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High performance metal stearates thermal stabilizers for poly vinyl chloride

Abstract

Low thermal stability of poly vinyl chloride (PVC) is one of the most serious problems that facing its processing. In the present study some single and mixed metal stearate soaps were prepared, characterized and evaluated for their performance in enhancing and imparts the thermal stability of PVC. Accordingly, calcium stearate, barium stearate, zinc stearate and also the mixed metal stearate salt of calcium, zinc and barium were prepared by the double decomposition of sodium stearate and single or mixed metal acetate solutions. The prepared metal stearates were characterized via spectroscopic analysis (FT-IR and XRD) in addition to micro analysis and thermal gravimetric analysis (TGA). The results showed that the prepared metal stearates are good thermal stabilizers for PVC; also the thermal stability of the prepared (Ca/Ba/Zn) stearate was enhanced when melamine, ammonium poly phosphate, magnesium hydroxide and Ca/Zn phosphate are mixed with it.

Keywords: poly vinyl chloride, thermal stabilizer, metal stearates

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Abbreviations: PVC, Poly vinyl chloride; XRD, X Ray Diffraction; TGA, Thermo Gravimetric Analysis; FT IR, Fourier Transform Infra Red; Ca/Ba/Zn, Calcium, barium, zinc; Ba/Zn Barium, zinc, Ca/Zn, Calcium, zinc; DOP, Dioctyl phathalate; ESBO, Epoxidized soya bean oil; min, Minute

Introduction

PVC is an important thermoplastic material on account of its versatility and low cost, however one major drawback of PVC is that it decomposes at temperature lower than its processing temperature, thermal degradation of PVC is the result of dehydrochlorination on exposure to heat[1,2] which generates polyene sequences in polymer chains that may produce an undesirable color in material during its molding and use,[3,4] in the advanced stages of degradation PVC losses mechanical, electrical and rheological properties as a result of chain scission and cross linking.[5] During PVC processing, dehydrochlorination of PVC affects on mass loss and as a result of that color change to yellow, orange, red, brown and finally black due to the number of conjugated bonds that formed.[6] To overcome this problem, thermal stabilizers used to protect PVC from chemical degradation effects of heat, without the use of thermal stabilizers PVC could not be the widely used polymer.[7] During the polymerization process the normal head to tail free radical reaction of vinyl chloride deviates from the normal path and result in sites of lower chemical stability or defect sites along some of the polymer chains, thermal stabilizer technology depends on prevention or repairing these defect sites.[7] Thermal stabilizers include a wide variety of metallic soaps, commercial heat stabilizers in the market are actually synergetic mixtures of various compounds (mixed metal stearates and metal hydroxides),[6] Magnesium hydroxide can be used with metallic soaps to enhance the thermal stability of PVC because it release weak Lewis acid (magnesium chloride) when it react with hydrochloric acid.

In general, the stabilizers are classified as lead salts,[8] organic tin,[9] soap salts[10] and rare earth stabilizers.[11] Stabilizers play the role in stability mainly through absorption of hydrochloric acid (HCl) released from partial PVC decomposition or substitution of labile sites of partially decomposed PVC macromolecules.[5] Application of lead salts and organic tin stabilizers are limited due to their toxicity, even they have high efficiency to stabilize PVC. The long-chain Carboxylate of metal ions are compounds of considerable commercial importance and are employed in various fields. For example, zinc soap is used as an ant moisture agent, a lubricant in polymers,[12] or a dispersing agent in pigments. Mixed metal stearates predominate in the flexible PVC applications in the United States and they find competition from lead based products in Europe for electrical wires and cable coatings.[7] In Europe mixed metal stabilizers are preferred for extruded rigid building profiles because they provide good weathering and physical properties to PVC in this use, The most popular commercial products are combination including Ca/Zn, Ba/Ca/Zn and Ba/Zn, Calcium/zinc mixtures are used to stabilize PVC food packing, mineral water bottles and pharmaceutical containers thought the world[7] Calcium/zinc has synergistic effect and present favorable stabilization; however Ca/Zn stabilizers have some disadvantages in long term stability due to its zinc content.

