

Epoxy Acids and Salts as Stabilizers for Vinyl Plastics

Although metal salts of fatty acids have been widely used as stabilizers of plasticized poly(vinyl chloride) and its copolymers, little information is available on the effect of free fatty acids on heat and light stability. A recent report (1) states that epoxystearic acid acts as a heat stabilizer for poly(vinyl chloride). In addition, no systematic study has been reported on the effect of structure of free fatty acids on heat and light stability. It is known that the stabilizing effect is improved when mixtures of metal salts are employed as stabilizers (2, 3). When the salts are used with an epoxy resin or epoxy plasticizer, a great improvement of both heat and light stability is achieved (2). Therefore, the incorporation of the metal salt and the epoxide into one molecule would be expected to produce an improvement of the stabilizing properties over those of the separate components.

Scope

The present work reports the results of the investigation of the effect on the heat and light stability of plasticized poly(vinyl chloride) using as stabilizers structurally different C_{18} fatty acids, namely a saturated acid (stearic), a monounsaturated acid (oleic), a saturated acid with an epoxy group (epoxystearic), and a monounsaturated acid with an epoxy group (epoxyoleic), the barium and cadmium salts of the acids and mixtures of the salts. In addition, the calcium and zinc salts of epoxyoleic acid and mixtures of these salts were studied at the same ratios as the barium and cadmium salts.

Experimental

Materials: The materials investigated and their characteristics are found in *Table 1*. The epoxyoleic acid was a naturally occurring material derived from *Vernonia anthelmintica* seed oil (4). The detailed method of preparation of the fatty acid salts was previously described (5).

Stabilizer Evaluation: The resin used for this study was Geon 101 [poly(vinyl chloride)], plasticized with DOP (di-2-ethylhexyl phthalate), employing the following recipe: Geon 101, 65%; plasticizer-stabilizer combination, 35%. For simplicity, stabilizer evaluation was made at both the 1 and 3% weight levels of the total mix. Mixtures of the barium and cadmium salts were evaluated for each of

the fatty acids at the following barium to cadmium salt weight ratios: 1:3, 1:1, 3:1, 5:1, and 10:1. The same ratios were evaluated for calcium and zinc salts of epoxyoleic acid.

The components of the various formulations were dry blended in a beaker with a spatula until a uniform mixture was obtained. The mixture was then placed on the moving rolls of a 4 x 8-in (10 x 20 cm) rubber mill at a temperature of approximately 160°C. and milled for approximately 7 min during which time the material was repeatedly cut and worked.

Test sheets were molded in a standard 6 x 6 x 0.075-inch (15 x 15 x 0.19 cm) mold. The mold containing the milled sheet which had been cut into 3-in (8 cm) squares and stacked in the mold was preheated in the press without pressure at about 160°C. for 10 min and then pressured to 1000 psi (70.3 kg/sq cm) and held for an additional 10 min at 160°C. The mold was then cooled under pressure until it was about room temperature and removed from the press. All formulations were milled and molded at 160°C. The specimens were equilibrated at 23°C. and 50% relative humidity for at least 24 hrs prior to testing.

Heat stability tests were conducted on the molded specimens in an air convection oven at 160°C. Samples were removed periodically and the relative heat stabilities were determined by measurement of the color change at 600 $m\mu$ wavelength with a visible range spectrophotometer. The percent transmittance at a wavelength of 600 $m\mu$ was chosen since it provided the best quantitative measure of the development of color in the specimens.

