-proof wrappers and cartons for food and related industries. Before application of the wax to the paper or pasteboard surface, it is customary to heat it to a temperature of the order of 100° F.—200° F., that is to a temperature 20° F.—60° F. above its melting point, higher temperatures being generally used in the coating of cartons and the like than in the coating of paper.

Unfortunately, paraffin wax, when heated to such temperatures, tends to oxidize, the degree of oxidation depending upon the length of time it is maintained at the elevated temperature. Some of the effects of oxidation observable in the resolidified product are loss of hardness and tensile strength, discoloration, and offensive odor. All of these are obviously undesirable from the standpoint of employment of the wax as a paper or carton coating material, particularly where the paper or carton is to be used in the packaging of such items as food products, drugs, and cosmetics.

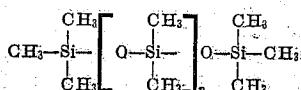
2

material identified in the trade as Dow Corning type 200 fluid. This material is described in published Dow Corning literature as according with the above empirical formula and is available in a number of viscosities, ranging from 100–1000 centistokes (at 25° C.). For my purpose I prefer the 100 centistoke material, but the more viscous grades may be applicable in some cases. Average molecular weights for the Dow Corning 200 series are reported in the "Journal of the American Chemical Society," vol. 68, pages 2284–90, 1946.

The Dow Corning fluids are generally described as a group of colorless, odorless, inert, mobile liquids which remain fluid at arctic temperatures. According to the literature, they have higher flash points than petroleum oils of equivalent viscosity; readily wet clean, dry surfaces of glass, ceramics, and metals; are incompatible with and free from solvent effect on rubber, synthetic rubbers, and organic plastics even at elevated temperatures; are insoluble in water and lower aliphatic alcohols; are soluble in most organic solvents; are stable to heat; are non-corrosive with respect to metals; are highly resistant to mineral acids and corrosive salt solutions; and have low dielectric constants and power factors over a wide frequency range. The physical properties of the 200 type fluids are as follows:

Viscosity at 25° C., centistokes	Min. flash point, °F.	Sp. gr. at 25° C.	Lb./gal. at 25° C.	Refractive Index at 25° C.	Thermal expansion coefficient per °C. ×10 ³		Viscosity, centistokes at—		
					–25 to 0° C.	25 to 100° C.	–40° F.	100° F.	210° F.
100	600	0.968	8.08	1.4030	0.926	0.969	650	82	32
200	615	0.971	8.10	1.4031	0.921	0.968	1,300	160	65
350	625	0.972	8.11	1.4032	0.917	0.966	1,950	260	135
500	625	0.972	8.11	1.4033	0.909	0.965	3,300	370	190
1,000	640	0.973	8.12	1.4035	0.900	0.963	6,500	735	260

In accordance with the present invention, I inhibit the oxidation of paraffin wax and thereby retard or mitigate the development of the undesirable qualities indicated, by incorporating in the wax a small proportion of a liquid organo-silicon polymer conforming with the empirical formula,



In my experimental work I have used the

Using the 100 centistoke material in the practice of my invention, I have found that .01% on the weight of the wax is about the minimum for satisfactory results. I have further found that the use of an amount exceeding about .04% on the weight of the wax is not advisable because the tensile strength of the wax may be thereby reduced.

The stabilizer is best incorporated in the wax with the wax in the liquid state. Generally, I heat the wax to a temperature of about 10°

above its melting point before mixing-in the stabilizer. After incorporation of the stabilizer, the

F. without agitation or introduction of air or oxygen.

TABLE I

Tests before and after stabilizing paraffin wax
with Dow Corning fluid 200

[100 centistokes]

	Unstabilized Unoxidized Wax	Wax + .01% DC Fluid	Unstabilized Wax after Oxidation	Stabilized Wax after Oxidation
M. P., ° F.	134.0	133.8	131.8	133.4
Color, Saybolt	30p	30p	Darker than 16	25
Color, NPA			Lighter than 1	Lighter than 1
Appearance	White (Opaque)	White (Opaque)	Lemon Yellow	White (Opaque)
Oil Separation (Sweating)	None	None	Excess	Slight
Tensile Strength	215	220	27	20
Penetration at 77° F.	16	17	33	28
Penetration at 85° F.	22	23	53	32
Penetration at 90° F.	29	28	141	97
Penetration at 100° F.	80	77	4.0	1.72
Cal. Oil, Per Cent	.45	.47	Rancid	Little noticeable odor.
Odor	None	None	1.45	.03
Acid Number	.02	.02	3.8	.40
Saponification Value	.50	.50		

heating may be continued until the wax has attained the proper temperature for a paper or carton coating operation or, depending upon circumstances, the source of heat may be withdrawn and the wax allowed to resolidify. Paper and container manufacturers operating on a large scale may elect to purchase unstabilized wax and to add the stabilizer as an incident of the heating of the wax to the processing temperature to avoid the necessity of twice melting the wax. It is best, however, for the producer of the wax to incorporate the stabilizer in order that the wax will be protected against surface oxidation during shipment and storage.