Hence extra substance must be added so as to improve the efficiency of stabilization such as epoxidized soya bean oil[13] Melamine which is an organic base which contains 66% nitrogen by mass so it can capture hydrochloric acid and, if mixed with resins, has fire retardant properties due to its release of nitrogen gas when burned or charred,[14] Ammonium polyphosphate which is an inorganic salt of polyphosphoric acid and ammonia containing both chains and possibly branching. Its chemical formula is $[NH_4 PO_3]_n$ and it can react with hydrochloric acid; ammonium polyphosphates decompose to form ammonia and phosphoric acid so it act as fire retardant.[15] Devrim Balkose et al.,[16] studied the synergism of calcium and zinc soaps on thermal stabilization of PVC using solvent casted films of PVC having commercial zinc and calcium soap in different proportions. The metal soaps were characterized by X-ray diffraction and X-ray emission techniques, The films that were heated at 80°C for 4 hr for the removal of the last traces of solvent methyl ethyl ketone were tested for thermal stability by heating at 160°C for 30 min. heated films

were characterized by infrared and ultraviolet-visible spectroscopy, DSC and TGA methods the migration of zinc, calcium and hydrogen cations to water at 80°C was also studied. UV spectroscopic analysis and ion migration indicated synergism on PVC heat stability for 4:1 Ca to Zinc soaps ratio

Experimental work

Materials

i. Suspended Poly vinyl chloride with k-value 70 was obtained from Egyptian company for petrochemicals (Alexandria)

ii. Filler: Coated calcium carbonate was obtained from green Egypt Company for carbonates (10th of Ramadan)

iii. Plasticizer: Dioctyl phthalate (DOP) was obtained from Egyptian Company for plasticizers and solvents (10th of Ramadan).

iv. Co stabilizer: Epoxidized soya bean oil (ESBO) was obtained from hairma - china

v. Stabilizer: akropan 2611px (Ca/Zn stabilizer) was obtained from akdeniz kimya-turkey

vi. palmera Stearic acid was obtained from palm oleo - china

vii. Barium acetate was obtained from VEB LABORCHEM APOLDA (Germany)

viii. Calcium acetate was obtained from BDH chemicals LTD (England)

ix. Zinc acetate-2-hydrate was obtained from Arab lab company (U.A.E.)

x. Magnesium hydroxide, Calcium/Zinc phosphate, Ammonium polyphosphate and Melamine were obtained from Sigma Aldrich (Germany)

Methods of preparation

Double decomposition (precipitation) process was used for preparation of single and mixed metal stearate soaps.[17,18]

Preparation of calcium, barium and zinc stearates

These salts were prepared by two steps:

i. Preparation of sodium stearate by neutralization reaction of equimolar ratio of stearic acid and sodium hydroxide.

ii. The solution of the prepared sodium stearate was added gradually to aqueous solution of M-acetate (M= Zn, Ca or Ba), The prepared M-stearate was washed and purified with boiled distilled water several times and finally with methanol and after that was subjected to drying in an electric oven at 50°C under vacuum

Preparation of mixed Ca/Ba/Zn-stearate

Sodium stearate was prepared as mentioned above, and then Ca/Ba/Zn-stearate was prepared by addition of sodium stearate solution gradually to aqueous solution of Ba, Ca, and Zn-acetate mixture, the prepared Ca/Ba/Zn-stearate was purified as mentioned before

Experimental techniques

PVC compound formulations

Commercial stabilizer (Akropn 2611 px), prepared single and mixed metal stabilizers were added in different ratios to PVC compound formulation that contain the following recipe.

Thermal stabilizer	ESBO	DOP	Calcium carbonate	PVC K70
3 or 5	2	50	60	100

Other additives such as Magnesium hydroxide, melamine, ammonium poly phosphate and Ca/Zn phosphate were added with the prepared metal stearates in order to increase the stabilization efficiency. All formulations were mixed on Bara bender at 150°C and 80 r.p.m., and then were prepared on a two roll mill of diameter 470 mm and width 300 mm with speed of slow roll at 24 rev./min. A sheet of 1 cm width and 20 cm length was prepared and tested in Metrastat static heat stability oven model IR-700 at 200°C for 2 hours in normal air according to ASTM D-2115.

Characterization of the prepared single and mixed metal stearates

Micro analysis of carbon and hydrogen

Micro analysis of carbon and hydrogen for prepared metal stearates were carried out on vario EI Elementar apparatus (Germany). The results are illustrated in Table 1. It is quite clear that the data of micro analysis given agree to a large extent with the calculated values for the proposed structures.

