KINETIC ANALYSIS OF THE THERMAL DEGRADATION OF POLYVINYL CHLORIDE STABILIZED WITH SUNFLOWER OIL-BASED METAL SOAPS

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Abstract

One of the major challenges encountered during the processing of polyvinyl chloride (PVC) is its susceptibility to thermal degradation. Although a variety of thermal stabilizers are currently employed in industry to mitigate this problem, the development of a universal and highly effective stabilizer remains an urgent research priority. In this context, the influence of metal soaps synthesized from sunflower oil on the thermal degradation of PVC was investigated using thermogravimetric analysis (TGA). The degradation kinetics were further evaluated by applying the Coats–Redfern method, based on different kinetic models, as well as the model-free Broido integral method. The TGA results demonstrated that the incorporation of metal soaps effectively delayed the onset of thermal degradation, with decomposition occurring at higher temperatures compared to pure PVC. Specifically, the temperatures corresponding to 5, 10, 20, and 50% mass loss for pure PVC were 256.56, 270.71, 279.25, and 313.14 °C, respectively. For PVC stabilized with calcium soap, these values increased to 267.60, 276.51, 291.41, and 323.73 °C, while for PVC stabilized with aluminum soap, the corresponding values were 270.01, 284.96, 301.46, and 335.19 °C. These findings were further corroborated by the higher activation energy values obtained from the Coats–Redfern analysis and the Broido method, indicating an improvement in thermal stability. Overall, the results suggest that sunflower-oil–based metal soaps enhance the thermal stability of PVC and may serve as promising alternatives for long-term stabilization applications.

Keywords: polyvinyl chloride, thermal stabilization, thermal degradation, sunflower oil, metal soaps

I. Introduction

The polymer industry has undergone rapid development since the previous century, and polymer-based compositions have become some of the most widely utilized materials in modern society. Global polymer production now exceeds 400 million tons annually, with forecasts indicating that this upward trend will continue in the near future. Among the most extensively produced polymers are polyethylene, polypropylene, and polyvinyl chloride (PVC), which serve as the basis for a broad range of consumer and industrial products used in virtually every aspect of daily life [1]. PVC is particularly versatile, as it can be processed into both plasticized (flexible) and unplasticized (rigid) composites. Common applications include pipes, profiles, cable insulation, fittings, and packaging materials for food and industrial goods [2]. While PVC exhibits favorable chemical and mechanical stability, its major drawback is its susceptibility to thermal degradation [3].

During processing and service, exposure to elevated temperatures initiates degradation processes. Beginning near the glass transition temperature, the release of hydrogen chloride (HCl) occurs due to structural defects in the polymer backbone. At approximately 140 °C, this process accelerates, with the evolved HCl exerting an autocatalytic effect that further promotes chain scission within the polymer matrix [4–6]. Continued heating leads to mass loss as HCl evolution progresses. Concurrently, chlorine elimination gives rise to double bonds along the polymer chain, which extend to form conjugated polyene sequences. As the population of these polyenes increases, progressive discoloration of the PVC composition occurs. Once the conjugated segments reach a length of approximately 5–7 double bonds—corresponding to an overall mass loss of around 1%—visible darkening of the polymer becomes evident [7–9].

Although PVC undergoes thermal degradation at relatively moderate temperatures, its processing typically requires exposure to much higher temperatures, in some cases exceeding 200 °C [10]. To prevent such degradation under processing conditions, thermal stabilizers are commonly employed. A wide range of stabilizers with different origins and chemical compositions are used in industry, including lead compounds [11], organotin compounds [12], calcium–zinc systems and metal soaps [13], organic stabilizers [14], and hydrotalcite-based additives [15].

Despite their effectiveness, the use of lead-based stabilizers has been progressively restricted due to growing evidence of their adverse effects on human health. Numerous toxicological studies have demonstrated that lead (Pb) is one of the most hazardous heavy metals, with widespread environmental distribution in water, soil, food, and plastic materials [16,17]. Once absorbed, lead accumulates in the body and is eliminated only very slowly, thereby amplifying its toxicological impact [18]. Clinical findings indicate that lead primarily affects the nervous system, hematopoietic system, kidneys, and reproductive functions [19]. In children, lead exposure is strongly associated with reduced cognitive ability, attention deficits, and intellectual disability, whereas in adults it is linked to hypertension, renal failure, and impaired calcium metabolism in bone tissue [20,21]. Consequently, the use of lead stabilizers has been banned in the European Union, although they are still employed in parts of Asia.

