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## Zinc stearate from galvanizing waste materials and its use as thermal stabilizer in PVC industries

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### Abstract

Galvanizing industries of Bangladesh produce profuse amount of environmentally hazardous solid waste materials like zinc dross which contains significant amount of valuable zinc and harmful heavy lead. Zinc was extracted as zinc chloride ( $ZnCl_2$ ) from zinc dross. Zinc stearate ( $ZnSt_2$ ) samples were prepared by precipitation method from stearic acid, sodium hydroxide and  $ZnCl_2$  by varying the amount of the reagents and product yield found within the range 96.06-99.18%. Characteristic peaks of  $ZnSt_2$  were investigated by Fourier Transform Infrared Spectroscopy (FTIR). Differential Scanning Calorimeter (DSC) onset curve assigned accurate melting point within the range 122.84-124.03°C. Surface morphology of  $ZnSt_2$  was observed by Scanning Electron Microscope (SEM) and products had semi-crystalline structure. Thermal stability of  $ZnSt_2$  was evaluated by Thermo-gravimetric Analyzer (TGA) that complied with literature. A combination of  $ZnSt_2$  and Calcium stearate ( $CaSt_2$ ) at 1:1 ratio was used as thermal stabilizer in the powder commercial grade PVC resin and performed better thermal stability. The dehydrochlorination temperature of PVC with mixed stearates was 344.67±1.04°C for 10% (w/w) loading whereas for PVC, PVC with 10% (w/w)  $ZnSt_2$  and PVC with 10% (w/w)  $CaSt_2$ , it was 269.83±1.04°C, 317.33±1.26°C and 323.33±2.08°C respectively.

**Keywords:** Zinc dross; Zinc stearate; PVC; Thermal stabilizer; Dehydrochlorination

### Introduction

Galvanization is a process of applying a protective thin layer of zinc coating to steel or iron in order to prevent corrosion and increase the attractiveness of commodity. In hot-dip galvanizing processes lead is used to reduce the surface tension of zinc, improve the fluidity of the bath, enhance drainage properties and thus limit excess zinc on the dipped product (Asgari *et al.*, 2007). The galvanizing industries are produced some wastes such as zinc dross which contains significant amount of heavy metals and also has tremendous health and environmental risk for human, animal and plant.

Zinc exhibits both the deficient or toxic effect on animal and plant. Zinc diet deficiency causes detrimental impact on growth, neuronal development and immunity but overdose zinc affects on health (Plum *et al.*, 2010).

Inhalation of zinc containing smoke, generally originated from galvanization industry, affects respiratory system like chest pain, cough, bronchiolar leukocytes and dyspnea (Prasad, 2008). Zinc can interrupt the activity in soil as it negatively influences the micro-organisms and earthworms. It also slows down the breakdown of organic matter and increase the pH of water. On the other hand, heavy metal lead inimical affect almost all organs and systems in human body including the heart, bones, intestines, kidneys, nervous, immune and reproductive systems (Rossi, 2008). Recycling or recovery of valuable materials from the zinc dross waste is economically rewarding and also minimizing environmental pollutions.

Zinc chloride is produced by hydrochloric acid (HCl) leaching from zinc dross. In presence of hot concentrated HCl,

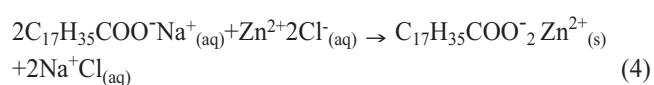
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both zinc and lead form their chlorides (reaction 1 and 2). The solubility product of  $\text{ZnCl}_2$  is  $127 \text{ mol}^3\text{L}^{-3}$  and  $\text{PbCl}_2$  is  $1.6 \times 10^{-35} \text{ mol}^3\text{L}^{-3}$  at  $25^\circ\text{C}$  (Hahne and Kroontje, 1973). As the solubility of  $\text{ZnCl}_2$  is million times greater than lead chloride,  $\text{PbCl}_2$  precipitates easily at room temperature. The  $\text{Fe}^{2+}$  can be oxidized to  $\text{Fe}^{3+}$  with oxidizing agent and easily be separated as  $\text{Fe}(\text{OH})_3$ . The residual solution is the aqueous solution of zinc chloride which can be dried or directly used for the preparation of  $\text{ZnSt}_2$ .