Table 1 Micro analysis of carbon and hydrogen

Molecular formula	% Hydrogen		% Carbon	
	Theoretical	Found	Theoretical	Found
$(C_{18}H_{35}O_2)_2Ca.2H_2O$	11.6	10.3	67.25	66.7
$(C_{18}H_{35}O_2)_2Ba.2H_2O$	10.09	9	58.48	58.6
$(C_{18}H_{35}O_2)_2Zn.2H_2O$	11.16	8.8	64.7	64.5
$(C_{18}H_{35}O_2)_2Ca/Ba/Zn$	11.86	9.6	72.7	75.7

Determination of metal content by loss in ignition method

The metal stearate salts were ignited at 1000°C for 1 hr in a muffle until the all organic content were lost then the percent of metal was calculated from the remained metal oxide (Table 2).

Table 2 Metal content by loss in ignition method

Molecular formula	% Metal	
	Found	Theoretical
$(C_{18}H_{35}O_2)_2Ca.2H_2O$	6.51	6.59
$(C_{18}H_{35}O_2)_2Ba.2H_2O$	20.21	19.5
$(C_{18}H_{35}O_2)_2Zn.2H_2O$	12.36	10.34
$(C_{18}H_{35}O_2)_2Ca/Ba/Zn$	11.856	12.9

FT-IR analysis

IR spectra of the prepared metal stearates were obtained at a resolution of 4 cm^{-1}, between 4000 and 400 cm^{-1}, using a model FT-IR 6100. The IR spectra of the prepared Ca-stearate, Ba-stearate, Zn-stearate and Ca/Ba/Zn-stearate give a good confirmation of their structures as shown in Figure 1A-1D. The absorption bands at 3428, 3426, 3386 and 3430 cm^{-1} can be attributed to water bonded to crystal respectively, while bands at 2918, 2917, 2919 and 2918 cm^{-1} can be attributed to the bonded methyl group stretching, absorption bands

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at 2850, 2849, 2851 and 2850 cm^{-1} can be attributed to the bonded Methylene group stretching, the bonded carboxylic group anti symmetric vibration bands appeared at 1578, 1512, 1545 and 1574 cm^{-1}, while the bonded CH bending bands appeared at 1471, 1444, 14668 and 1468 cm^{-1}, absorption bands that appeared at 1112, 1110, 1045 and 1110 cm^{-1} can be attributed to the bonded C-O stretching of Carboxylate group, finally bands that appeared at 724, 717, 709 and 721 cm^{-1} can be attributed to the Carboxylate bending.

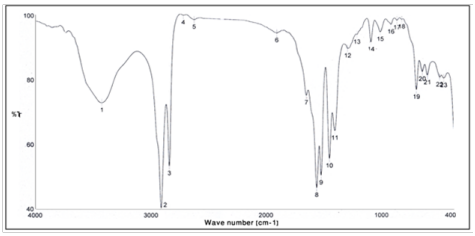

Figure 1(A) IR spectrum of calcium stearate.

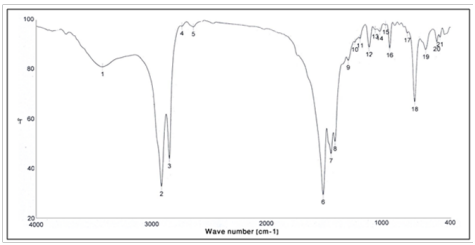

Figure 1(B) IR spectrum of barium stearate.

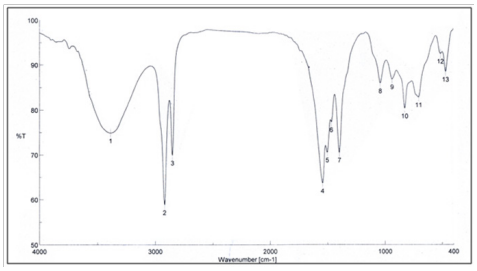

Figure 1(C) IR spectrum of zinc stearate.

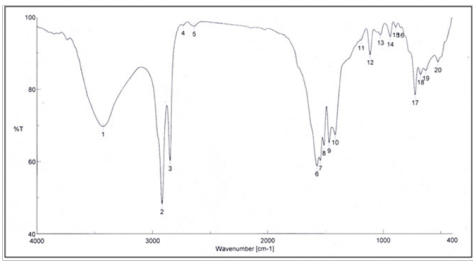

Figure 1(D) IR spectrum of calcium, barium, zinc stearate.

