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Thermal Degradation Kinetics of Polyvinyl Chloride Stabilized with Palm Based Mixed Metal Carboxylates

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Abstract. Despite its diverse applications, especially as building materials, polyvinyl chloride (PVC) is easily degraded by heat. Thermal degradation poses a significant problem during extrusion, with the worst case being that the PVC resins cannot be formed into final products. Additionally, the degradation process releases hydrogen chloride, which is harmful to the environment. Mixed metal (Ca/Zn) carboxylates are popular PVC thermal stabilizers, offering a more environmentally friendly alternative to lead salts and a more cost-effective option than organotin, another commonly used industrial thermal stabilizer. However, conventional mixed Ca/Zn thermal stabilizers, typically based on stearic acid, remains relatively costly for PVC compounders in developing countries. Recently, we have developed a Ca/Zn thermal stabilizer from palm fatty acid distillate (PFAD), referred to as Ca/Zn Palmate. This thermal stabilizer is as effective as the conventional stearic-acid-based Ca/Zn thermal stabilizer but significantly more affordable, owing to the lower cost of PFAD. Continuing this research, we studied the thermal degradation kinetics of PVC resin stabilized with the developed palm-based mixed Ca/Zn thermal stabilizer. Degradation data were collected from dehydrochlorination tests carried out within the 170-190°C temperature range, with varying doses of Ca/Zn Palmate, Ca/Zn ratios, and co-stabilizer amounts. The degradation followed the A2 model of Avrami-Erofeev. The activation energy for unstabilized PVC was found to be 124.4 kJ/mol, while for stabilized PVC, it ranged from 128.8 to 167.2 kJ/mol. The results showed that increasing the Ca/Zn Palmate dose or the Ca/Zn ratio led to higher activation energy. The highest activation energy occurred when the co-stabilizer doses were equivalent to the dose of Zn Palmate.

Keywords: Activation Energy, Avrami-Erofeev Model, Ca/Zn Thermal Stabilizer, Dehydrochlorination, Dibenzoylmethane, Pentaerythritol

INTRODUCTION

Polyvinyl chloride (PVC) is the third most widely used polymer in the world, after polyethylene and polypropylene, with an annual consumption of over 40 million tons (Gou *et al.*, 2023). However, PVC degrades easily at processing temperatures (170°C and

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190°C) due to the presence of labile chlorines in its structure (Benavides *et al.*, 2001). Thermal degradation releases hydrogen chloride and results in the formation of polyene structures, which degrade the color and mechanical properties of PVC (Altarawneh *et al.*, 2022). To address this issue, thermal stabilizers are added to the PVC resin before processing (Li *et al.*, 2019). There are three main types of PVC stabilizers, i.e., lead, mixed metal, and tin-based thermal stabilizers (Markarian, 2007). Among these, mixed metal-based thermal stabilizers are widely used due to their effective stabilization properties, non-toxicity, and relatively low cost (Fang *et al.*, 2009). The most commonly used mixed metal pairs are Ca/Zn, which are typically used in the form of mixed Ca/Zn carboxylates (Atakul *et al.*, 2005).

Recently, we developed a mixed Ca/Zn thermal stabilizer where palm fatty acid distillate (PFAD) was used as a carboxyl source. This developed thermal stabilizer is known as Ca/Zn Palmate (Putrawan *et al.*, 2024, 2022). PFAD is a secondary product of palm oil refining (Mba *et al.*, 2015). As the world's largest palm oil producer, Indonesia produces up to 1.6 million tons of PFAD annually (Golden Agri-Resources, 2020), primarily through its extensive production of cooking oil. Ca/Zn Palmate has proven to be effective and competitive with stearate-based Ca/Zn thermal stabilizers currently available on the market (Putrawan *et al.*, 2022). This creates opportunities for more affordable Ca/Zn thermal stabilizers, given that PFAD is sold at $870 per ton (Agropost, 2024), while stearic acid is available at $1200 per ton (Echemi, 2024). In addition, this effort promotes byproduct valorization and encourages the use of more environmentally friendly alternative additives. It can also extend the life cycle of PVC products, aligning with the development of green technologies.

