NOTE

Application of Thermogravimetric Analysis to Predict the Thermal Rating of Solid Insulating Materials

INTRODUCTION

The degradation of polymeric insulation materials and systems is governed by several factors. In service, the combined influence of thermal and electrical stresses, in addition to mechanical abuses, plays a critical role in determining the life of insulation materials. A knowledge of the useful service life of a material before application is therefore essential in order to get better performance and to even avert premature failures. Generally, the 'life-test' is conducted by long-term ageing studies, which is time consuming and expensive. However, this is the universally accepted standard practice to determine the thermal rating or temperature index of materials. Details of such tests (conventional method), failure criteria, and methods of calculation are described in detail elsewhere.[1]

With the rapid developments of newer polymeric materials as candidates for insulation application, and also the limitations of longer time and cost associated with the conventional method, it became very essential to look for a suitable alternative that is rapid, cheap, and accurate. In this quest for an accurate and quicker method to estimate the life of a polymer, thermogravimetric analysis (TGA) was identified as a shortcut method in 1965.[2] Several attempts have been made since to optimise the various parameters of TGA and to transfer the data from the thermogram to the Arrhenius equation in order to calculate the temperature index.[3,4] Statistical theory also appears in the literature.[5]

The DiCerbo method[3] suggests drawing two tangents to the thermogram, one at the point at which the curve starts to deviate from its initial straight line portion and the other along the steep portion of the weight loss curve. A mathematical formula has been derived to calculate the temperature index. However, this method is not very accurate for materials with complex degradation behaviour. This is so because this method explains only the role of individual parameters on the nature of the thermogram without concrete scientific explanation. Toop[4] tried to derive the thermal index by calculating the activation energy, thus establishing the much desired correlation between the two. Here again, the exact degradation point in the thermogram, at which the activation energy is to be calculated and considered for thermal rating, is not suggested. Other attempts[6] to refine Toop's approach also do not seem to clearly define this aspect.

Extensive work was therefore conducted at the authors' laboratory on several baking-type insulating varnishes by subjecting them to conventional ageing and thermogravimetry. The findings were very encouraging and gave a new direction to the problem. The results of the study have been accepted by the funding agencies.[7]

Encouraged by the results, work was further extended to other well-established solid insulation materials. This article is the result of that exercise.

EXPERIMENTAL

Six important and very widely used solid insulation materials were chosen and subjected to thermogravimetric analysis. The details of materials are given in Table I.

It was suggested by DiCerbo[3] that the slowest possible heating rate should be used for the analysis, 1°C/min heating rate. Further, Toop's method[4] requires three thermograms at different heating rates to fit the data in the model suggested by him. Accordingly, the following test conditions were adopted in the Shimadzu Thermal Analyser Model DT 30 for the study:

1. weight of sample: 10–20 mg
2. heating rates: 1, 2, and 5°C/min
3. atmosphere: air; flow rate: 30 mL/min
4. temperature range: ambient to 600°C

The thermograms of all the samples are shown in Figures 1–6.

RESULTS AND DISCUSSION

As mentioned earlier, Toop's method[4] does not specify the points in the thermogram at which the activation energy should be calculated and used to determine the temperature index. Therefore, it was felt that there is a need to optimize the parameters in order to achieve a more accurate temperature rating of a material. Further, the bent portion in the thermogram represents the degradation of the material. In the present study, the activation energy

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Table I Materials Used for the Study

Sample No.	Material	Trade Name/Source	Sample Form
1	Poly(vinyl chloride) (PVC)	Indigenous/IPCL	Fine granules
2	Crosslinked poly(ethylene) (XLPE)	Indigenous/Torrent Cables	Solid cake
3	Capacitor grade tissue paper	Natron PK/Julius, Glatz, Germany	Film
4	Poly(propylene) (BOPP)	Shin-etsu/Japan	Film
5	Poly(ethylene terephthalate) (PET)	Indigenous/Garware Plastics	Film
6	Poly(tetrafluoroethylene) (PTFE)	'Champion'/indigenous	Solid cake

was calculated at 10 points in the thermogram at the bent portion (except for XLPE) and the average value was substituted in the formula to calculate the temperature index. In the case of XLPE, however, the bent portion was not considered for calculating the activation energy, because the sharp deviation of curve at 12% weight loss suggests that the material has already degraded before reaching that point. Therefore, the activation energy at 10 points between 3 to 12% weight loss was calculated. It has been suggested by Toop[4] that a plot of reciprocal temperature versus logarithm of heating rate, which is a straight line, can be used to calculate the activation energy. The formula is:

$$\text{slope} = 0.457 \frac{E}{R} \quad (1)$$

where E = activation energy (cal/mol) and R = gas constant.