Thermal gravimetric analysis (TGA)

The thermal stability of the prepared metal stearates was investigated using TGA analysis under nitrogen gas at a heating rate of 10°C/min over a temperature range from room temperature up to 1000°C using TGA {Perkin-Elmer 7 series USA}, Thermal gravimetric results are illustrated in Figures 2A-2D respectively. From TGA results of the prepared metal stearates we can conclude that in the first stages the salts are nearly stable while weight loss was 19.34% at 300°C for calcium stearate, 7.67% at 315°C for barium stearate, 13.1% at 170°C for zinc stearate and 12.239 % at 310°C for Ca/Ba/Zn stearate and this may be attributed to loss of water molecules from prepared salts. Above 300°C a sharp loss due to stearate degradation was observed for all prepared metal stearates except zinc stearate where its sharp loss observed at 170°C and this mean that zinc stearate is lower stable than other salts. All organic content was lost at 500 C for all prepared metal stearates and the remained weight was a metal oxide

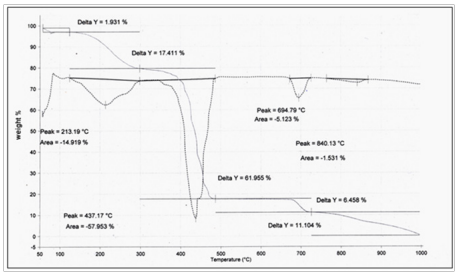

Figure 2(A) TGA of the prepared Ca-stearate.

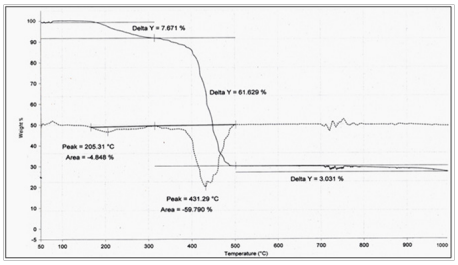

Figure 2(B) TGA of the prepared Ba-stearate.

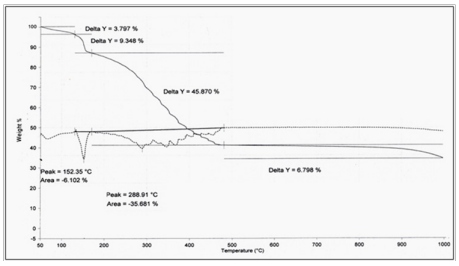

Figure 2(C) TGA of the prepared Zn-stearate.

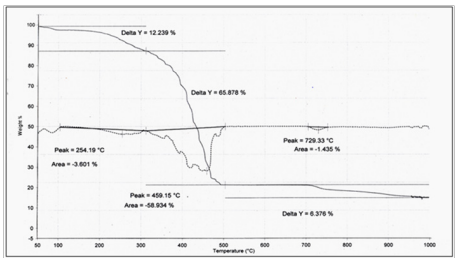

Figure 2(D) TGA of the prepared Ca, Ba, Zn-stearate.

X-ray diffraction (XRD) analysis

The results of XRD for the prepared (Ca, Ba, Zn and Ca/Ba/Zn-stearates) are illustrated in Figure 3A-3D respectively and Table 3. There is some agreement in the position and intensity of some of the principle phases of the prepared Ca, Ba, Zn and Ca/Ba/Zn-stearates with that of standard ICDD, but also some variation in other minor peak which may be due to other constituents of traces element's present.

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Table 3 D values dA0 and relative intensity I/I0 of X-ray diffraction patterns of prepared stearates