Tin-based stabilizers represent another widely used class of additives for the preparation of PVC-based composites. Organotin compounds, in particular, have been shown to play a crucial role in enhancing the thermal stability of PVC. These stabilizers suppress dehydrochlorination, elevate the decomposition temperature, and additionally offer benefits in terms of color retention and transparency [22]. For example, pentaerythritol stearate ester-based tin(II) alkoxides have been demonstrated to exert a particularly strong stabilizing effect, while dioctyltin maleate stabilizers have also been reported to significantly improve the thermal resistance of PVC [23,24]. Nevertheless, tin-based stabilizers also exhibit several disadvantages. Foremost among these is their high production and application cost, which restricts large-scale industrial use. Furthermore, ecotoxic effects and the bioaccumulation potential of certain organotin derivatives have been reported, raising significant concerns for both environmental and human health [25].

Calcium and zinc soaps, regarded as more environmentally benign alternatives, remain less effective compared with lead- and tin-based stabilizers. A major limitation is that $ZnCl_2$, generated during PVC degradation, acts as a catalyst that accelerates dehydrochlorination. This phenomenon, commonly referred to as "zinc burning," compromises the long-term stability of the polymer mass [26]. In addition, emerging evidence regarding the adverse health effects of excessive zinc exposure has cast further doubt on its ecological safety [27].

Hydrotalcites have also been explored as stabilizers, although their industrial application remains limited. Despite their effectiveness in small concentrations, their relatively high cost and their tendency to increase polymer viscosity—thereby impairing processability—hinder their widespread adoption [28].

In recent years, considerable attention has shifted toward bio-based stabilizers derived from renewable raw materials. Such additives include metal soaps synthesized from vegetable oils, epoxy compounds, and various metal chelates. For instance, calcium and zinc soaps prepared from free fatty acids, a by-product of palm oil refining, have been evaluated as PVC stabilizers. The results indicated that these compounds can serve as promising alternatives to conventional synthetic metal soaps [29]. Similarly, tannin–metal complexes, particularly tannin–calcium systems, have demonstrated multifunctional stabilizing effects in PVC formulations [30].

Although Ca–Zn-based plant-derived soaps have been extensively studied, stabilizers derived from other metals remain comparatively underexplored. Importantly, the ongoing challenge lies in identifying stabilizers that are simultaneously eco-friendly, cost-effective, and efficient. Toward this aim, recent studies have investigated the effect of calcium and aluminum soaps synthesized from sunflower oil on the thermal stability of PVC. In these studies, the kinetics of PVC thermal degradation were analyzed, and the activation energy was determined using Coats–Redfern and Broido integral methods, providing deeper insight into the stabilizing efficiency of these bio-based compounds.

II. Experimental part

For the synthesis of metal soaps derived from sunflower oil, Final brand commercial sunflower oil was selected as the raw material. The oil was subjected to saponification in an ethanol–water medium in the presence of alkali. Following the synthesis of the water-soluble soap, the product was diluted with water to obtain a homogeneous solution. The excess alkali that did not participate in the reaction was subsequently neutralized using a standard hydrochloric acid solution. The physicochemical properties of the sunflower oil employed in the synthesis are presented in Table 1 [31].

Table 1. Properties of sunflower oil

Saturated fatty acids, %	Oleic acid, %	Linoleic acid, %	Linolenic acid, %	Free fatty acids, %	Iodine value, g/100g	Saponification value, mg/g	Moisture, %
10.7	20.7	67.6	≤ 1	0.062	136	191	0.05%

To obtain aluminum and calcium soaps, aqueous 20% solutions of $CaCl_2$ and $Al_2(SO_4)_3 \cdot 18H_2O$ salts were prepared in slightly more than the stoichiometric amount and added to the previously synthesized soap solution. The corresponding metal soaps were precipitated, collected by filtration through filter paper, thoroughly washed several times with distilled water, and subsequently dried. The dried precipitates were ground in a mortar and applied as stabilizers to the polymer matrix.

For the preparation of polymer samples, Rusvinyl PVC-S grade poly(vinyl chloride) was used as the base polymer. The synthesized calcium and aluminum soaps were separately incorporated into the PVC at a concentration of 5 phr (parts per hundred resin) and mechanically blended. To fabricate test specimens of both the mixtures and neat PVC, the blends were pressed using an Ecopress 102 (Turkey) heating press at 180 °C under a pressure of 150 bar.