Zinc stearate [ $\text{Zn}(\text{C}_{18}\text{H}_{35}\text{O}_2)_2$ ] is one of the important hydrophobic ionic surfactants of metal soaps that is commercially important and finds applications in areas such as thermal stabilizer specially for polyvinyl chloride (PVC), synergic stabilizer for Ba/Cd and Pb stabilizer systems, gloss imparting agent in paint industry, metal release agent in rubber, polyurethane and polyester processing system, die release agent in powder metallurgy, lubricant in cosmetics to improve texture, waterproofing agents for plastics, acid scavenger and processing aid for certain polyolefin applications etc. (Akanni *et al.*, 1992; Fang, 2009; Rahman *et al.*, 2011).

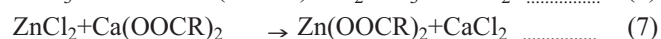
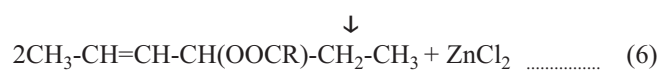
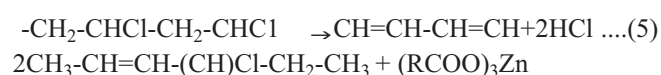
$\text{ZnSt}_2$  soap is manufactured by using one of the three processes: double decomposition (precipitation process, reaction 3 and 4), direct reaction of carboxylic acid with metal oxides, hydroxides or carbonates and direct reaction of metals with molten fatty acids (Gönen *et al.*, 2005). In precipitation process, the fatty acid is first saponified in relatively large volume of heated water with an equimolar to a slight excess quantity of a strong alkali solution (NaOH) to form water soluble alkali stearate soap (reaction 3). The desired water insoluble  $\text{ZnSt}_2$  is subsequently produced by adding metal salt solution ( $\text{ZnCl}_2$ ) at a temperature below the boiling point of water under atmospheric pressure (equation 4).



Poly(vinyl chloride), PVC, is one of the common commodity plastic, and its production is the third largest, after polyethylene and polypropylene (Yoshioka *et al.*, 2008). It is cost-effective, highly versatile and is used in many construction applications as water, sewage and drainage pipes, and a variety of extruded profiles (van Es *et al.*, 2008). Though PVC is one of the most important commercial plastic materials but it is thermally unstable at processing temperature. Until the discovery of thermal stabilizers, PVC was not an industrially very useful polymer, as it could not be processed to useful articles without degradation at elevated temperatures (Folarin and Sadiku, 2011).

Thermal degradation of PVC occurs by autocatalytic dehydrochlorination reaction (Zipper reaction, reaction 5) with the subsequent formation of conjugated double bonds (Moulay, 2010; Sánchez-Jiménez, *et al.*, 2010; Šimon, 1992). After the loss of first HCl molecules, the subsequent unsaturated structure formed in a PVC chain is an allylic chlorine structure which stimulates the next loss of HCl molecule. The repeated process leads to the chain or zip dehydrochlorination. The process starts at the glass transition temperature ( $70^\circ\text{C}$ ) by elimination of HCl which is the main volatile product upto  $300^\circ\text{C}$  (Klaric *et al.*, 2012). The elimination of HCl from polymer backbone leads discoloration of polymer polyene sequences of 5 to 25 double bonds (Tahira *et al.*, 2014). Depending on the number of conjugated double bonds formed, it becomes yellow, orange, red, brown and finally black (Sabaa and Mohamed, 2007). The splitting off of HCl from the polymer backbone affects the physical, chemical and the mechanical properties of the PVC. It is well established that  $\text{ZnSt}_2$  react with the allylic chlorine atoms (reaction 6) which leads the PVC esterification (Chen *et al.*, 2014). Thus, the by-product of the stabilization reaction is  $\text{ZnCl}_2$  which is strong Lewis acids and is powerful catalyst of the substitution reaction as well as the dehydrochlorination. It's accumulation in the polymer matrix has a detrimental effect on PVC thermal stability (Chen *et al.*, 2014; Hoang *et al.*, 1976; Michel and Van Hoang, 1981). On the other hand, the previous stabilization reaction through the O-alkylation is not permanent because the unsaturated ester is able to react with HCl even at moderate temperature to generate again allylic chlorine atoms and carboxylic acid (Michel and Van Hoang, 1981). Calcium stearate ( $\text{CaSt}_2$ ) not only absorbs HCl but also exchanges

ions with  $ZnCl_2$  (Xu *et al.*, 2014). In the ion exchange reaction, the active  $ZnSt_2$  is generated and the undesirable  $ZnCl_2$  is consumed (reaction 7). The accumulation of  $CaCl_2$  in the polymer matrix has no adverse effect on the blackening and the cross-linking of the polymer. In consequences, the main role of the  $CaSt_2$  is delay the accumulation of  $ZnCl_2$  in the polymer matrix and to serve as a reserve to increase the lifetime of the  $ZnSt_2$  (Hoang *et al.*, 1976).