XRD of Ba/Ca/Zn-stearate		Standard ICDD of Ca- stearate card No. 25-1570		XRD of prepared Ca-stearate		Standard ICDD of Ba-stearate card No. 09-0848		XRD of prepared Ba-stearate		Standard ICDD of Zn-stearate card No. 05-0079		XRD of prepared Zn-stearate	
Relative intensity I/I_0	d(A^0)	Relative intensity I/I_0	d(A^0)	Relative intensity I/I_0	d(A^0)	Relative intensity I/I_0	d(A^0)	Relative intensity I/I_0	d(A^0)	Relative intensity I/I_0	d(A^0)	Relative intensity I/I_0	d(A^0)
-	-	100	30.9	-	-	-	-	-	-	-	-	-	-
86.4	21.364	-	-	-	-	-	-	-	-	-	-	-	-
74.1	15.12	5	15.4	100	15.84	28	15.9	100	15.34	-	-	-	-
100	14.57	-	-	-	-	-	-	-	-	-	-	-	-
31.3	10.99	5	10.8	-	-	-	-	-	-	13	10.5	19.8	10
24.5	9.37	10	24.8	50.29	24.12	13	9.57	25.68	9.15	-	-	-	-
46.3	8.84	-	-	-	-	-	-	-	-	27	8.34	33	8.046
20.9	7.41	-	-	-	-	-	-	-	-	-	-	-	-
48.1	4.505	40	4.41	44.73	4.37	17	4.58	31.57	4.58	100	4.53	36.4	4.507
47.8	4.29	25	4.3	22.11	4.104	-	-	-	-	-	-	-	-
62	4.17	-	-	-	-	7	4.18	-	-	-	-	-	-
46.8	4.027	-	-	-	-	7	4.02	5.73	4.09	-	-	-	-
41.6	3.846	-	-	-	-	8	3.87	10.86	3.91	67	3.92	46.9	3.88
58.5	3.707	-	-	-	-	7	3.68	-	-	-	-	-	-
-	-	-	-	20.78	9.47	12	11.8	21.13	11.46	100	13.28	100	13.8
-	-	-	-	26.77	5.821	5	4.46	5.3	4.41	3	6.36	1.6	6.435
-	-	-	-	12.35	3.87	-	-	-	-	7	2.26	7.1	2.37

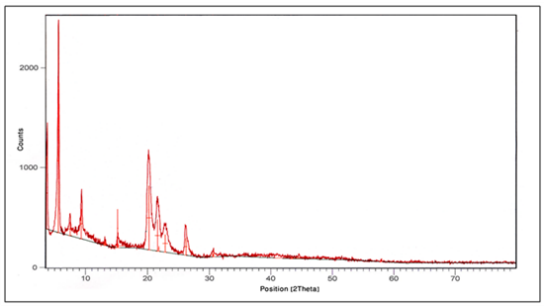

Figure 3(A) XRD patterns of the prepared Ca-stearate.

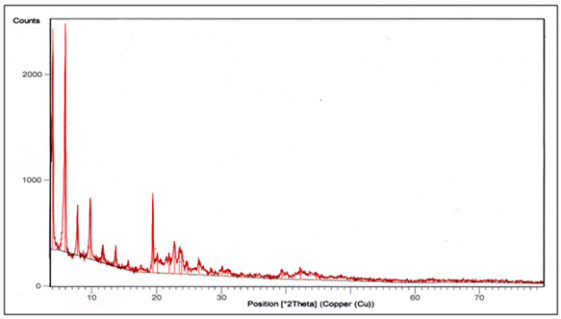

Figure 3(B) XRD patterns of the prepared Ba-stearate.

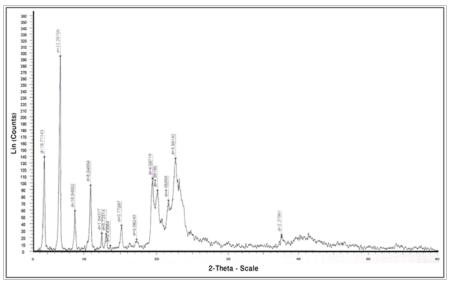

Figure 3(C) XRD patterns of the prepared Zn-stearate.

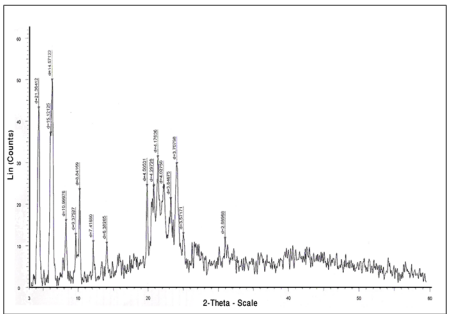

Figure 3(D) XRD patterns of the prepared Ca, Ba, Zn–stearate.

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Evaluation of the prepared metal stearates as thermal stabilizers for PVC

The prepared metal stearates were evaluated as thermal stabilizers for PVC by measuring Static thermal stability for the prepared formulations in metra stat oven model IR-700 at 200°C for 2 hr in normal air , the results are illustrated at Tables 4 & 5 and Figure 4A & 4B Poly (vinyl chloride) compositions degrade by discoloration on prolonged exposure to heat.