Building on previous work, this research focuses on investigating the degradation kinetics of PVC resin treated with a Ca/Zn Palmate. Understanding degradation kinetics is crucial for estimating thermal stability, which is directly related to improving stability and addressing the need for recycling PVC-based products. Knowledge of degradation kinetics also helps in understanding the effects of heat on PVC structure, which is a key aspect of material science and polymer chemistry. The study utilizes hydrogen chloride release data, measured through dehydrochlorination tests, as the basis for analysis. These tests were conducted under various conditions, including different temperatures, Ca/Zn Palmate doses, Ca/Zn ratios, and co-stabilizer amount. The two co-stabilizers used were pentaerythritol and dibenzoylmethane. Pentaerythritol enhances PVC stability by forming zinc-complex and scavenging hydrogen chloride, thus preventing the *zinc-burning* effect (Abbås and Sörvik, 1976; Briggs and Wood, 1971; Liu *et al.*, 2007)). Meanwhile, dibenzoylmethane helps PVC maintain its white color (Li and Yao, 2011). The resulting data were summarized in the form of degradation kinetic equations.

METHOD

Thermal Degradation Kinetics

The dehydrochlorination test offers insights into the release of hydrogen chloride from the PVC structure, tracking the process from the onset of degradation to severe damage. When the released hydrogen chloride during the thermal degradation is absorbed into water, the thermal degradation can be assessed by measuring the conductivity of the absorbing water. *The induction time* is defined as the point at which hydrogen chloride begins to be released from the PVC, marked by a conductivity increase of 3 µS/cm in the absorbing water (Putrawan *et al.*, 2023). The point at which the PVC undergoes severe damage is termed the *stability time*, recorded when the absorbing water exhibits a conductivity increase of 50 µS/cm (Putrawan *et al.*, 2023).

Figure 1 shows a typical curve from a dehydrochlorination test. At the beginning of the test, the conductivity of the absorbing water stays constant at a low initial value. As hydrogen chloride begins to release from the PVC, the conductivity increases slowly, then rapidly rises as hydrogen chloride is released in large quantities until the PVC is severely degraded.

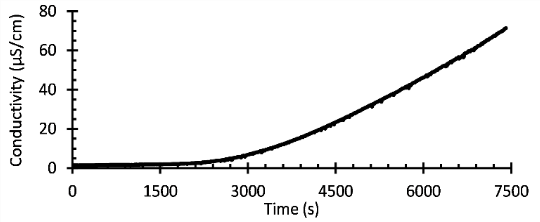


Fig. 1: Typical dehydrochlorination curve.

Referring to the general decomposition reaction described in the literature (Vitázek *et al.*, 2021), the thermal degradation of PVC can be represented as follows:

$$PVC_{(undegraded)} \xrightarrow{k_{(T)}} PVC_{(degraded)} + HCl_{(g)} \quad (1)$$

where $k_{(T)}$ is the rate constant. The reaction rate can be expressed in differential (2) and integral (3) forms:

$$d\alpha/dt = k_{(T)}\, f_{(\alpha)} \quad (2)$$

$$g_{(\alpha)} = k_{(T)}\, t \quad (3)$$

where α is conversion, *t* is time, *T* is the absolute temperature, and functions $f_{(\alpha)}$ and $g_{(\alpha)}$ are kinetic models in differential and integral forms, respectively. The rate constant can be obtained by plotting $d\alpha/dt$ vs $f_{(\alpha)}$ or $g_{(\alpha)}$ vs t.

There are various kinetic models available in literature. For PVC degradation, the Avrami-Erofeev model is suitable (Guangbao *et al.*, 2020). This model represents the differential function $f_{(\alpha)}$ as follows (Fatemi *et al.*, 1986):

$$f_{(\alpha)} = n\,(1-\alpha)[-\ln(1-\alpha)]^{1-1/n} \quad (4)$$

where α is a conversion and *n* is a constant (reaction number), which is the sum of the number of steps to produce stable product nuclei and the number of dimensions in which the product nuclei grow. Table 1 presents the $f_{(\alpha)}$ and $g_{(\alpha)}$ expressions of Avrami-Erofeev models for *n* equals to 2, 3, and 4, which are commonly occurring cases.