Light stability tests were conducted by exposing the molded specimens to the light emitted by the twin carbon arcs of a weatherometer. During exposure the samples were necessarily heated; the temperature averaged about 65°C. Specimens were examined at 24-hr intervals for four signs of degradation: spotting, discoloration, stiffness, and tack formation. These factors were evaluated on a numerical scale of 1 to 5 as follows:

1. No degradation.
2. Slight degradation.
3. Moderate degradation.
4. Severe degradation.
5. Very severe degradation.

Table 1. Characteristics of C₁₈ Acids and Salts Investigated as Stabilizers

| Materials | Melting Point °C. | Acid Number | | Iodine Value | | Barium, Cadmium, Calcium or Zinc % | | Oxirane Oxygen % | |
|-----------------------|-------------------|--------------|-----------|--------------|--------|------------------------------------|--------|------------------|--------|
| | | Found | Theory | Found | Theory | Found | Theory | Found | Theory |
| | | Stearic Acid | 68.7-69.4 | 197.8 | 197.2 | 0.2 | 0 | — | — |
| Oleic Acid | 14.4-15.8 | 197.8 | 198.6 | 88.9 | 89.8 | — | — | — | — |
| Epoxy stearic Acid | 57.7-58.5 | 174.7 | 188.0 | 0.2 | 0 | — | — | 4.69 | 5.36 |
| Epoxyoleic Acid | 29.8 | 180.9 | 189.3 | 87.6 | 85.6 | — | — | 5.06 | 5.40 |
| Barium Stearate | 245-246 | .2 | 0 | 0.2 | 0 | 20.29 | 20.89 | — | — |
| Barium Oleate | 134-137 | 15.1 | 0 | 72.3 | 72.5 | 18.00 | 19.61 | — | — |
| Barium Epoxystearate | 143-145 | 1.3 | 0 | 1.3 | 0 | 17.44 | 18.76 | 4.09 | 4.37 |
| Barium Epoxyoleate | 128-129 | 1.2 | 0 | 72.1 | 69.7 | 17.86 | 18.86 | 4.22 | 4.39 |
| Cadmium Stearate | 105-106 | — | — | 0.4 | 0 | 17.71 | 17.77 | — | — |
| Cadmium Oleate | 54-55 | — | — | 52.4 | 75.2 | 15.70 | 16.65 | — | — |
| Cadmium Epoxystearate | 99-101 | — | — | 1.6 | 0 | 14.70 | 15.89 | 4.12 | 4.52 |
| Cadmium Epoxyoleate | 93-94 | — | — | 73.9 | 72.2 | 14.17 | 15.98 | 4.42 | 4.55 |
| Calcium Epoxyoleate | 80-82 | 1.6 | 0 | 79.3 | 80.5 | 6.09 | 6.35 | 4.76 | 5.07 |
| Zinc Epoxyoleate | 102-103 | — | — | 77.6 | 77.4 | 9.77 | 9.96 | 4.48 | 4.88 |

The failure of a compound was arbitrarily chosen as the length of exposure needed for moderate degradation to occur by any one of the above modes of breakdown or for slight degradation to occur in any three modes of breakdown.

Processing Effects

All materials showed good stability during milling. The effect on the molded sheets, due in part to the heat during processing, varied considerably as shown in *Table 2*. Both 1 and 3% of stearic acid produced the following results. Surface exudate formed on the sheets in less than an hour, and the surface of the mold plates was severely attacked by the 3% level leaving a permanent stain, while the 1% level produced only a slight stain. None of the other materials tested attacked the plates. At both stabilizer levels the freshly molded sheets containing epoxystearic acid and epoxyoleic acid were transparent and appeared to have the same color (pale amber); however, upon conditioning overnight, the samples containing epoxyoleic acid bleached, even though they were held in the dark. The barium salts at 1% produced faint amber colored sheets and at 3% the sheets were pale amber. The increased color at the higher level was not unexpected since it is known that increasing barium content increases the color with some barium salts (6). The sheets obtained using the mixed salts ranged from a faint yellow tint at the high cadmium ratio to a pale amber at the high barium ratio. The 3% level tended to produce a more opaque sheet as will be shown in the following discussion.

Heat Stability

The heat stabilizing effects are shown in *Figure 1* for the 1 and 3% levels of stearic, oleic, epoxystearic and epoxyoleic acids during exposure at 160°C. An unstabilized control is also included. The percent transmittance at 600 mμ wavelength is plotted vs heating time in hours. The color which had developed during processing of the test specimens as previously discussed is indicated by the transmittance at 0 time. The 1 and 3% stearic and 1% oleic acid levels produced the same results. At both levels, the stearic and oleic acids produce lower transmittance than the unstabilized control with the oleic acid producing slightly poorer results at the 3% level.