Table 4 Stability time of prepared stabilizers against commercial one akropan 2611 px 3phr

Thermal stabilizer	Stability time (minute)
akropan 2611 px (3phr)	40
Ca stearate (3phr)	15
Ba stearate (3phr)	20
Zn stearate (3phr)	0
Ca/Ba/Zn stearate (3phr)	52

Table 5 Stability time of prepared stabilizers against commercial one akropan 2611 px 5phr

Thermal stabilizer	stability time (minute)
akropan 2611 px (5 phr)	50
Ca/Ba/Zn stearate (5phr)	58
Ca/Ba/Zn stearate + Mg(OH)2 (4:1) phr	48
Ca/Ba/Zn stearate + ammonium poly phosphate (4:1) phr	42
Ca/Ba/Zn stearate + melamine (4:1) phr	40
Ca/Ba/Zn stearate + Ca/Zn phosphate (4:1) phr	30
Ca/Ba/Zn stearate + Ca/Zn phosphate (3:2) phr	48
Ca/Ba/Zn stearate + Mg(OH)2+ melamine (3:1:1) phr	45
Ca/Ba/Zn stearate + ammonium poly phosphate+ melamine (3:1:1) phr	48
Ca/Ba/Zn stearate + Mg(OH)2+ ammonium poly phosphate (3:1:1) phr	43

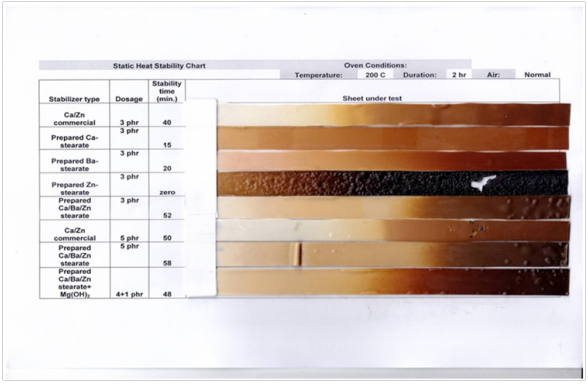


Figure 4(A) Thermal stability chart of PVC formulations 1.

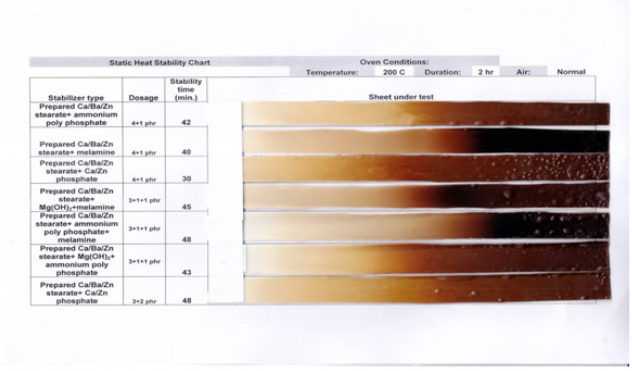


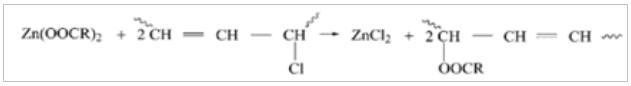
Figure 4(B) Thermal stability chart of PVC formulations 2.

The degree of discoloration is related to the condition of exposure, such as length of period and temperature. When the conditions of exposure are fixed and controlled, then the relative resistance to discoloration due to heat of two or more compositions is able to be determined. The precision of heat stability testing is also dependent on the thickness of the specimens and the history of heat exposure prior to testing. This practice allows for control or the reporting of these variables. This practice is particularly applicable for determining gross differences in the heat stabilities of poly (vinyl chloride) compositions that are detectable as a color change. It is not intended to measure absolute thermal stability. Although the observed color changes may be evidence of degradation, molecular degradation phenomena such as chain-scission or cross-linking may not be identifiable. While discoloration caused by exposure to elevated temperature is commonly regarded as evidence of degradation in poly (vinyl chloride) compositions, this practice is able to predict the relative discoloration in processing, provided that the compositions in question are tested at the relative maximum temperatures developed in processing. This practice is not applicable to materials that will cross-contaminate during oven exposure.