Table 1. Differential and integral functions of Avrami-Erofeev model (Guangbao *et al.*, 2020)

Model	f(α)	g(α)
A2	$2(1-\alpha)[-\ln(1-\alpha)]^{1/2}$	$[-\ln(1-\alpha)]^{1/2}$
A3	$3(1-\alpha)[-\ln(1-\alpha)]^{2/3}$	$[-\ln(1-\alpha)]^{1/3}$
A4	$4(1-\alpha)[-\ln(1-\alpha)]^{3/4}$	$[-\ln(1-\alpha)]^{1/4}$

Conversion (α) is defined as the fraction of the initial mass that has been degraded and is expressed as:

$$\alpha = (m_i - m)/(m_i - m_f) \quad (5)$$

where *m* is the weight of PVC and index *i* and *f* stand for the initial and final conditions, respectively. When dehydrochlorination is assessed through conductivity measurement, the amount of degraded PVC can be estimated from the volume of absorbed water and the concentration of hydrogen chloride. The concentration of hydrogen chloride is related to the conductivity of water by the following equation (Atakul *et al.*, 2005):

$$K = F \cdot \sum|Z_i| \cdot C_{HCl,w} \cdot \mathrm{U_{HCl}} \quad (6)$$

where K is conductivity, F is the Faraday constant (96485 C/mol), $\Sigma|Z_i|$ is the total charge number, $C_{HCl,w}$ is the concentration of HCl in water, and U_{HCl} is the ion mobility, which is the sum of the electric mobilities of H^+ and Cl^- ions in aqueous HCl at 25°C.

The change in rate constant with temperature is described by the Arrhenius equation:

$$\ln k = \ln A - (E_A/R) \cdot (1/T) \quad (7)$$

where A is the pre-exponential factor, E_A is the activation energy, and R is the gas constant. A plot of ln k concerning $1/T$ provides the activation energy and pre-exponential factor from the slope and intercept of the line, respectively.

Materials

Pentaerythritol (≥98%) and dibenzoylmethane (≥98%) were purchased from Merck. PVC resin (a k value of 66-67), calcium stearate (white powder, 6.5–6.7% of calcium), and zinc stearate (white powder, 10.5–11.5% of zinc) were supplied by a local supplier. Calcium and zinc palmate, i.e., calcium and zinc carboxylates derived from PFAD, were prepared using the previous method (Putrawan *et al.*, 2024).

Procedure

Dehydrochlorination tests were conducted using an apparatus as described previously (Putrawan *et al.*, 2023). Conductivity was measured using a Martini Conductivity Meter, model Mi-170 (Milwaukee brand), connected to a computer. Data was recorded every 10 seconds. Each test utilized 1 g of PVC resin containing Ca/Zn Palmate, with and without co-stabilizers. The test temperature varied between 170°C and 190°C. The PVC resin and thermal stabilizer were mixed using a blender, and 200 ml of distilled water was used to absorb the hydrogen chloride released during degradation. The variations included Ca/Zn Palmate dosage, Ca/Zn ratio, and dosage of co-stabilizer. The ratio of pentaerythritol/dibenzoylmethane was kept equal to 1. Table 2 presents the formula variations in detail. Each variation was tested twice. Dose is expressed in phr (*part per hundred resins*).

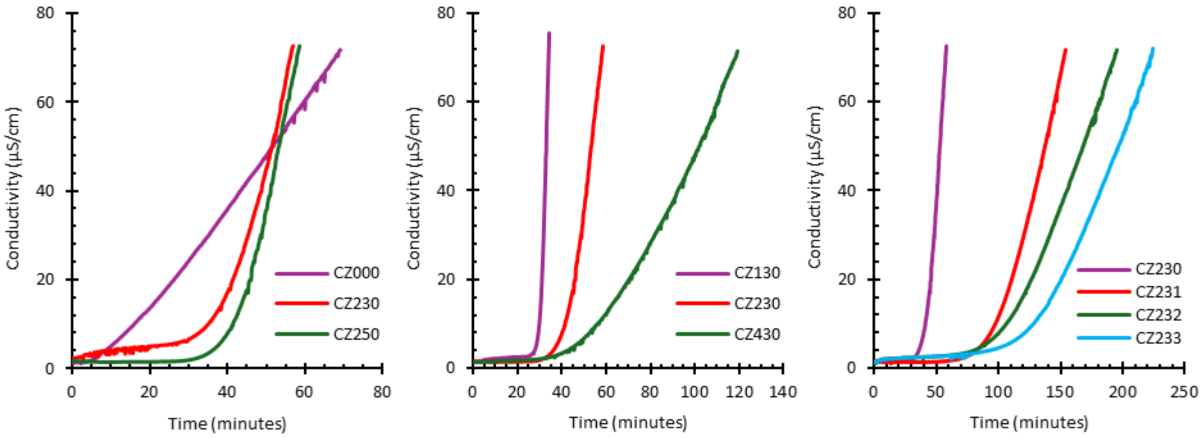
Table 2. Experimental runs

Formula	Ca/Zn ratio (-)	Ca/Zn dose (phr)	Co-stabilizer dose (phr)
CZ000		0	0
CZ230	2	3	0
CZ250	2	5	0
CZ130	1	3	0
CZ430	4	3	0
CZ231	2	3	1
CZ232	2	3	2
CZ233	2	3	3