The epoxystearic and epoxyoleic acids at both levels show stabilizing ability as demonstrated by the higher percent transmittance compared to the control. The initial transmittance (0 time) of both the 1 and 3% epoxyoleic acid samples is considerably higher than the corresponding epoxystearic acid samples, probably due in part to the bleaching previously mentioned.

An additional test was made using a mixture of equal parts by weight of oleic acid and epoxystearic acid at the 3% level. Before testing the molded specimen was amber colored having a transmittance of 27%. After the first 15 minutes' exposure at 160°C. the transmittance increased to 41%. With further exposure there was a constant rate of decrease in transmittance to complete failure at 3¼ hours. This produced stability (transmittance) about midway between that shown by the 1% epoxystearic and the 3% epoxyoleic acids.

Figure 2 shows the stabilizing effect of the cadmium salts of stearic, oleic, epoxystearic and epoxyoleic acids at the

Table 2. Processing Effects

| Stabilizer | Characteristics of Molded Sheet | | |
|-------------------|---------------------------------|-------------------|--|
| Stearic Acid | 1 and 3%; | Semi-transparent; | Dark amber |
| Oleic Acid | 1 and 3%; | Transparent; | Dark amber |
| Epoxystearic Acid | 1 and 3%; | Transparent; | Pale amber |
| Epoxyoleic Acid | 1 and 3%; | Transparent; | Pale amber |
| Barium Salts | 1%; | Transparent; | Faint amber |
| | 3%; | Transparent; | Pale amber |
| Cadmium Salts | 1 and 3%; | Transparent; | Slight yellow tint |
| Zinc Salt of | | | |
| Epoxyoleic Acid | 1 and 3%; | Opaque; | Black |
| Calcium Salt of | | | |
| Epoxyoleic Acid | 1%; | Transparent; | Amber |
| | 3%; | Semi-transparent; | Dark amber |
| Mixed Salts | 1 and 3%; | Transparent; | Ranged from faint yellow tint to amber |

1% level. All samples initially demonstrate about the same transmittance and have about the same rate of degradation, with the exception of the epoxyoleic which shows better stability with complete failure at 2 hrs while the other three samples failed at 1½ hrs.

At the 3% level the cadmium salts show a greater difference in their stabilizing effects (Figure 3). The stearic acid salt has the highest initial transmittance, but also exhibits the most rapid degradation with almost complete degradation at 2 hrs. In contrast, the epoxyoleic acid salt which has a lower initial transmittance, shows little change up to 6 hrs followed by an increasing rate of degradation. It should be noted that at the increased stabilizer level there is a decrease in the initial transmittance, as compared to the 1% level, with the exception of the stearic acid salt which shows essentially no decrease. This is not due to color formation, but is the effect of the various salts on the transmittance. It can be seen that the cadmium salts greatly increase the stability, as compared to the acids.

The stabilizing effect of the barium salts at the 1 and 3% levels is shown in Figures 4 and 5, respectively. Although there is a great increase in heat stability at the 3% level and a slower rate of degradation, the transmittance is decreased compared to the 1% level. In the case of the oleic salt, there is a 33% decrease in transmittance at the 3% level. As previously mentioned, this is due mainly to color formation caused by the barium. The barium salts, with the exception of the stearic salt, show a lower stabilizing ability than the corresponding cadmium salts.

The effect of the calcium salt of epoxyoleic acid on the heat stability is seen in Figure 6. The unstabilized control, starting with about 18% transmittance (0 time), shows rapid degradation on heating with complete failure at 1½ hrs. Both the 1 and 3% calcium salt levels show more stability than the control. Unlike the control, they show an increase in transmittance during the first 15 minutes' heating with the 1% level exhibiting the greatest increase and highest transmittance. In general, the calcium salts show a lower stabilizing ability than the barium salts.