From Static thermal stability results of the prepared PVC formulations we can conclude that:

i. The prepared Ca-stearate and Ba-stearate enhanced thermal stability of PVC but in a medium extent and the effect of Ba-stearate is better than the effect of Ca-stearate and this behavior may be due to that Ba-stearate is more thermally stable than Ca-stearate. (This behavior can be observed by comparing the thermo grams of Ca & Ba stearates)

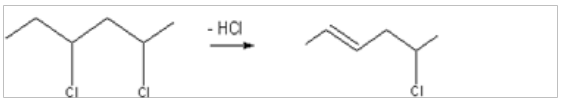
ii. The prepared Zn-stearate did not enhanced the thermal stability of PVC but increased the degradation of PVC, because Zinc stearate react with defect sites on PVC to displace the labile chlorine atoms



this reaction leads to formation of respective chloride salts, which can be very damaging to the PVC because $ZnCl_2$ is strong Lewis acid and catalyses the dehydrochlorination by forming a stable H^+ZnCl_3 complex. (This conclusion was reported by Manzoor W, et al).[19]

iii. Prepared Ca/Ba/Zn-stearate with 3 phr dose enhanced thermal stability of PVC to a good extent and is very better than Ca and

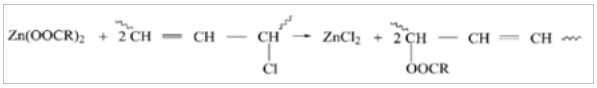
Ba stearates in long term stability. This effect was attributed to the synergistic effect of combining alkaline earth metal carboxylates (calcium and barium stearates) with covalent metal carboxylates (zinc stearate) on PVC [7, 10] De hydrochlorination of PVC during processing or exposure to heat take place according to the following equation



Evolved hydrochloric acid reacts with zinc stearate to produce zinc chloride according to the following equation

$$(C_{17}H_{35}COO)_2Zn + 2\,HCl \rightarrow 2\,C_{17}H_{35}COOH + ZnCl_2$$

Also zinc stearate reacts with PVC defect sites to produce zinc chloride.



Zinc chloride is strong Lewis acid and catalyses dehydrochlorination process, but in the presence of calcium and barium stearates zinc chloride undergo ester exchange reaction, thus regenerating the zinc stearate which may react with another molecule from hydrochloric acid or PVC (this conclusion was reported by H. Ismet Gokcel et al).[17]

$$ZnCl_2 + (C_{17}H_{35}COO)_2\,Ca \rightarrow (C_{17}H_{35}COO)_2\,Zn + CaCl_2$$

$$ZnCl_2 + (C_{17}H_{35}COO)_2\,Ba \rightarrow (C_{17}H_{35}COO)_2\,Zn + BaCl_2$$

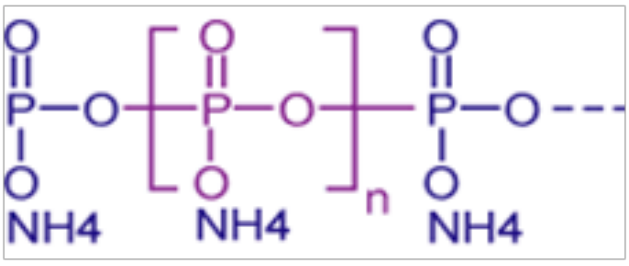
Calcium and barium chlorides are weak Lewis acids and would not catalyses' dehydrochlorination of PVC

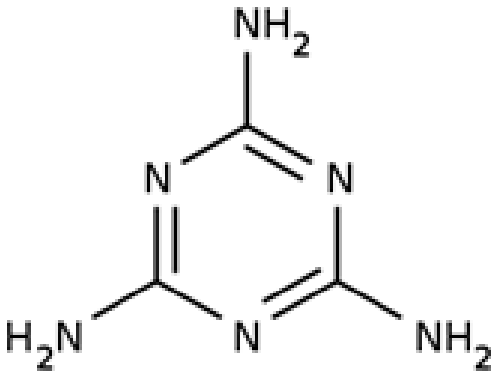
iv. Prepared Ca/Ba/Zn-stearate with 5 phr dose is better than commercial Ca/Zn stabilizer in long term thermal stabilization of PVC but the commercial Ca/Zn is better than prepared one in enhancing the initial color of the tested PVC formulation

v. Addition of magnesium hydroxide with the prepared Ca/Ba/Zn enhance the initial color of the tested PVC formulation and this behavior can be attributed to the formation of weak magnesium chloride Lewis acid during the reaction of evolved hydrochloric acid with magnesium hydroxide