RESULTS AND DISCUSSION

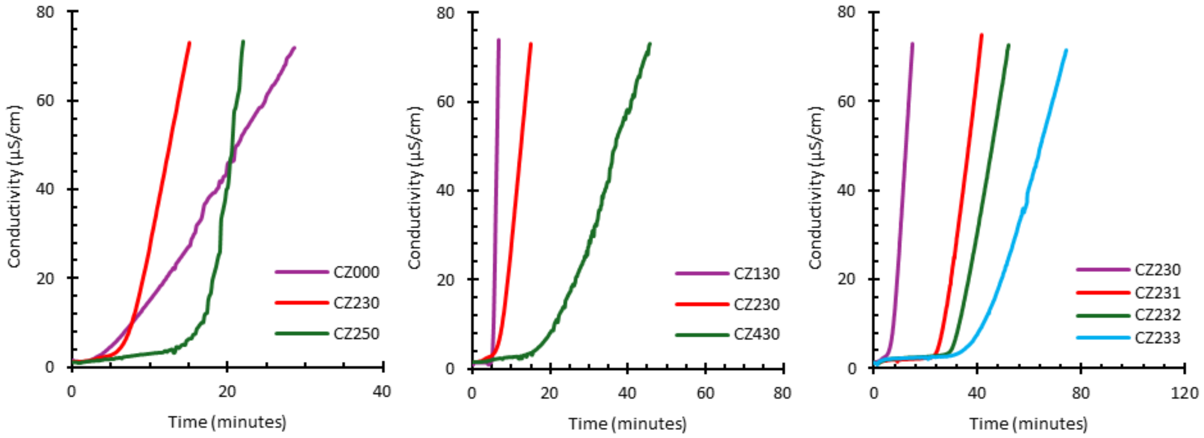
Induction and Stability Times

Figures 2, 3, and 4 present the dehydrochlorination curves obtained at 170°C, 180°C, and 190°C, respectively. Figures 5, 6, and 7 show the induction and stability times extracted from these curves. Variations in the formulas CZ000, CZ230, and CZ250 illustrate the effect of the Ca/Zn Palmate dose on the induction and stability times at a Ca/Zn ratio of 2. Increasing the dose of Ca/Zn Palmate led to a longer induction time. This is attributed to the role of Zn Palmate ($Zn(Palm)_2$), a zinc carboxylate, in removing labile chlorines, as demonstrated by the reaction in Figure 8(a), which delays the release of hydrogen chloride from the PVC structure. As the dose increases, more $Zn(Palm)_2$ is available to eliminate labile chlorines and thus extend the induction time. However, as more $Zn(Palm)_2$ eliminate labile chlorine, more $ZnCl_2$ is formed, and more Calcium Palmate ($Ca(Palm)_2$) is consumed to regenerate $Zn(Palm)_2$ through the reaction in Figure 8(b). The reaction in Figure 8(c), which binds hydrogen chloride to extend stability time, therefore, becomes less effective. Consequently, the stability time did not significantly increase with a higher dose, even though the amount of $Ca(Palm)_2$ increases in line with the dose at a constant Ca/Zn ratio.