Effects of Salt Mixtures

Varying the ratio of the salts at the 1% level from 1:3 to 10:1 of barium to cadmium, as expected, produced some variation in heat stability with the 3:1 ratio producing the

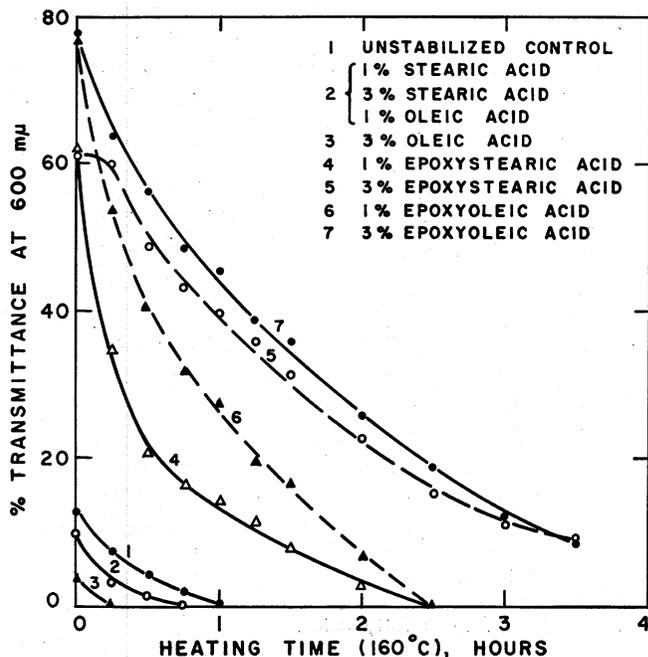


Figure 1. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing 1 and 3% of various fatty acids.

best stability. All of the fatty acid salt mixtures studied had a transmittance of about 80% before exposure to heat. There was good stability up to about 1 hr for all the samples with essentially no change in transmittance which was followed by a rapid rate of degradation. This rate varied for the different fatty acids, with the epoxystearic producing the longest time to reach complete failure (0 transmittance). All had greater stability than a commercial liquid barium-cadmium complex (5).

At the 3% level (Figure 7), a much greater difference was produced by the different fatty acids as demonstrated by the 1:1 ratio of barium to cadmium which was generally

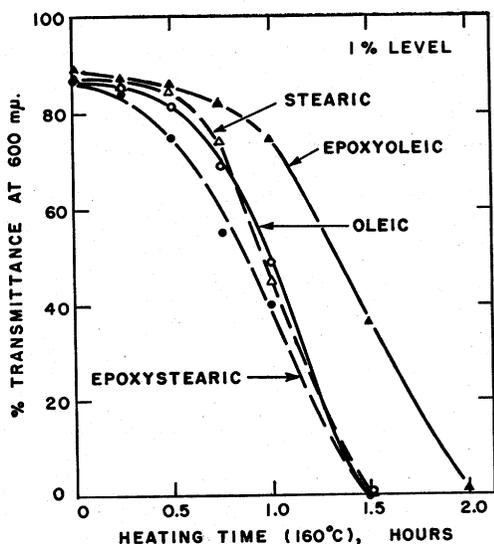


Figure 2. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing 1% cadmium salt of various fatty acids.

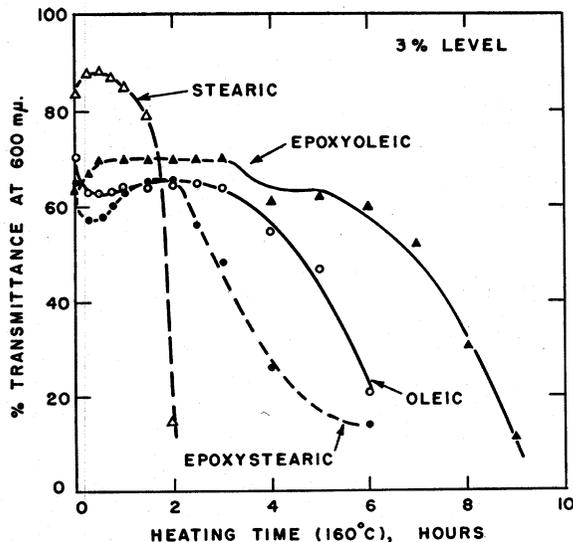


Figure 3. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing 3% cadmium salt of various fatty acids.