$$Mg(OH)_2 + 2HCl \rightarrow MgCl_2 + 2H_2O$$

vi. Addition of ammonium poly phosphate with the prepared Ca/Ba/Zn enhance the initial color of the tested PVC formulation and this behavior can be attributed to the reaction of evolved hydrochloric acid with ammonium phosphate unites





vii. Addition of melamine with the prepared Ca/Ba/Zn enhance the initial color of the tested PVC formulation and this behavior can be attributed to the reaction of evolved hydrochloric acid with amino groups in melamine

viii. Addition of Ca/Zn phosphate with the prepared Ca/Ba/Zn enhance the initial color of the tested PVC formulation but the addition of 2 phr from Ca/Zn phosphate with 3phr from the prepared Ca/Ba/Zn enhance the initial color of tested PVC formulation more than the 1 to 4 phr and this behavior can be attributed to the synergistic effect of combining calcium with zinc in Ca/Zn phosphate.

ix. Mixing of melamine with ammonium poly phosphate or magnesium hydroxide and magnesium hydroxide with ammonium poly phosphate enhance the initial color better than single addition.

Conclusion

i. Mixed metal stearate is better than single metal stearate as thermal stabilizers for PVC

ii. Zinc stearate did not enhanced thermal stability of PVC but increased the degradation of it

iii. Ba-stearate is better than Ca-stearate in thermal stabilization of PVC

iv. By carefully choosing the percent of zinc ion and the other metal ion in the mixed metal stearates, a good thermal stability can be obtained

v. Prepared Ca/Ba/Zn-stearate is better than commercial Ca/Zn (akropan 2611px) stabilizer in long term thermal stabilization of PVC not in holding the initial color

vi. Increasing the dose of prepared Ca/Ba/Zn-stearate enhanced the thermal stability of PVC

vii. Addition of some substances that can absorb the evolved hydrochloric acid during dehydrochlorination process such as magnesium hydroxide, ammonium poly phosphate, melamine and Ca/Zn phosphate with the prepared Ca/Ba/Zn stearate enhance the holding of initial color of thermally tested PVC, also these substances have fire retardant properties and may be increase the fire resistance of the prepared PVC formulation (Figure 5A, 5B).

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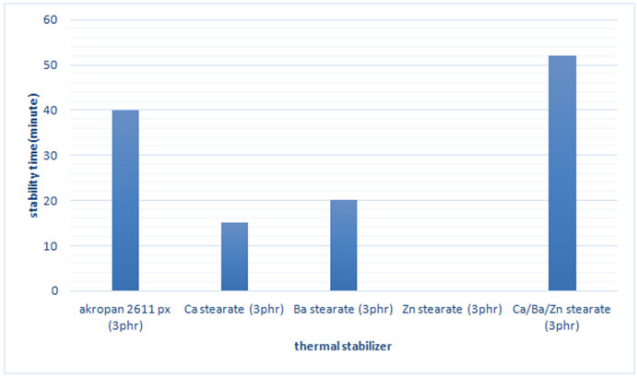

Figure 5(A) Stability time of prepared stabilizers against commercial one (akropan 2611 px) (3phr).

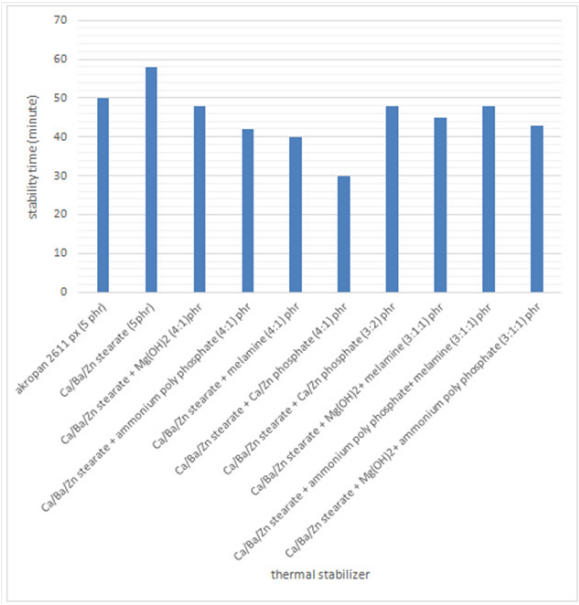

Figure 5(B) Stability time of prepared stabilizers against commercial one (akropan 2611 px) (5phr).

Acknowledgments

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Conflicts of interest

The author declares that there are no conflicts of interest.

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