(a) (b) (c)

Fig. 2: Effects of (a) Ca/Zn Palmate dosages, (b) Ca/Zn ratios, and (c) co-stabilizer dosages on dehydrochlorination curves of PVC resin (170°C)



(a) (b) (c)

Fig. 3: Effects of (a) Ca/Zn Palmate dosages, (b) Ca/Zn ratios, and (c) co-stabilizer dosages on dehydrochlorination curves of PVC resin (180°C)

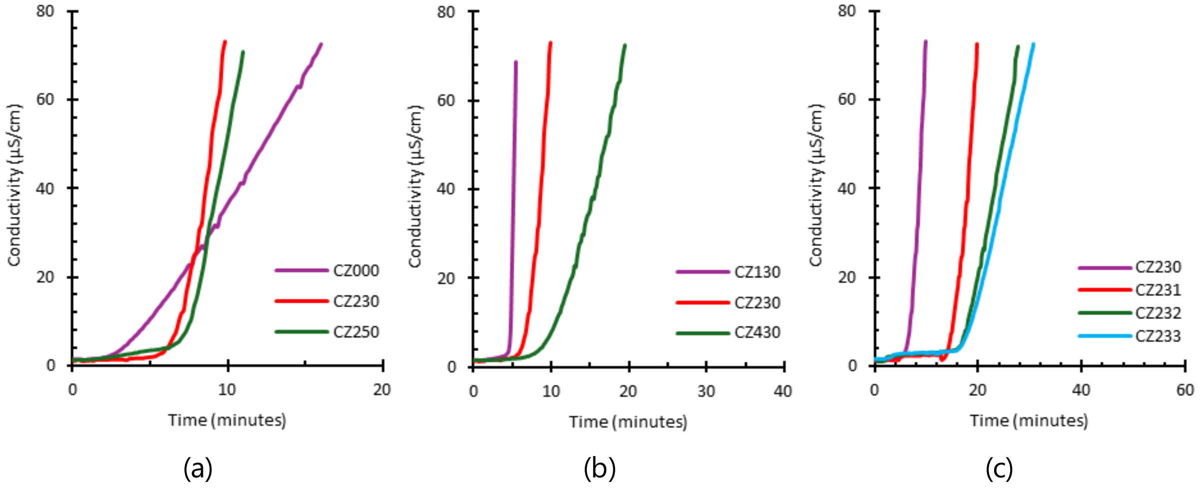


Fig. 4: Effects of (a) Ca/Zn Palmate dosages, (b) Ca/Zn ratios, and (c) co-stabilizer dosages on dehydrochlorination curves of PVC resin (190°C)

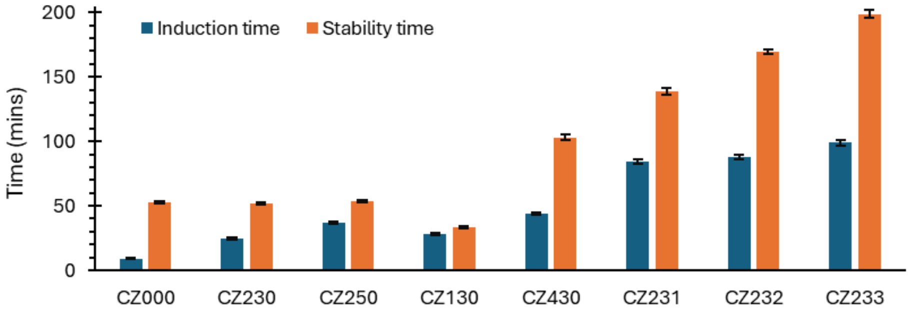


Fig. 5: Induction and stability times (170°C)

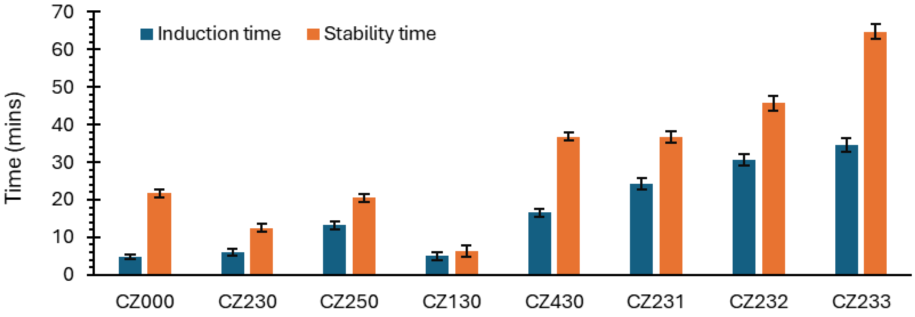


Fig. 6: Induction and stability times (180°C)