The purpose of the research reported here was to extract economically valuable zinc and lead from galvanizing waste and to minimize the environmental pollutions. This study addresses the preparation and characterization of  $ZnSt_2$  by using extracted  $ZnCl_2$  and its potential application as thermal stabilizer of PVC resin.

## Materials and methods

### Materials

The zinc dross was collected from a galvanizing plant located at Chittagong, Bangladesh. The zinc dross contained 66.18% lead, 23.56% zinc and other insoluble impurities. Analytical grade stearic acid (Poole, England), hydrochloric acid (Merk, Germany), sodium hydroxide (Poole, England), hydrogen peroxide (Active fine chemicals, Bangladesh) were used in this research. The commercial grade PVC (LG Chem (Taiwan) Ltd) was collected from local market. Mineral (ash) content and volatile content (in house oven dried at 105°C) of PVC were 8.41% and 0.045% respectively.

### Methods

#### Production of $ZnCl_2$ and $PbCl_2$

The zinc dross (10 g) was leached in HCl for 10 hrs at 100 °C temperature. The leached liquor was filtered at hot condition and concentrated to such a point that crystallization began on cooling. It was then filtered to separate  $PbCl_2$ . The pH of the solution was kept between 3.9-4.0 by adding ammonium hydroxide and HCl. The solution was diluted 100 ml and heated nearly to boil. Then the solution boiled two minutes with 0.4 ml  $H_2O_2$  (30%) and followed by freezing in refrigerator for 3 days. Iron was precipitated as  $Fe(OH)_3$  and  $ZnCl_2$  solution was obtained after filtration.  $ZnCl_2$  was investigated by atomic absorption spectroscopy (AAS) (AAnalyst 800, Perkin Elmer).

#### Preparation of $ZnSt_2$

Eight batches of  $ZnSt_2$  were produced with reagents variation by precipitation process (Table I). The Batch 1 was produced by using analytical grade  $ZnCl_2$  and other seven batches (Batch 2 to Batch 8) were synthesized from extracted  $ZnCl_2$ . The stearic acid (5 g) was added in 200 ml preheated water in a beaker and stirred on a hot plate magnetic stirrer at 60-70 °C temperature and 120 rpm. Then, NaOH solution was slowly mixed with melted stearic acid and heated until clear NaSt was produced. Zinc chloride solution was slowly added in the NaSt solution and heated at 80-90 °C for 30 mins to produce  $ZnSt_2$ . The final  $ZnSt_2$  solution was filtered and washed with water. The wet  $ZnSt_2$  was dried and stored.

#### $ZnSt_2$ characterization

A Fourier transform infrared (FTIR) spectrophotometer (Model: IRPrestige-21, Shimadzu Corporation, Japan) was used to investigate the characteristics functional group peak of  $ZnSt_2$ . Approximately 2 mg of dried  $ZnSt_2$  sample and

**Table I.  $ZnSt_2$  production with reagents variation**

Reagent	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Batch 6	Batch 7	Batch 8
Stearic acid	equimolar	equimolar	10% excess	5% excess	equimolar	equimolar	equimolar	equimolar
$ZnCl_2$	equimolar	equimolar	equimolar	equimolar	10% excess	5% excess	equimolar	equimolar
NaOH	equimolar	equimolar	equimolar	equimolar	equimolar	equimolar	10% excess	5% excess

200 mg of potassium bromide (KBr) were blended and triturated with an agate mortar. The mixture was compacted using an IR hydroaulic press at a pressure of 6 tons for 60 s. The spectra of ZnSt<sub>2</sub> samples (in the form of KBr discs) were obtained using the spectrophotometer with a wave number no range of 4000-400 cm<sup>-1</sup>. The melting point of ZnSt<sub>2</sub> was measured in melting point apparatus (Model- WRS-1B, Sunon Wealth Elec. Mech. Ind. Co. Ltd). The sample (1-2 mg) was taken in a capillary tube and placed in the apparatus. The melting point was observed with increasing temperature 1 °C/min.

#### *Morphological and elemental analysis of ZnSt<sub>2</sub> using SEM-EDX*

The particle morphology and elemental composition of ZnSt<sub>2</sub> were investigated by using a high-resolution SEM-EDX (Model No. JOEL JSM-6490 LA, Joel Ltd. Japan) instrument at 3.0 nm and an accelerating voltage of 10 kV.