That the addition of effective amounts of the stabilizer does not work a change of any significance in the characteristics of the wax is shown by the accompanying Table 1 giving the

The determinations of Table II, submitted in further illustration of the improved resistance to oxidation of paraffin wax processed according to the invention, were made after an accelerated oxidation procedure in which air was introduced into the wax, maintained at a temperature of 392° F., at a rate of approximately .53 cubic foot per hour. In this procedure, the wax, 40 cc., is contained within a 10" x 1" Pyrex glass tube immersed in an agitated bath maintained at the stated temperature, the air being passed into the wax via a $\frac{1}{8}$ " glass tube. Such vigorous conditions, of course, are never actually encountered in use of paraffin wax in paper coating and related work, but such tests are nevertheless of great value in determining the effectiveness of materials proposed as anti-oxidants for paraffin waxes.

TABLE II

Accelerated oxidation tests

	Oxidizing Time	Odor	Color (Saybolt)	Appearance of Solid Wax	Oil Separation	Acid Number	Saponification Value	Calif. Oil
Unstabilized wax, 132/134° F., M. P.	None	OK	+30	White	None	.02	.50	.45
Wax + .01% D. C. Fluid	None	OK	+30	do	None	.02	.50	.45
No Inhibitor	1 hr.	rancid	+20	do	Yes ¹	0.8	3.00	.75
+ .01% D. C. Fluid	1 hr.	do	+25	do	No	0.7	2.6	.50
No Inhibitor	2 hr.	do	+17	Yellow White	Yes ¹	1.4	3.5	1.10
+ .01% D. C. Fluid	2 hr.	do	+20	Cream White	No	1.0	2.5	0.70
No Inhibitor	3 hr.	very rancid	-12	Lemon	Yes ¹	1.8	4.1	1.90
+ .01% D. C. Fluid	3 hr.	rancid	+16	Light Lemon	No	1.2	3.2	0.79
No Inhibitor	4 hr.	very rancid	-15	Dark Lemon	Yes ¹	2.5	6.2	2.70
+ .01% D. C. Fluid	4 hr.	rancid	+10	Light Lemon	Yes ¹	2.0	4.1	0.81
No Inhibitor	5 hr.	rank	B-16	Light Yellow	Yes ¹	3.6	10.4	3.00
+ .01% D. C. Fluid	5 hr.	rancid	-8	Very Light Yellow	Yes ¹	2.3	7.8	1.00
No Inhibitor	6 hr.	rank	B-16	Dark Yellow	Yes ²	4.0	12.2	3.20
+ .01% D. C. Fluid	6 hr.	rancid	B-16	Light Yellow	Yes ¹	2.8	9.1	1.10
No Inhibitor	7 hr.	rank	B-16	Dark Yellow	Yes ²	5.1	15.3	3.70
+ .01% D. C. Fluid	7 hr.	rancid	B-16	Light Yellow	Yes ²	3.1	11.4	1.21
No Inhibitor	8 hr.	rank	1 NPA	Very Dark Yellow	Yes ³	6.4	18.2	4.00
+ .01% D. C. Fluid	8 hr.	do	1 NPA	Light Yellow	Yes ³	4.6	13.2	1.72

¹ Free oil present in small quantities. ² Medium quantities. ³ Large quantities.

characteristic of a representative wax before and after stabilization. Table 1 also shows, columns 3 and 4, the effect of oxidation on the stabilized and unstabilized wax. The determinations in these columns were made after resolidification of samples which had been heated in a 100 cc. beaker, filled to capacity, for 8 hours at 375°

Table III illustrates the effect of progressive additions of the inhibitor. It will be observed therefrom that the tensile strength of the wax decreases as the proportion of stabilizer is increased, also that the stabilizer has an effect on the hardness of the wax as measured by its penetrability.

TABLE III

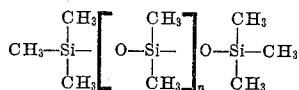
Progressive additions of inhibitor

132/134° F. Wax, Per Cent.....	99.99	99.98	99.96	99.94	99.92	99.90	99.80	99.60	99.40	99.20	99.00
Dow Corning Fluid 200, 100 Centistokes, Per Cent.....	.01	.02	.04	.06	.08	.10	.20	.40	.60	.80	1.00
Tests:											
Melting Point, ASTM.....	133.8	133.6	133.4	133.4	133.4	133.4	133.4	133.4	133.4	133.2	133.2
Penetration at 77° F.....	17	17	17	17	17	17	17	17	18	18	18
Penetration at 85° F.....	23	25	25	26	26	26	26	27	28	28	28
Penetration at 90° F.....	28	29	29	30	34	34	35	35	35	36	36
Penetration at 100° F.....	77	78	79	79	79	80	80	90	90	90	98
Tensile Strength.....	220	195	190	170	160	140	135	130	125	120	110

My invention, of course, is not limited to paraffin wax as employed in the production of moisture-proof wrapping papers and containers. I have described it with particular reference to the paper industry because, at the moment, it is in that field that the invention appears to have the greatest commercial promise.

I claim:

A paraffin wax stabilized against oxidation by the incorporation of from about .01 per cent to .04 per cent of a liquid organo-silicon polymer of the Dow Corning fluid 200 conforming with the empirical formula



MILES A. JOANEN.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
297,766	Chemin	Apr. 29, 1884
2,383,521	Sowa	Aug. 28, 1945
2,416,504	Trautman	Feb. 25, 1947

OTHER REFERENCES

- Rachow et al., "Journal of the American Chemical Society," 63, 798-800.
 30 "Commercial Waxes," Bennett (1944), pages 10, 11 and 53.