In order to use this formula to calculate the slope and subsequently, the activation energy, a computer programme was developed at the authors' laboratory. The temperatures corresponding to 10 weight loss values (in the range 1–18%, depending on the type of sample) were read off from the 3 thermograms for all the samples and used to calculate the slope and activation energy. The av-

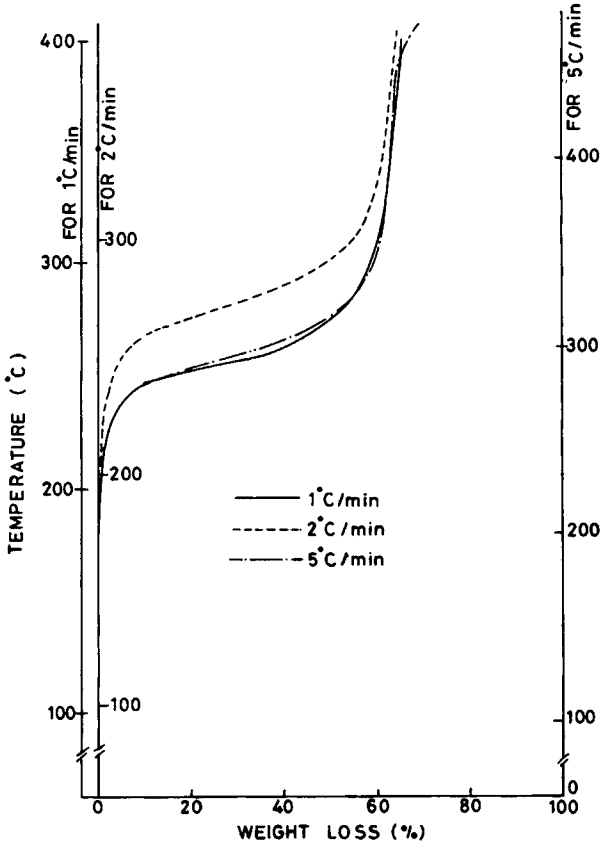

Figure 1 Thermograms for poly(vinyl chloride) (PVC).

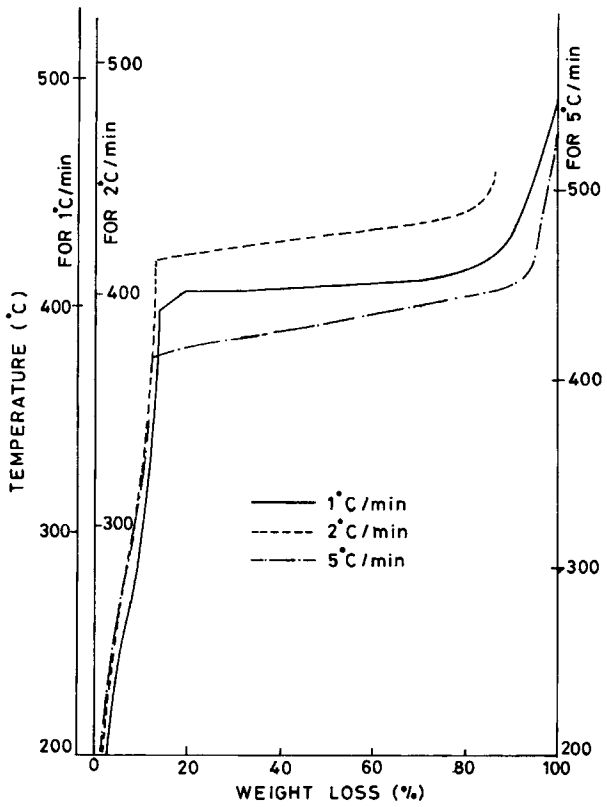

Figure 2 Thermograms for crosslinked poly(ethylene) (XLPE).