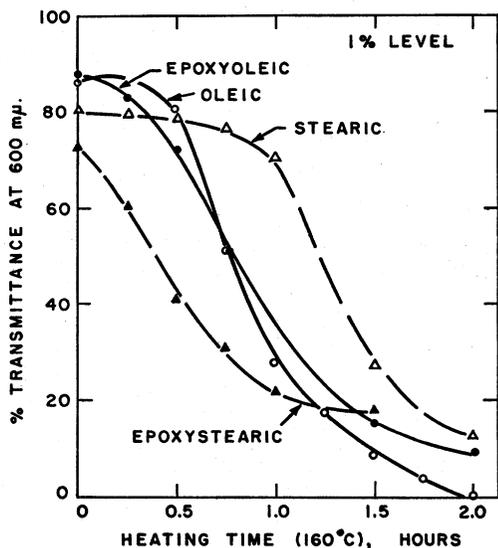


Figure 4. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing 1% barium salt of various fatty acids.

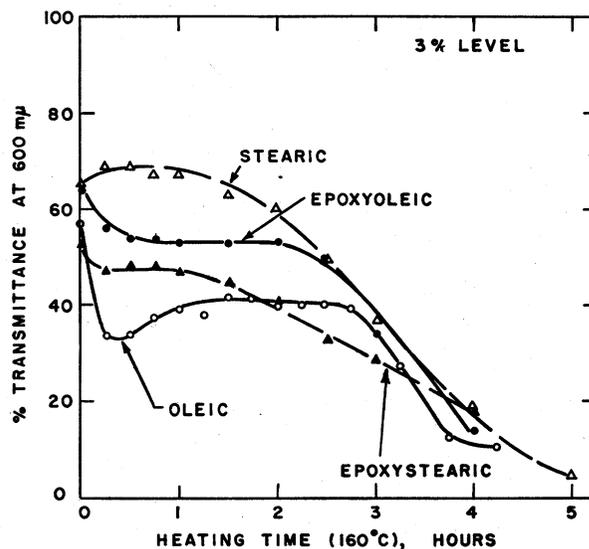


Figure 5. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing 3% barium salt of various fatty acids.

found to produce about the best stability for this stabilizer level. It should be noted that at this level there is a marked effect on the initial transmittance of the different fatty acids. This is not a color factor, but is mainly associated with the higher concentration of the salts. The stearic mixture exhibits the highest initial transmittance; however, it also shows the fastest rate of degradation, producing stability somewhat below that of the commercial liquid barium-cadmium complex (5). The epoxyoleic which shows a lower initial transmittance has a much slower rate of degradation. The lowest transmittance and the slowest apparent rate of degradation is demonstrated by the oleic plot which shows little change up to 8 hrs. The stability at this level is about 3 times greater than that found at the 1% level.

The effect on heat stability of various mixtures of calcium and zinc salts of epoxyoleic acid at the 1, 1.65 and 3% levels is shown in Figure 8 for the ratio of 5:1 of calcium to zinc. The ratio of 5:1 was found to produce the best effect of the ratios studied. Also shown for comparison are the 1 and 3% levels of a commercial calcium-zinc stabilizer (59-V-11), mainly a mixture of palmitic and stearic acids. The 1% commercial sample shows high initial transmittance (0 time) with a short period of stability followed by rapid failure ($\frac{3}{4}$ hr). The 3% commercial sample shows slightly lower initial transmittance followed by a sharp drop in transmittance and then a longer period of stability (about 2 hrs), then rapid failure ($2\frac{1}{2}$ hrs). At the 1% level, the epoxyoleic salt mixture shows slightly greater stability with