Thermal degradation studies of the prepared samples were carried out using a NETZSCH STA449F3 Jupiter (Germany) thermogravimetric analyzer. Thermogravimetric analysis (TGA) was performed under a nitrogen atmosphere up to 900 °C with a constant heating rate of 30 °C/min. The activation energies corresponding to the dehydrochlorination and initial decomposition stages of PVC were determined from the thermogravimetric data using the Coats–Redfern and Broido integral methods [32,33]. Both approaches are based on the Arrhenius equation, which describes the temperature dependence of reaction rate constants. In the Broido method, the activation energy is evaluated according to the Broido equation (Eq. 2.1):

$$ln\left(ln\left(\frac{1}{1-\alpha}\right)\right) = -\frac{E_a}{R}\frac{1}{T} + constant \quad (2.1)$$

- α – degree of conversion for the selected reaction stage

$$\alpha = \frac{w_0 - w_t}{w_0 - w_f} \quad (2.2)$$

- w_t – residual fraction (%) at a selected temperature
- w_f – residual fraction (%) at the end of the selected temperature interval
- w_0 – initial fraction (%) at the beginning of the selected temperature interval
- E_a – activation energy (J/mol)
- R – universal gas constant (8.314 J·mol^{-1}·K^{-1})
- T – absolute temperature (K)

For the kinetic calculations, the Coats–Redfern equation, derived from the Arrhenius law, was applied (Eq. 2.3).

$$ln\left[\frac{g(\alpha)}{T^2}\right] = ln\left[\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right] - \frac{E_a}{RT} \quad (2.3)$$

- α – degree of conversion for the Coats–Redfern method

$$\alpha = \frac{w_0 - w_t}{w_0 - w_f} \quad (2.4)$$

- $g(\alpha)$ – integral form of the reaction model
- A – characteristic coefficient (pre-exponential factor) (min^{-1} or s^{-1})

At high activation energy values, the Coats–Redfern method can be expressed as follows:

$$ln\left[\frac{g(\alpha)}{T^2}\right] = ln\left(\frac{AR}{\beta E_a}\right) - \frac{E_a}{RT} \quad (2.5)$$

($ln\left(\frac{AR}{\beta E_a}\right)$ – characterizes the intersection point

The most commonly used kinetic models for the dehydrochlorination stage are presented in Table 2.

Table 2: Kinetic models for dehydrochlorination stage

Model	Mechanism	$g(\alpha)$
F1	First-order reaction	$-ln(1-\alpha)$
D3	Three-dimensional diffusion	$\left[1-(1-\alpha)^{1/3}\right]^2$
A2	Avrami–Erofeev (n = 1/2)	$[-ln(1-\alpha)]^{1/2}$
R3	Contracting sphere	$1-(1-\alpha)^{1/3}$

For the determination of activation energy, linear regression analysis is employed. In the Coats–Redfern method, the regression is constructed using $Y = ln\left(\frac{g(\alpha)}{T^2}\right), X = \frac{1}{T}$. In the Broido method, linear regression is performed using $Y = ln\left(ln\left(\frac{1}{y}\right)\right), X = \frac{1}{T}$. Based on the constructed regression plots, the activation energy and the determination coefficient are obtained.

III. Results and discussion

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of PVC samples demonstrated that bio-based metal soaps markedly enhance the thermal stability of the polymer. For the unstabilized PVC sample, a rapid decrease in mass was observed beginning at approximately 220 °C, whereas in PVC samples stabilized with metal soaps, significant weight loss did not occur until after 240 °C.

During the initial degradation stage, namely dehydrochlorination, the polymer lost nearly 60% of its mass. In neat PVC, this process proceeded within a narrower temperature range. Specifically, a 5% mass loss was recorded at 256.56 °C. With increasing temperature, mass loss accelerated: 10% and 20% mass losses occurred at 270.71 °C and 279.25 °C, respectively. A 50% mass loss and a 60% mass loss—marking the completion of dehydrochlorination and the end of the initial decomposition

stage—were observed at 313.14 °C and 341.77 °C, respectively. The TGA–DTA–DDTA curves of pure PVC are presented in Fig.1.