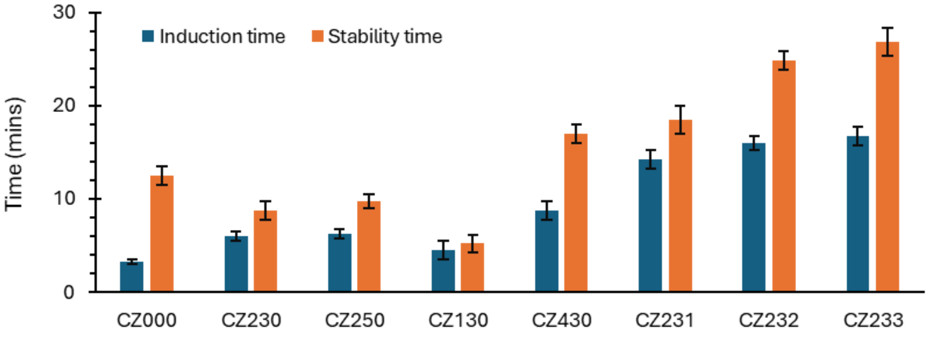


Fig. 7: Induction and stability times (190°C)

(a) $$Palm - Zn - Palm + PVC - Cl \rightarrow PVC - Palm + Cl - Zn - Cl$$

(b) $$Palm - Ca - Palm + Cl - Zn - Cl \rightarrow Cl - Ca - Cl + Palm - Zn - Palm$$

(c) $$Palm - Ca - Palm + H - Cl \rightarrow Cl - Ca - Cl + Palm - H$$

Fig. 8: Stabilization mechanisms

Variations in the formulas CZ130, CZ230, and CZ430 illustrate the effect of the Ca/Zn ratio on induction and stability times at a dose of 3 phr without co-stabilizers. Both induction and stability times increased with the Ca/Zn ratio. For all Ca/Zn ratios, the induction time was longer compared to PVC resin without a thermal stabilizer. However, at a Ca/Zn ratio of 1, the stability time was shorter than that of PVC resin without a thermal stabilizer. This is attributed to the "zinc-burning" effect of zinc carboxylate, where $ZnCl_2$ catalyzes PVC degradation more aggressively than HCl (Liao *et al.*, 2011). A low Ca/Zn ratio means there is insufficient $Ca(Palm)_2$ to decompose $ZnCl_2$, exacerbating the degradation.

The influence of the co-stabilizer is illustrated by the formulas CZ230, CZ231, CZ232, and CZ233. Stability time increased with the co-stabilizer dose and was significantly improved by the addition of a co-stabilizer. However, the induction time remained nearly constant. This occurred because the co-stabilizers used, pentaerythritol and β-dibenzoylmethane, primarily enhance long-term stability and initial color, respectively. Therefore, in the Ca/Zn Palmate-pentaerythritol-dibenzoylmethane system, the induction time is largely determined by the $Zn(Palm)_2$ dose and the Ca/Zn ratio.

As is known, color changes are a visible indicator of thermal degradation (Shen *et al.*, 2023). All PVC resins, originally white, turned maroon, blackish brown, or black after dehydrochlorination tests, as these tests exceeded the stability time, which represents severe degradation. To more clearly illustrate how color changes with temperature and thermal stabilizer formula, all PVC samples were heated for the same duration. Figure 9 shows the color of PVC samples after being heated for 15 minutes under various temperatures and thermal stabilizer formulas, along with the fresh (unheated) samples for comparison. A more severe degradation results in a darker color of the PVC resin.

The results of the dehydrochlorination tests are consistent with the observed changes in color: degradation accelerates with increasing temperature, severe degradation is delayed as the Ca/Zn ratio increases, the addition of co-stabilizers enhances heat resistance, and a higher dose (CZ250) provides greater stability than a lower dose (CZ230). After 15 minutes of heating, PVC samples with formulas CZ000, CZ230, CZ250, and CZ130 turned dark red or black at 180°C and 190°C, whereas the sample with formula CZ430 darkened at 190°C. This occurred since, under these conditions, the samples were heated close to or beyond their respective stability times.