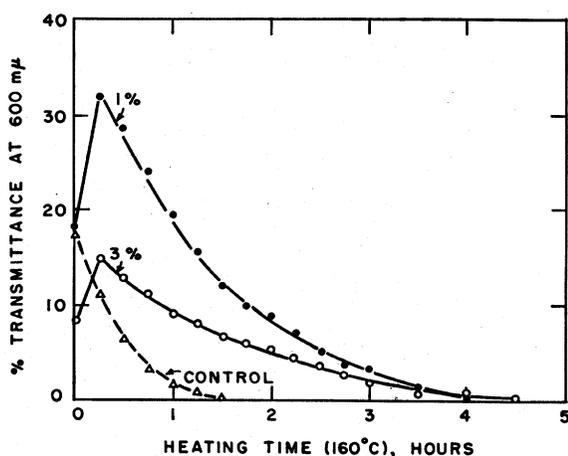


Figure 6. Variation in transmittance with time of conditioning at 160°C for plasticized poly(vinyl chloride) containing calcium salts of epoxyoleic acid and unstabilized control.

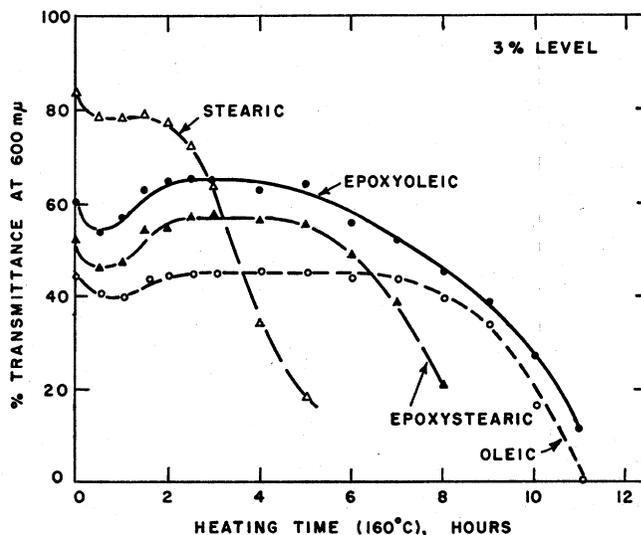


Figure 7. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing 3% (Ba/Cd) salt mixtures at 1:1 ratio for various fatty acids.

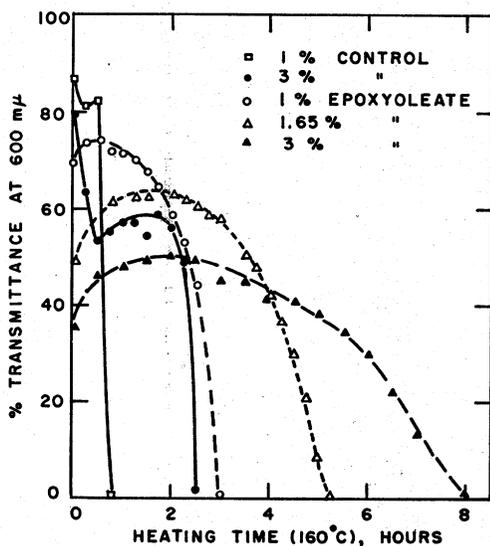


Figure 8. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing various levels of (Ca/Zn) salt mixtures of epoxyoleic acid at 5:1 ratio and control.

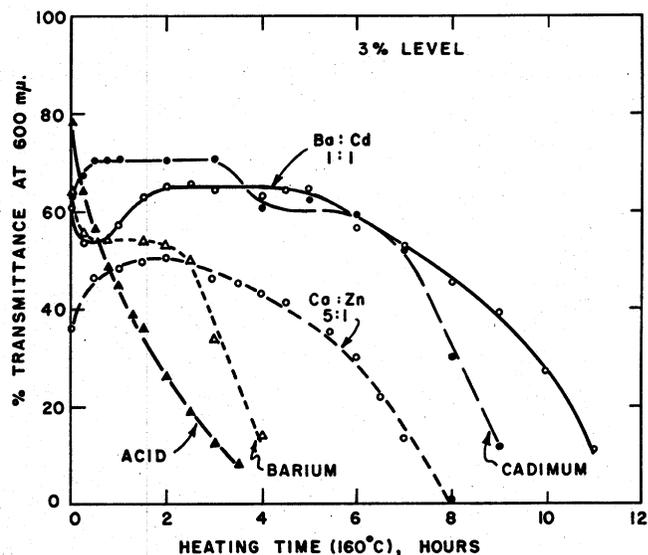


Figure 9. Variation in transmittance with time of conditioning at 160°C. for plasticized poly(vinyl chloride) containing epoxyoleic acid and salts at 3% level.