#### *TG and DSC analysis of ZnSt<sub>2</sub> and PVC*

A thermo-gravimetric analyzer (TGA-50, Shimadzu, Japan) was used to determine the thermal behavior of ZnSt<sub>2</sub>, PVC and ZnSt<sub>2</sub> loaded PVC. The sample (8-10 mg) was loaded in aluminum pan and heated from 30 to 600 °C at a heating rate of 10 °C per min. under 10 ml/min nitrogen gas flow. A Differential Scanning Calorimeter (DSC) (Model: DSC-60, Shimadzu Corp., Japan) was used to determine accurate melting point and phase transition of ZnSt<sub>2</sub>. The sample (2-4 mg) was taken in aluminum pan and pellet was formed under pressure and heated from 30 to 200 °C at a heating rate of 10 °C per min. under 20 ml/min nitrogen gas flow.

#### *Dehydrochlorination of PVC*

The performance of stabilizer (ZnSt<sub>2</sub> and CaSt<sub>2</sub>) in PVC was estimated by measuring dehydrochlorination temperature of PVC and thermogravimetric analysis. Test samples were prepared by adding metal stearate into powder PVC and mixed properly by an extruder (LME230, Alpha Group, USA). The dehydrochlorination temperature of PVC was measured by digital melting point apparatus (WRS-1B, supplier- Sunon Wealth Elec. Mech. Ind. Co. Ltd). Test samples (1-2 mg) were taken in a capillary tube. A strip of universal indicator

paper was placed on open part of the capillary tube. One drop of distilled water was used to wet the indicator paper which confirmed the sealing of the tube. The dehydrochlorination temperature was observed with initial temperature set up at 140 °C and 3 °C/min rise up.

## **Results and discussion**

#### *Analysis of ZnSt<sub>2</sub>*

The acid leached 100 g zinc dross produced 49.80 g PbCl<sub>2</sub> and 26.70 g ZnCl<sub>2</sub> and about 37.75 g remained unreacted. Atomic absorption spectroscopy analysis of aqueous ZnCl<sub>2</sub> confirmed the absence of iron (detection limit 0.02 ppm) and lead (detection limit 0.5 ppm). This conformation also verified by EDX elemental analysis of ZnSt<sub>2</sub>.

#### *Analysis of ZnSt<sub>2</sub>*

The characteristic peaks of ZnSt<sub>2</sub> at 1540 and 1398 cm<sup>-1</sup> were observed in the FTIR spectrum. These bands are due to antisymmetric and symmetric carboxylate stretching bands (COO<sup>-</sup> and COO<sup>-</sup>) respectively (Sakai & Umemura, 2002). The coordination of oxygen atom to zinc atom was in chelating form because the IR peak at 1540 cm<sup>-1</sup> was observed instead of IR peak at 1576 cm<sup>-1</sup> (Benavides *et al.*, 1994). Antisymmetric and symmetric methylene stretching, and methylene scissoring bands (CH<sub>2</sub>, CH<sub>2</sub>, and CH<sub>2</sub>) were observed at about 2916, 2846, and 1458 cm<sup>-1</sup> respectively (Gönen *et al.*, 2005). These bands are due to the alkyl chain in the ZnSt<sub>2</sub> structure. The absence of asymmetric stretching vibration of COO<sup>-</sup> (Carboxylate of NaSt<sub>2</sub>) around 1560cm<sup>-1</sup> indicates that all NaSt<sub>2</sub> was converted into ZnSt<sub>2</sub>. On the other hand, absence of C=O stretching around 1700 cm<sup>-1</sup> revealed that product was free from stearic acid.

#### *Effects of reagents on elemental composition of ZnSt<sub>2</sub>*

EDX elemental compositions of ZnSt<sub>2</sub> were performed at three different points of the sample and average values of these results are reported in Table II. From the table, it is revealed that the composition of ZnSt<sub>2</sub> produced by analytical grade ZnCl<sub>2</sub> (Batch 1) and extracted ZnCl<sub>2</sub> (Batch 2) show similar results which assign the purity of extracted