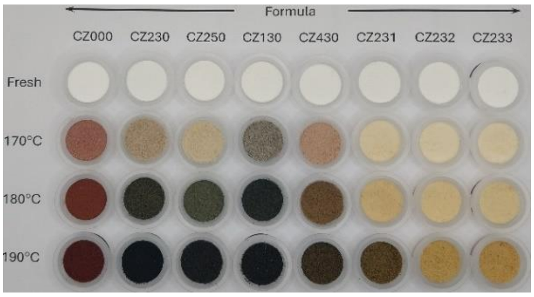


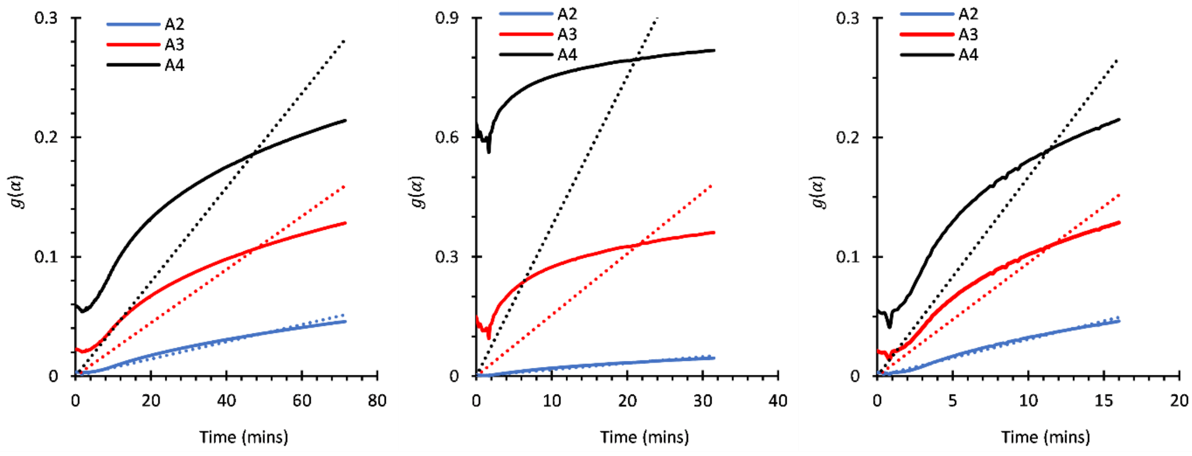
Fig. 9: PVC color after 15 minutes of heating

Kinetic Parameters

As previously mentioned, the Avrami-

Erofeev model was adopted in this study because it has been proven to be suitable for PVC degradation. Figure 10 shows typical Avrami-Erofeev integral function plots versus time, obtained for PVC without thermal stabilizer using various function models listed in Table 1. According to Eq. (3), at a given temperature, a suitable model will yield a linear relationship between the integral function and time, with the slope corresponding to the reaction rate constant. The A2 model produced a strong linear correlation, further confirming that the Avrami-Erofeev model with a reaction number of 2 (A2 model) accurately represents the degradation behavior of PVC.

Table 3 presents the reaction rate constants for PVC degradation at different temperatures and for various Ca/Zn Palmate formulas. The table also includes the activation energy and Arrhenius pre-exponential factor, calculated from the reaction rate constant data across the temperature range.



(a) (b) (c)

Fig. 10: Avrami-Erofeev integral function plots vs. time for PVC without thermal stabilizer at (a) 170°C, (b) 180°C, and (c) 190°C; dotted lines represent linear trendlines

Table 3. Kinetic parameters

Formula	Rate constant (10^{-5} s^{-1})			Activation energy (kJ/mol)	ln A
	170°C	180°C	190°C		
CZ000	1.19	2.72	5.15	124.4	23.24
CZ230	1.17	4.72	5.41	131.2	27.44
CZ250	1.06	2.15	5.00	133.3	31.50
CZ130	12.00	30.76	54.15	128.8	26.61
CZ230	1.17	4.71	5.41	131.2	27.44
CZ430	0.58	0.149	0.33	146.7	27.80
CZ230	1.17	4.72	5.41	131.2	27.44
CZ231	0.38	1.43	2.21	150.4	28.05
CZ232	0.29	1.17	2.03	167.2	32.88
CZ233	0.34	0.88	1.94	147.8	33.76

For all formulas, the reaction rate constant increases with rising temperature. It is due to the increased frequency of intermolecular collisions. In general, the pre-exponential factor increases with higher activation energy. This relationship suggests that the degradation mechanism of PVC stabilized by Ca/Zn Palmate across different formulas follows a similar pathway, indicating a homologous reaction series, as reported in the literature (Vrandečić *et al.*, 2004).