higher transmittance, compared to the 3% commercial stabilizer. At higher levels, the effect of increasing the epoxyoleic salt level is seen to extend the stability accompanied by a loss in transmittance with increase in stabilizer content. All levels show greater heat stability than either the 1 or 3% levels of the commercial stabilizer. In addition, the commercial stabilizers show a dry waxy exudate after heat treatment not shown by the epoxyoleic samples.

A great difference in heat stability was noted due to the different ratios of the salts. High zinc caused short stability followed by rapid degradation. As the calcium ratio increased over the zinc ratio, the stability increased up to the 5:1 ratio of calcium to zinc; above this ratio the stability again was found to decrease, but at a slow rate of degradation.

Examination of the data obtained for the mixtures of the salts at the levels investigated shows that the stearic and oleic acid salts produced a synergistic effect. Contrary to a recent report (7) the epoxystearic acid salt mixtures also exhibited synergism. The epoxyoleic acid salt mixtures also produce a synergistic effect for the barium-cadmium mixtures and the calcium-zinc mixtures. The calcium-zinc combination produced a marked synergistic effect.

In Figure 9 the heat stabilizing effect of epoxyoleic acid at the 3% level is summarized for the acid, barium salt, cadmium salt, the 1:1 ratio of these salts and the 5:1 ratio of the calcium-zinc salts. The acid produces high initial transmittance followed by rapid failure. The barium salt shows a lower initial transmittance and some improvement in stability. The calcium-zinc mixture has a still lower initial transmittance but a much greater stability. The cadmium salt with higher transmittance shows a further increase in the stability. The 1:1 mixture of the barium and cadmium salts initially shows stability somewhat lower than that of the cadmium salt, but after 7 hrs shows a greater resistance to degradation than the cadmium salt.

Light Stability

The results of the ability of the various materials to stabilize against degradation due to light are shown in Tables 3 and 4. The time to failure was determined as described in the experimental procedure. The color present in the

molded test specimens diminished or completely disappeared after about 24 hours' exposure in the weatherometer. The stearic acid failure was due to heavy exudate and the other acids failed due to tack formation. All failures of the salts and mixed salts were due to spotting.

The stearic and oleic acids produce no light stabilizing effects at either level. The epoxystearic and epoxyoleic acids at the 1% level exhibit stability as good or better than the salts or mixtures of the salts at this level. At the 3% level these acids are not nearly as effective as the salts. The effectiveness of the acids improves from epoxystearic to epoxyoleic at the 1% level, but is the reverse at the 3% level due, in part, to compatibility.

The cadmium salts of stearic, oleic and epoxystearic produce about the same stability. The greatest effect is demonstrated by the epoxyoleic salt at the 3% level which produces stability about a third greater than the other cadmium salts.

A more definite pattern is shown by the barium salts with the effectiveness increasing in the order stearic < epoxystearic < epoxyoleic < oleic. At the 1% level the greatest effect is noted by the oleic salt.

In the case of both cadmium and barium salts the greatest stability occurs where there is unsaturation present.

The calcium salt of epoxyoleic acid at the 1% level is more effective than either the barium or cadmium salts of the different acids with the exception of the barium oleic salt. At the 3% level the calcium salt is about equal to the barium epoxyoleic acid salt.