As evident from the DDTA curve, energy absorption increased sharply within the temperature interval corresponding to dehydrochlorination. The abrupt mass loss at this stage is attributable to the autocatalytic effect of hydrogen chloride (HCl) released during degradation. Moreover, the lower energy absorption recorded in the DTA curve during the initial decomposition stage, compared with that in the subsequent stages of secondary organic mass decomposition and charring, further corroborates the autocatalytic role of HCl.

For the PVC sample stabilized with aluminum soap synthesized from sunflower oil, a 5% mass loss was recorded at 270.01 °C. As in the case of pure PVC, degradation intensified with rising temperature, leading to rapid weight reduction; however, compared with neat PVC, the onset of degradation was delayed and the rate of mass loss was lower. Specifically, 10%, 20%, and 50% mass losses occurred at 284.96 °C, 301.46 °C, and 335.19 °C, respectively. Completion of the dehydrochlorination stage, corresponding to a 60% mass loss, was observed at 357.72 °C. The observed increase in decomposition temperatures confirms the thermal stabilizing effect of aluminum soap. The TGA–DTA–DDTA curves of PVC stabilized with aluminum soap are presented in Fig.2.

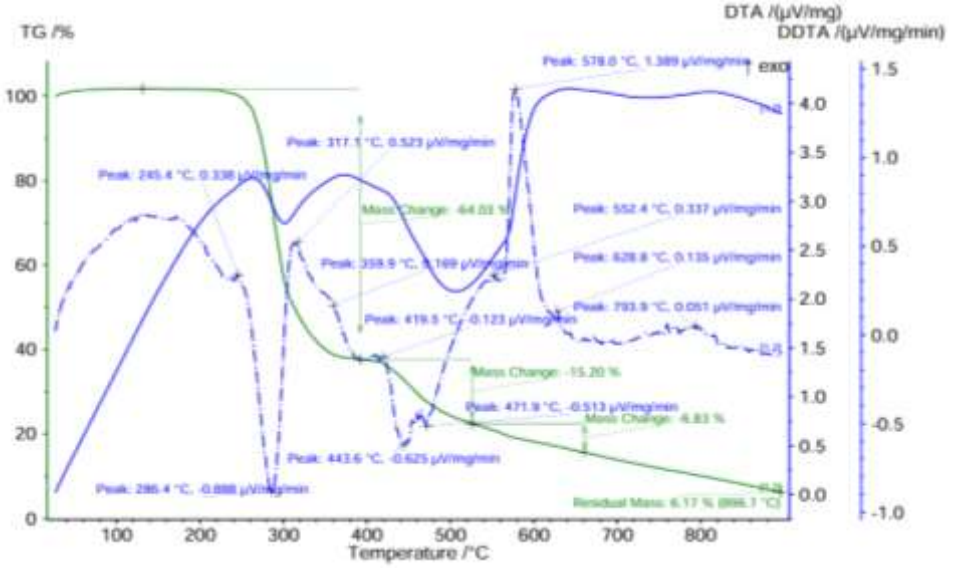

Fig.1. *TGA-DTA-DDTA curves of pure PVC*

As observed from the DDTA curves, energy absorption in the temperature interval corresponding to the dehydrochlorination stage was lower than that of pure PVC. This behavior can be attributed to the exothermic reaction occurring when the hydrogen chloride (HCl) released during degradation is neutralized by the metal soap stabilizer. The reduced energy uptake indicates that HCl was effectively scavenged by the aluminum soap.

Furthermore, the hydrophobic gel-like structure of the sunflower oil–based aluminum soap likely hindered the diffusion of HCl into the polymer phase. This suggests that the aluminum soap reduced the autocatalytic effect of HCl by providing a barrier effect. A comparison of the DDTA and DTA curves further revealed that energy absorption in the second decomposition stage was generally higher. The lower energy absorption observed in the pyrolysis region, relative to pure PVC, may be explained by the formation of $AlCl_3$ during dehydrochlorination, arising from the interaction of HCl with aluminum soap, and by the catalytic role of this salt in subsequent degradation reactions [34].