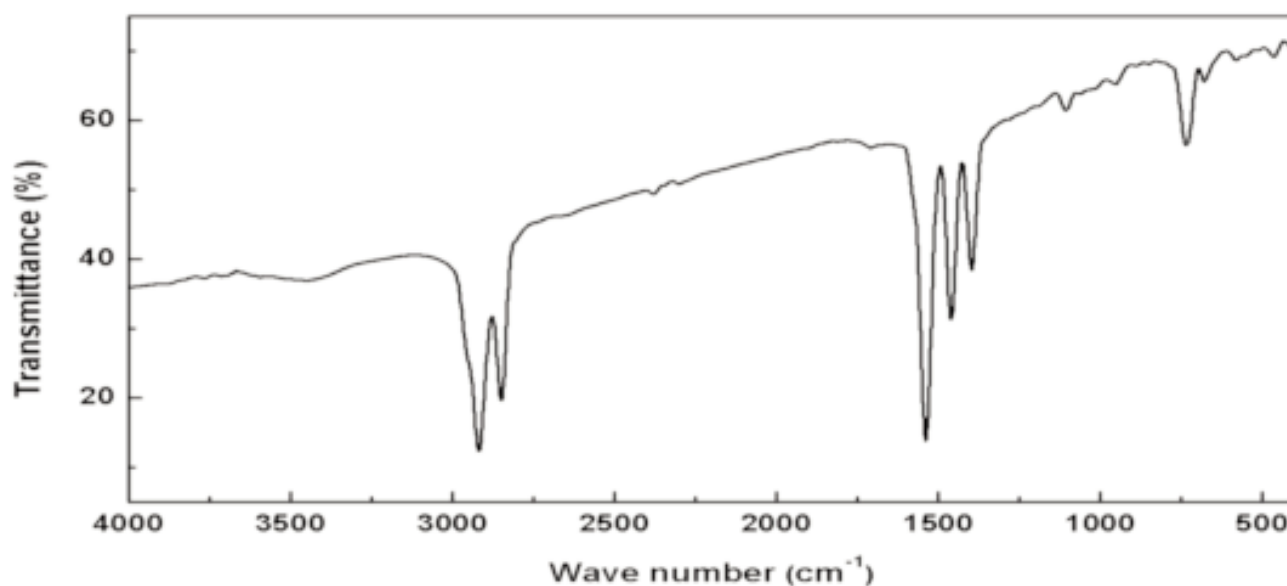


Fig. 1. FTIR analysis of ZnSt<sub>2</sub> (Batch 2)

ZnCl<sub>2</sub>. All ZnSt<sub>2</sub> products have higher zinc content than theoretical value owing to the formation of coordination of O with Zn<sup>2+</sup> ion rather than ionic form. The molar ratio of oxygen atom to zinc atom of ZnSt<sub>2</sub> is lower than the theoretical value confirmed the coordination of O atoms with Zn<sup>2+</sup> ion which is consistent with FTIR observation.

higher than equivalent ratio because maximum conversion of limiting reagent was performed here. Maximum yield were found in Batch 3 and 4 because almost all stearic acid was combined with zinc. The melting points of ZnSt<sub>2</sub> were lower than reported value in the literature assigned the presence of other fatty acid soaps in ZnSt<sub>2</sub>.

Table II. EDX elemental analysis of ZnSt<sub>2</sub>

Elements	Theoretical value	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Batch 6	Batch 7	Batch 8
C	68.43	69.10±1.94	69.11±2.15	68.62±0.99	68.54±1.22	67.63±1.46	67.74±0.90	69.41±2.21	69.39±1.14
O	10.12	8.75±0.66	8.67±1.07	9.47±1.27	9.42±1.55	8.49±1.14	8.56±1.09	8.15±1.09	8.11±1.12
Zn	10.34	11.6±1.29	11.13±1.39	10.82±0.30	10.95±0.59	12.79±1.27	12.61±1.52	11.35±1.12	11.41±1.46
H (Theoretical value)	11.09	11.09	11.09	11.09	11.09	11.09	11.09	11.09	11.09
Molar ratio of O to Zn atom	4	2.96	2.92	3.28	3.23	2.49	2.55	2.69	2.66