When comparing the formulas CZ000, CZ230, and CZ250, an increase in dose led to higher activation energy (better stability). This is expected, as discussed earlier, since increasing the dose enhances the stability of the PVC resin, as reflected in the longer stability times at higher doses. However, the increase in activation energy was not proportional to the dose of Ca/Zn Palmate. The rise in activation energy when the dose was increased from zero to 3 phr was more substantial than the increase from 3 phr to 5 phr. This suggests there is a maximum dose beyond which further increases offer only marginal improvements in stabilization. A similar effect was observed in the literature (Atakul *et al.*, 2005) for Ca/Zn stearate, where activation energy was 127.02 kJ/mol at 1 phr, 129.99 kJ/mol at 3 phr, and 130.28 kJ/mol at 4 phr.

Among the Ca/Zn ratios studied, the formula CZ130 with a ratio of 1 exhibited the highest reaction rate constant and the lowest activation energy, indicating that this formula makes PVC the most susceptible to degradation. This aligns with the stability time data, where the CZ130 formula resulted in the shortest stability time, even shorter than that of PVC resin without a thermal stabilizer. Similarly, for stearate-based Ca/Zn mixed metals, the best stability time is also provided at a high Ca/Zn ratio, i.e., 4 (Liao *et al.*, 2011).

The activation energy of formulations with varying co-stabilizer doses followed the order: CZ230<CZ233<CZ231<CZ232. In other words, the activation energy increased from no co-stabilizer up to a dose of 2 phr, then decreased when the dose reached 3 phr. With two types of co-stabilizers and an equal dose ratio between them, a total co-stabilizer dose of 2 phr means each co-stabilizer is present at 1 phr. Additionally, with a Ca/Zn Palmate dose of 3 phr and a Ca/Zn ratio of 2, the dose of Zn Palmate is also 1 phr. Therefore, the highest activation energy occurs when the dose of each co-stabilizer matches the dose of Zn Palmate.

Several researchers have investigated the degradation kinetics of PVC and reported activation energies. Using three model-free methods, activation energies of 152.9–294 kJ/mol were found for new PVC sheathing and 145.5–305.0 kJ/mol for aged PVC sheathing (Wang *et al.*, 2019). The average activation energy for pure PVC was calculated to be 119.8 kJ/mol, which decreased to 110.6 kJ/mol with the addition of a stoichiometric amount of ZnO (Altarawneh *et al.*, 2022). Activation energies of 114 and 202 kJ/mol were found for the first and second stages of PVC dehydrochlorination, respectively (Sánchez-Jiménez *et al.*, 2010). The activation energies for the dehydrochlorination stage of PVC degradation published between 1990 and 2000 were within the range of 114 and 247 kJ/mol (Wang *et al.*, 2019). The activation energies for PVC degradation stabilized by Ca/Zn Palmate in this work, which range from 129 to 167 kJ/mol, align well with the values reported in the literature.

The fact that the activation energy obtained in this study falls within the range reported in the literature highlights that the Ca/Zn Palmate formulation investigated here exhibits a thermal stabilizing effect comparable to that of commonly used thermal stabilizers. These results provide sufficient evidence to suggest that the developed Ca/Zn Palmate can serve as a viable alternative for PVC processing. However, the optimal formulation depends on the required processing time, which varies across different applications. In addition to thermal stabilizers, PVC processing often requires other additives such as lubricant, processing aid, impact modifier, and plasticizer (Campisi *et al.*, 2025; Edo *et al.*, 2024). The need for these additives and their appropriate dosages also differs depending on the specific application. Moreover, the PVC processing performance is influenced by the interaction between the thermal stabilizer and the other additives used. Therefore, the most suitable formulation must be tailored to the intended application

CONCLUSIONS

The degradation of PVC resin stabilized with Ca/Zn Palmate at various Ca/Zn Palmate doses, Ca/Zn ratios, and co-stabilizer doses has been studied by assessing its dehydrochlorination curves in the 170–190°C range. The degradation reaction followed the A2 model of Avrami-Erofeev. It was found that the activation energy of unstabilized PVC is 124.4 kJ/mol and that of the stabilized PVC falls in the 128,8-167.2 kJ/mol range. The greater the Ca/Zn Palmate dose or Ca/Zn ratio, the higher the activation energy. Under the studied range of co-stabilizer doses, the highest activation energy occurs when the dose of each co-stabilizer matches the dose of Zn Palmate. The activation energies for PVC degradation stabilized by Ca/Zn Palmate align well with the activation energy values reported in the literature.

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