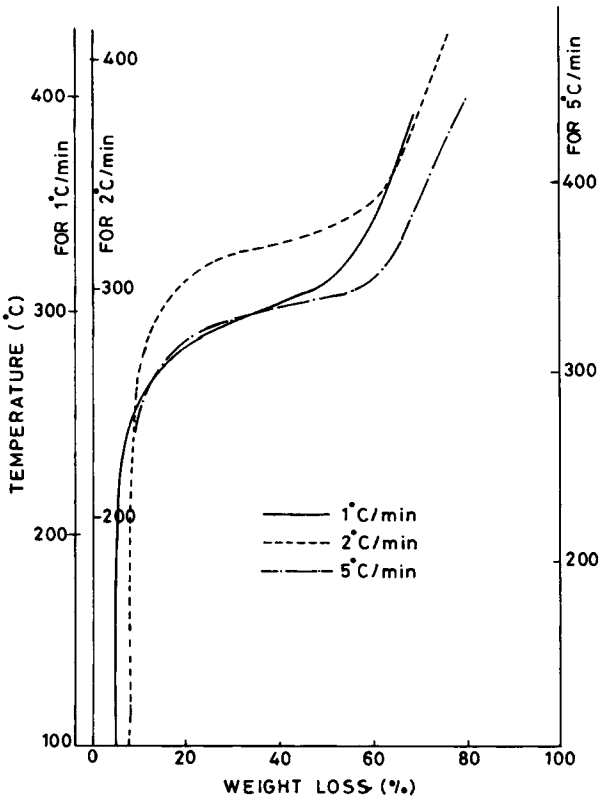

Figure 3 Thermograms for capacitor grade tissue paper.

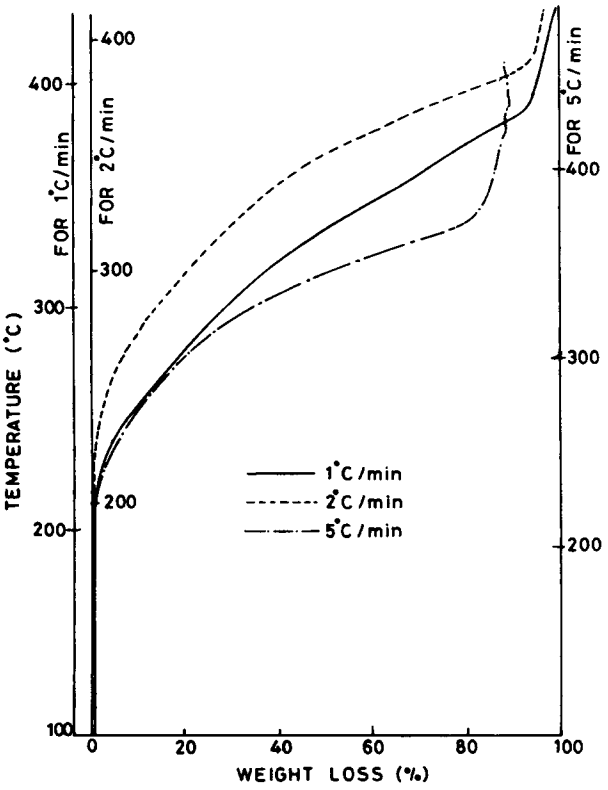

Figure 4 Thermograms for poly(propylene) (BOPP).

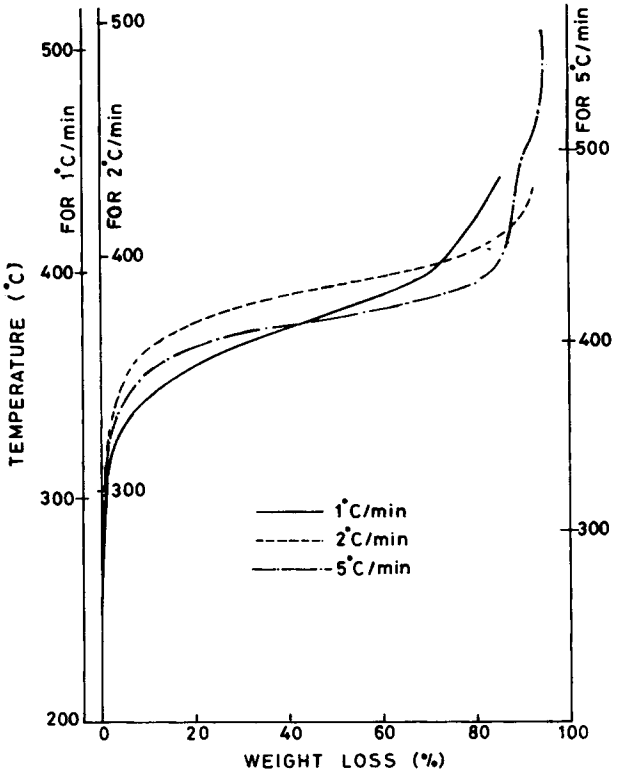

Figure 5 Thermograms for poly(ester) (PET).

erage activation energies (E) thus calculated for all the six materials are given in Table II.