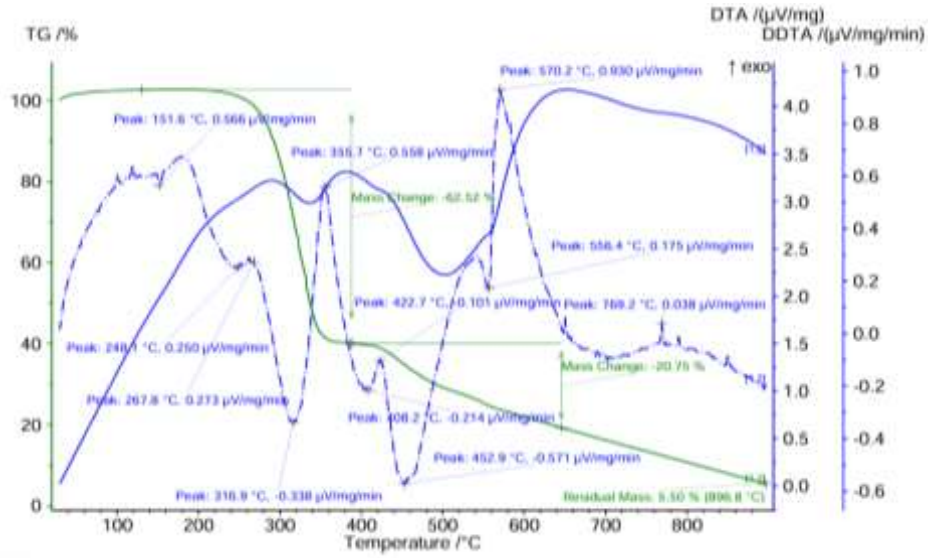

Fig.2. *TGA-DTA-DDTA curves of stabilized PVC with aluminum soap*

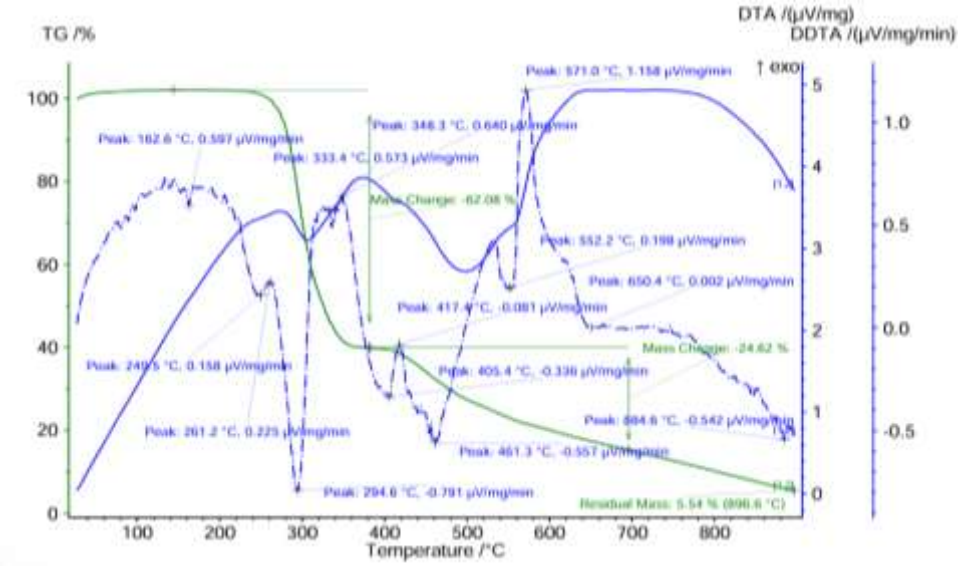

Fig.3. *TGA-DTA-DDTA curves of stabilized PVC with calcium soap*

The TGA results of the calcium soap–stabilized PVC sample indicated that a 5% mass loss occurred at 267.60 °C, a 10% loss at 276.51 °C, and a 20% loss at 291.41 °C. These findings demonstrate that calcium soap contributed to improved thermal stability of PVC. A 50% mass loss was observed at 323.73 °C, while a 60% mass loss—marking the completion of the dehydrochlorination stage—was recorded at 353.27 °C. In contrast to the aluminum soap–stabilized system, no significant variation in energy absorption was observed in the DDTA and DTA curves (Fig.3).

The percentage mass loss of PVC samples at 250, 300, 350, 400, and 450 °C is summarized in Table 3. As can be seen, the incorporation of thermal stabilizers exerted little influence on the second decomposition stage, with stabilization being primarily evident during the dehydrochlorination stage.