A very definite effect on light stability with respect to the different acids is produced when mixtures of barium and cadmium are used, with stability increasing in the order stearic < epoxystearic < oleic < epoxyoleic. It can be seen that the structural effect of unsaturation and epoxide both improve the stability of the mixtures and that the combination of unsaturation together with the epoxide produces the greatest stability. The higher level of the salt also produces the greatest stability. The mixed barium and cadmium salts of epoxyoleic acid were found to be slightly better than a commercial barium-cadmium complex (Marl M) which failed at 264 and 696 hrs at the 1 and 3% levels respectively.

Table 3. Light Stability

| Materials | Hours to Failure | | | | | | | |
|--------------|------------------|-----|--------------|-----|-------|-----|------------|-----|
| | Stearic | | Epoxystearic | | Oleic | | Epoxyoleic | |
| | 1% | 3% | 1% | 3% | 1% | 3% | 1% | 3% |
| Acid | 0 | 0 | 192 | 240 | 0 | 0 | 264 | 72 |
| Cadmium Salt | 168 | 480 | 120 | 504 | 192 | 504 | 168 | 744 |
| Barium Salt | 96 | 264 | 168 | 360 | 336 | 528 | 168 | 432 |
| Calcium Salt | — | — | — | — | — | — | 240 | 408 |

| Barium: Cadmium Ratios | Stearic | | Epoxystearic | | Oleic | | Epoxyoleic | |
|------------------------|---------|-----|--------------|-----|-------|-----|------------|-----|
| | 1% | 3% | 1% | 3% | 1% | 3% | 1% | 3% |
| 1:3 | 168 | 360 | 144 | 408 | 216 | 504 | 240 | 720 |
| 1:1 | 168 | 336 | 168 | 408 | 216 | 480 | 288 | 624 |
| 3:1 | 144 | 336 | 144 | 360 | 192 | 504 | 288 | 600 |
| 5:1 | 144 | 336 | 168 | 360 | 216 | 504 | 288 | 504 |
| 10:1 | 144 | 336 | 168 | 408 | 192 | 360 | 240 | 624 |

Table 4. Light Stability

| Calcium: Zinc Ratios | Epoxyoleic | |
|----------------------|------------|-----|
| | 1% | 3% |
| 1:3 | — | 408 |
| 1:1 | 360 | 768 |
| 3:1 | — | 456 |
| 5:1 | 192 | 480 |
| 10:1 | — | 408 |

A decrease in stability at the high barium ratio is noted in the case of the oleic salt mixture at the 3% level. In contrast, the high cadmium level increases the stability of the epoxyoleic salt mixture at the higher stabilizer level.

The calcium-zinc epoxyoleic salt mixtures show that the best effect is obtained when the weight ratio of 1:1 is employed (Table 4). Much greater stability is found to be produced by these salts compared with a commercial calcium-zinc stabilizer (59-V-11) which failed at 192 and 360 hrs for the 1 and 3% levels, respectively. The exceptional stability of these salts is further demonstrated by the results obtained employing a 1:1 metal ratio of the salts at a 1.65% level which showed 456 hours to failure.

Summary

At the stabilizer levels investigated stearic and oleic acids do not produce heat or light stability, but apparently contribute to degradation. Epoxystearic and epoxyoleic acids do possess ability to stabilize against heat and light degradation. The epoxyoleic acid produced slightly better stabilization than the epoxystearic acid. The barium salts of the acids greatly improve heat and light stability over that of the free acids. Heat stability at the higher level increased in the order: oleic < epoxystearic < epoxyoleic < stearic. In general the cadmium salts further improved the heat and light stability. Heat stability increased in the order: stearic < epoxystearic < oleic < epoxyoleic. Barium and cadmium combined further increased both heat and light stability, except that the mixed epoxyoleic salts showed slightly lower light stability than the cadmium salt of epoxyoleic acid. The stability of the mixed salts increased in the order: stearic < epoxystearic < oleic < epoxyoleic. The cadmium salt of epoxyoleic acid which possesses the combination of monounsaturated and an epoxy group showed the greatest light stability of all the systems that were investigated and produces heat stability which approaches that found for the mixed salts of epoxyoleic acid.

Mixtures of the calcium and zinc epoxyoleic salts showed exceptional stabilizing ability when compared with a commercial control.

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