#### Effects of reagents on yield and phase transition temperature of ZnSt<sub>2</sub>

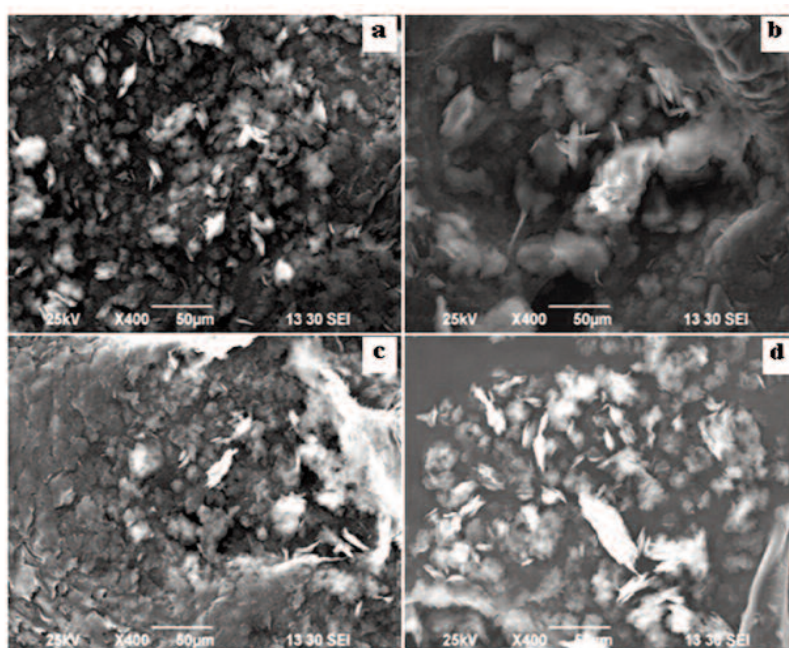
The yield percentages and phase transition temperature (melting point) of different ZnSt<sub>2</sub> products are reported in the Table III. The yield percentages for excess reagents were

#### Morphological analysis of ZnSt<sub>2</sub>

The crystal morphology of ZnSt<sub>2</sub> samples are presented in Fig. 2. From the figure it is clear that the particles of batch 3 product (ZnSt<sub>2</sub> produced from excess stearic acid) are large but the crystal is less sharp than the crystals of the other prod-

**Table III. Effect of reagents on melting point and yield percentage of ZnSt<sub>2</sub>**

Name of the sample	Melting point from melting point apparatus (°C)	Melting point from DSC thermogram (°C)	Limiting reagent for yield (%) calculation	Yield (%)
Batch 2	119-121	123.24	Stearic acid	96.06
Batch 3	116-120	122.84	ZnCl <sub>2</sub>	99.18
Batch 4	119-123	-	ZnCl <sub>2</sub>	98.08
Batch 5	119-123	124.03	Stearic acid	98.32
Batch 6	118-122	-	Stearic acid	97.85
Batch 7	118-122	123.24	Stearic acid	98.56
Batch 8	118-122	-	Stearic acid	98.34

**Fig. 2. SEM micrograph of ZnSt<sub>2</sub> products (a) Batch 2 (b) Batch 3, (c) Batch 5 and (d) Batch 7**

ucts of ZnSt<sub>2</sub> due to presence of unreacted stearic acid. The presence of excess stearic acid in reactor causes large agglomerated crystal growth. These observations are consistent with literature (Gönen *et al.*, 2005). The products obtained from excess ZnCl<sub>2</sub> (Batch 5) and excess NaOH (Batch 7) showed smaller crystal than batch 3 product but larger than equivalent ratio (Batch 2) product. The particles of equivalent ZnSt<sub>2</sub> (batch 2) were more orderly packed and its crystal had sharper edges than other products.

#### Thermal analysis of ZnSt<sub>2</sub>

The DSC thermograms of ZnSt<sub>2</sub> are plotted in the Fig. 3. All products had a large sharp endothermic peak in the DSC thermogram owing to the melting of ZnSt<sub>2</sub>. The melting tran

sition peak maxima are reported in the Table III which are more accurate and exact melting point. The melting point from DSC curve concluded that the product of all condition is almost pure form.

The TG thermogram of ZnSt<sub>2</sub>, PVC and ZnSt<sub>2</sub> loaded PVC are shown in the Fig. 4 within the temperature range of 40 to 500 °C. From the TG thermogram, the remaining mass of ZnSt<sub>2</sub> above 450 °C is 12.2% whereas above this temperature no weight loss occurred. The ZnSt<sub>2</sub> started to lose weight around 200°C at which degradation starts. The remaining mass of ZnSt<sub>2</sub> at the end of thermal treatment could be oxide and carbonate of zinc metal (Akanni *et al.*, 1992). The major decomposition products of ZnSt<sub>2</sub> might be carbon dioxide, alkanes, and ketones as gas products (Artok and Schobert, 2000).