Table 3. Mass losses of PVC samples at 250, 300, 350, 400 and 450° C

Sample	Temperature, ° C				
	250	300	350	400	450
	Mass loss, %				
PVC-pure	3.05	41.57	61.35	64.29	69.98
PVC-Aluminum soap	2.03	18.90	57.94	62.89	67.32
PVC-Calcium soap	1.49	29.66	59.44	62.57	67.84

The calculation of activation energies for the degradation reactions in the presence of thermal stabilizers was primarily focused on the dehydrochlorination stage. In this study, the Coats–Redfern and Broido integral methods were applied to evaluate the activation energies of PVC samples. The calculations were performed using thermogravimetric data, with linear regression established for four kinetic models of the Coats–Redfern method, as well as the Broido method. The corresponding regression plots are presented in Fig. 4, 5, and 6 for pure PVC, PVC–aluminum soap, and PVC–calcium soap, respectively.

As illustrated in all three graphs, the first-order reaction model (F1) of the Coats–Redfern approach exhibited the closest correlation with the linear regression derived from the Broido method. Although the F1 model is most frequently employed to describe the dehydrochlorination process, other kinetic models were also evaluated. These include the three-dimensional diffusion model (D3), which accounts for gas diffusion through the polymer matrix; the Avrami–Erofeev model (A2), which describes the reaction in terms of nucleation and crystal growth; and the three-dimensional shrinking-core model (R3), which characterizes the process as spherical nucleation.

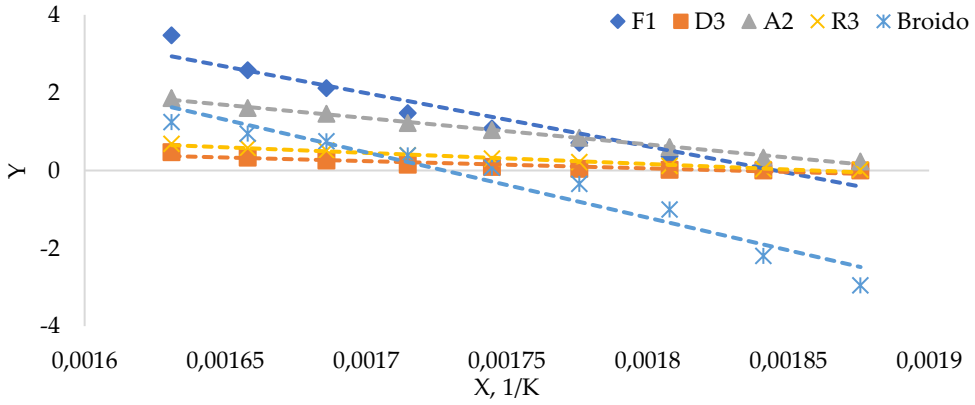

Fig.4. *Linear regression plot of PVC-pure constructed according to the Coats–Redfern and Broido method*

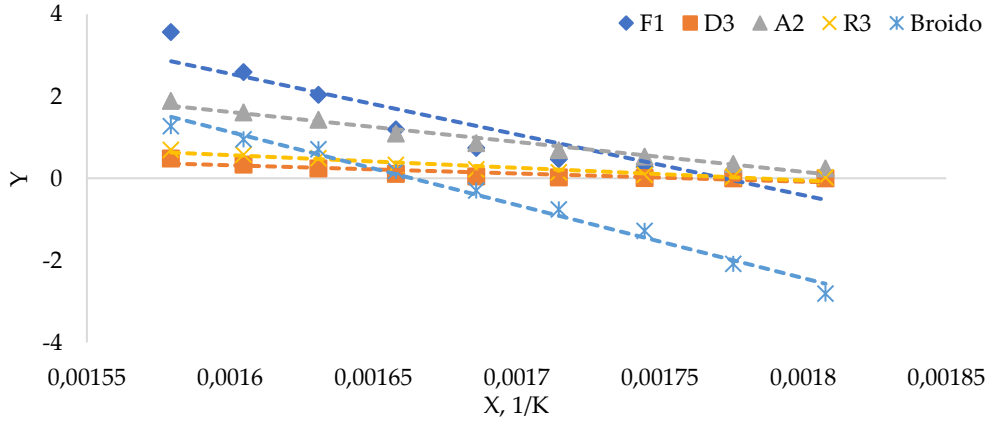

Fig.5. *Linear regression plot of PVC-pure constructed according to the Coats–Redfern and Broido method*

The results of the kinetic calculations demonstrated that the activation energy values obtained using the F1 model of the Coats–Redfern method and the model-free Broido method are consistent with values reported in the scientific literature. Although the activation energies derived from the alternative models were comparatively lower, they nevertheless provide useful insights into the degradation mechanism. The calculated results are summarized in Table 4.