The temperature index was then calculated using the following formula,[4]

$$4.301 = \frac{E}{2.303RT} + \log\frac{Ep(x)}{Rh} \qquad (2)$$

where E = activation energy (cal/mol); R = gas constant; T = temperature index (°K); h = heating rate (°K/h);

Table II Activation Energies of Various Materials

Sample No.	Material	Average Activation Energy (cal/mol)
1	Poly(vinylchloride) (PVC)	25724.7
2	Crosslinked poly(ethylene) (XLPE)	23546.1
3	Capacitor grade tissue paper	29447.6
4	Poly(propylene) (BOPP)	40733.0
5	Poly(ethylene terephthalate) (PET)	30384.4
6	Poly(tetrafluoroethylene) (PTFE)	35176.8

Table III Temperature Indices of Materials

Sample No.	Material	Assigned Class (Maximum Use Temperature, °C)	Temperature Index (Calculated)	Ref.
1	Poly(vinyl chloride) (PVC)	70–85	84	9
2	Crosslinked poly(ethylene) (XLPE)	90	92	8
3	Capacitor tissue paper	105	113	9
4	Poly(propylene) (BOPP)	130	127	9
5	Poly(ethylene terephthalate) (PET)	150	149	9
6	Poly(tetrafluoroethylene) (PTFE)	260	257	9

$x = E/R\theta$; θ = Temperature (°K) obtained from the thermogram of lowest heating rate (i.e., 1°C/min) that corresponds to the endpoint in the thermal endurance curve (conventional method). In the present study, the temperature corresponding to the midpoint of the bent portion in the curve, which is used to calculate the activation energies, is taken as θ. This is based on the earlier work on insulating varnishes.[7] $p(x)$ = a factor to be taken from the standard table.[4] The calculation of temperature index using eq. (2) for one of the materials (PVC) is illustrated below: from Table II.

$$E = 25724.7$$

$$R = 1.987$$

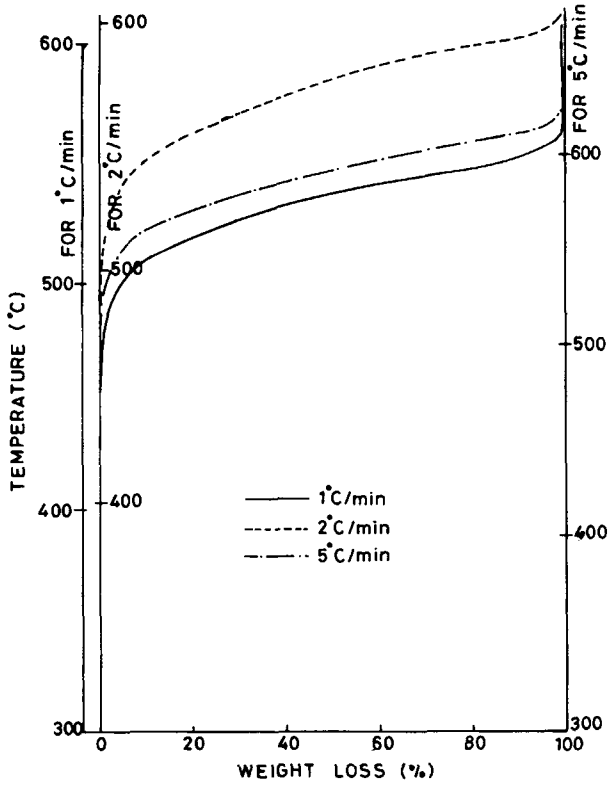

Figure 6 Thermograms for poly(tetrafluoroethylene) (PTFE).

$$\theta = 513°K$$

$$x = \frac{E}{R\theta} = 25.23684;$$

from reference 4:

$$p(x) = 1.61 \times 10^{-14}$$

$$h = 60\ (°K/h)$$

Substituting the above values in eq. (2),

$$4.301 = \frac{25724.7}{2.303 \times 1.987 \times T} + \log \frac{25724.7 \times 1.61 \times 10^{-14}}{1.987 \times 60},$$

On simplification,

$$T = 356.65°K \text{ or } 83.65°C.$$

The temperature indices thus calculated for all six materials are given in Table III.

From the Table III, it is clear that the calculated thermal indices match fairly well with their respective temperature class. Therefore it is concluded that the TGA method can be successfully used to determine the thermal rating of solid insulation materials with fairly good correlation with their expected class. It is suggested that only the bent portion in the thermogram of a material with simple degradation characteristics be considered for determining the temperature index.

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