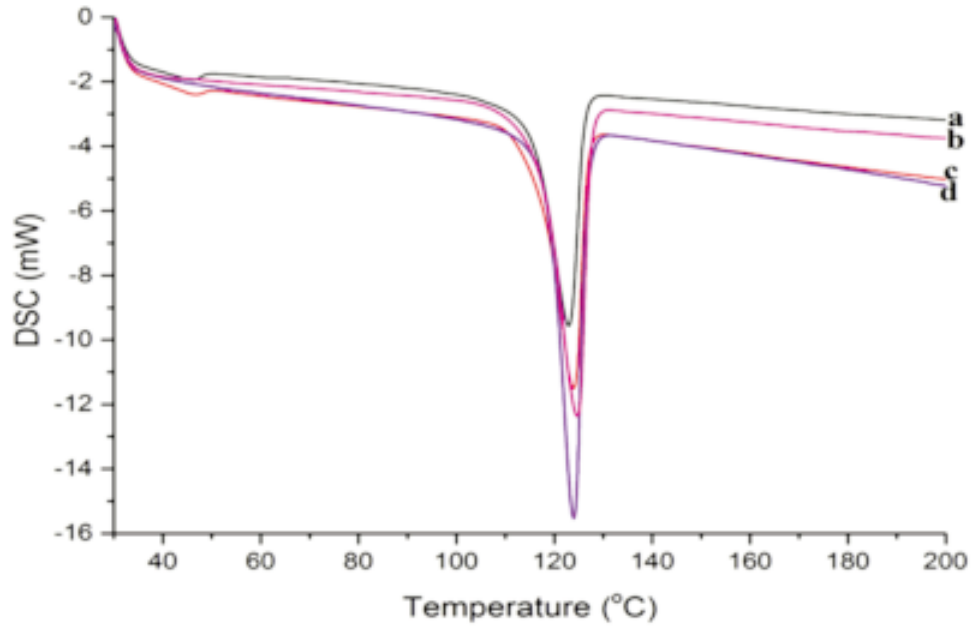


Fig. 3. DSC thermogram of ZnSt<sub>2</sub> (a) Batch 2 (b) Batch 3 (c) Batch 5 and (d) Batch 7

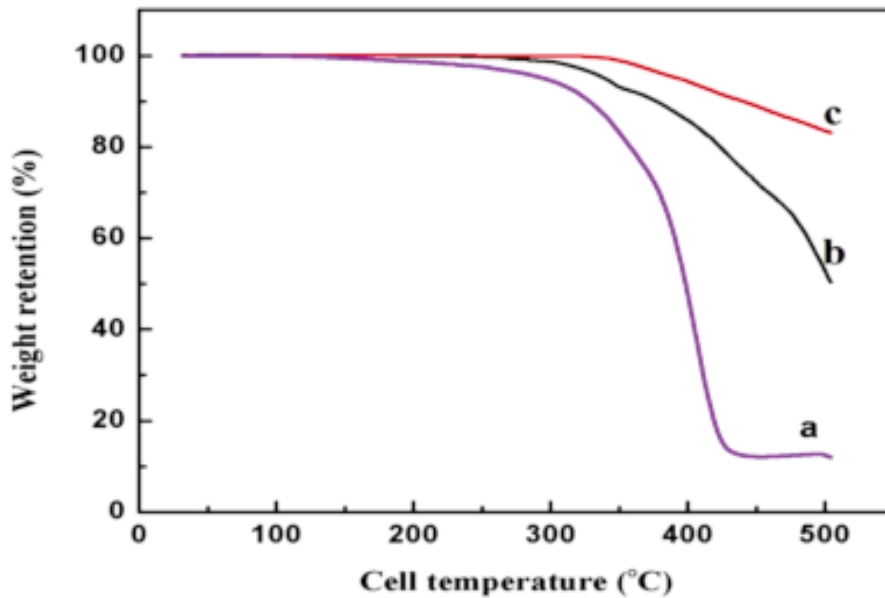


Fig. 4. TGA thermogram of (a) ZnSt<sub>2</sub> (b) PVC and (c) PVC+ 10% ZnSt<sub>2</sub>

#### Analysis of PVC thermal stabilizer

The dehydrochlorination temperature of ZnSt<sub>2</sub> and CaSt<sub>2</sub> loaded PVC are plotted in Fig. 5 (a). It was observed that incorporation of both metal stearates increases the dehy-

drochlorination temperature of PVC resin and with the addition of 10% ZnSt<sub>2</sub> and CaSt<sub>2</sub> it was increased from  $269.83 \pm 1.04$  °C to  $317.33 \pm 1.26$  °C and  $323.33 \pm 2.08$  °C respectively. Above 10% both stearates loading had slight effect on the dehydrochlorination temperature and are not