As shown in the table, the incorporation of aluminum and calcium soaps led to an increase in the thermal stability of PVC. Furthermore, the observed increase in the pre-exponential factor indicates that, while the stabilizers enhance the decomposition onset temperature, the degradation process ultimately accelerates at higher temperatures. This suggests that thermal stabilizers play a more significant role in delaying the onset of dehydrochlorination rather than influencing the subsequent stage of degradation, which involves main-chain scission and charring.

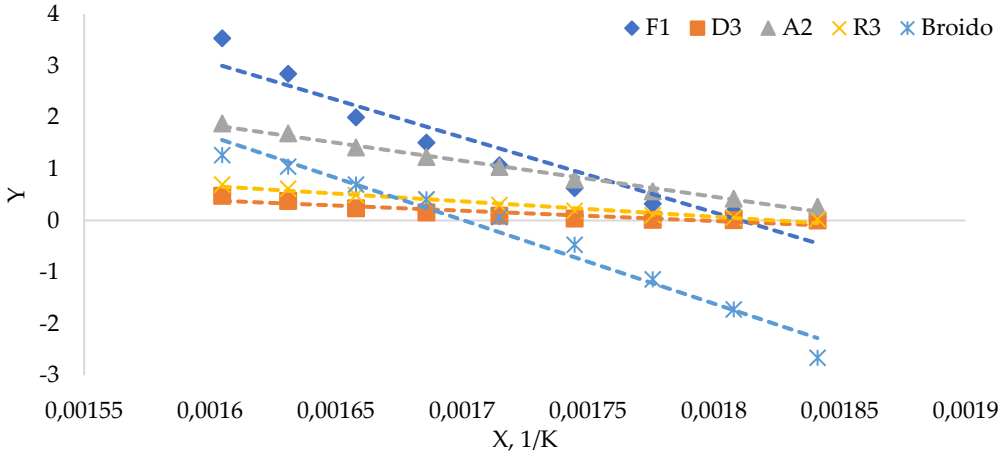

Fig.6. *Linear regression plot of PVC-pure constructed according to the Coats–Redfern and Broido method*

Table 4. Activation energies, determination coefficients, and pre-exponential factors of PVC samples

Mothod	Model	Activation energy (Ea), Determination coefficient (R^2), Pre-exponential factor (A)	PVC-pure	PVC-Aluminum soap	PVC-Calcium soap
Coats-Redfern	F1	Ea, kJ/mol	113.74	123.02	120.97
		R^2	0.93	0.87	0.92
		ln(A), s^{-1}	34.08	35.13	35.24
	D3	Ea, kJ/mol	15.47	16.65	16.62
		R^2	0.86	0.79	0.85
		ln(A), s^{-1}	10.24	10.43	10.50
	A2	Ea, kJ/mol	56.29	60.63	57.95
		R^2	0.99	0.97	0.99
		ln(A), s^{-1}	20.98	21.48	21.16
	R3	Ea, kJ/mol	23.72	25.56	24.81
		R^2	0.98	0.94	0.97
		ln(A), s^{-1}	12.57	12.82	12.75
Broido		Ea, kJ/mol	139.52	148.13	134.99
		R^2	0.94	0.99	0.97
		ln(A), s^{-1}	38.03	38.73	36.62

IV. Conclusion

The effect of calcium and aluminum soaps synthesized from vegetable oil on the thermal stability of polyvinyl chloride was investigated. Thermogravimetric results demonstrated that these metal soaps enhanced the thermal stability of the polymer. To more precisely evaluate their influence, the activation energies of thermally stabilized and unstabilized PVC samples were calculated using the multi-model Coats–Redfern method and the Broido integral method. The results indicated that the incorporation of metal soaps increased the activation energy of the dehydrochlorination stage, confirming their stabilizing effect. Conversely, it was observed that the stabilizers exerted little to no influence on the subsequent decomposition of the organic residue following dehydrochlorination.

CONFLICT OF INTEREST.
Authors declare that they do not have any conflict of interest.

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