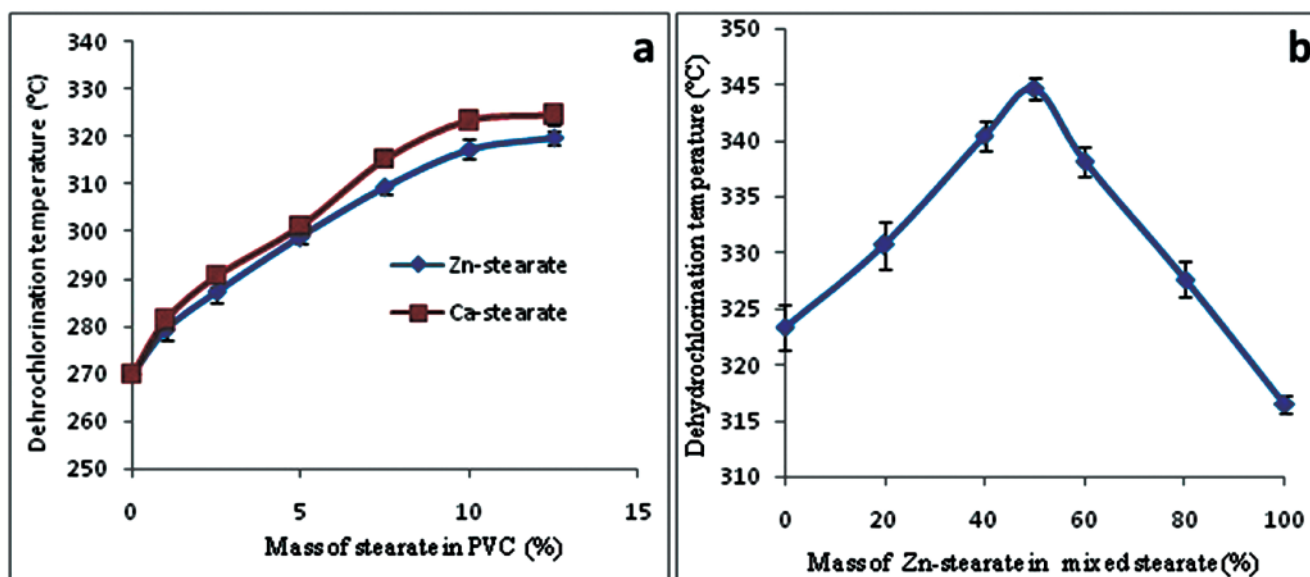


Fig. 5. Dehydrochlorination temperature of PVC after loading (a)  $\text{ZnSt}_2$  and  $\text{CaSt}_2$  and (b) mixed stearate

economically feasible. The thermogravimetric analysis of PVC and PVC with  $\text{ZnSt}_2$  revealed the similar result which is showed in above Fig. 4. From TG thermogram (Fig. 4 b), it was observed that first weight loss of unstabilized PVC was found above 271 °C. This weight loss assigned the dehydrochlorination of PVC. On the other hand, the initial degradation temperature of stabilized PVC shifted to above 319 °C because of incorporation of 10%  $\text{ZnSt}_2$ . The combined  $\text{ZnSt}_2$  and  $\text{CaSt}_2$  was more effective thermal stabilizer than individual and plotted in the Fig. 5 (b) for 10% (w/w) mixed stearate loading. This zinc chloride was consumed by calcium chloride and regenerated  $\text{ZnSt}_2$ . The dehydrochlorination temperature of 10% (w/w) mixed stearates (50%  $\text{ZnSt}_2$  + 50%  $\text{CaSt}_2$ ) mixture was observed  $344.67 \pm 1.04$  °C.

### Conclusion

It could be concluded that acid leaching was the chemically and economically feasible process for extraction of zinc and lead from zinc dross. The extracted aqueous  $\text{ZnCl}_2$  was highly pure which could be directly used for the production of  $\text{ZnSt}_2$ . The high yield percentages of  $\text{ZnSt}_2$  confirmed the fast and complete reaction rate. The variation of reagents had slightly affected on particle morphology, elemental composition of  $\text{ZnSt}_2$ , yield, thermal response and stabilizing effi-

ciency of  $\text{ZnSt}_2$  but best quality  $\text{ZnSt}_2$  was obtained by equivalent reagents ratio. Both  $\text{ZnSt}_2$  and  $\text{CaSt}_2$  performed excellent thermal stabilizer for PVC. Upto 10% metal stearates or their mixture would performed optimum thermal stabilizer and above this percentage were not economically feasible.

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