Thermogravimetric analysis of novel brake friction materials

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Abstract

Thermogravimetry (TG) refers to the measurement of weight changes when a specimen is heated to a scheduled heating program. In general, TG involves the measurement of weight loss as a function of temperature. Derivative Thermogravimetry (DTG) is a method of expressing the results of TG by giving the first derivative curve as a function of temperature. This paper presents the TG and DTG analysis of four novel formulations of brake friction material, namely samples S1, S2, S3, and S4, with reference to the TG/DTG curves obtained for the raw materials (individual ingredients) of the formulations. Due to the inherent variation of constituents, each sample exhibited different TG/DTG curves thus indicating the uniqueness of each formulation. Sample 1 decomposed in three stages. Samples S2, S3 and S4, however, displayed a recognisable TG curve pattern although the rate of decomposition and reaction occurred at different rates and temperatures. Sample S3 registered the lowest total weight loss of 9.28% while the highest total weight loss of 23.8% was observed in sample S4. The TG results revealed notable decomposition between 450°C and 580°C in all samples, corresponding to the degradation of certain ingredients such as barium, Nipol and iron oxide. Coupling TG to other thermal analysis procedures such as the EGA is necessary to detect and ascertain exactly which component is decomposed at each stage and temperature. The selection of suitable friction material depends on the braking conditions and related particular requirements.
Keywords: thermogravimetry, derivative thermogravimetry, brake friction material.

1 Introduction

Friction materials consist of a mixture of fibres, abrasives, lubricants, fillers and organics and therefore regarded as a complex composite [1]. The multi-component nature of any friction materials is not unusual as the brake lining material should be able to withstand very high pressure and thermal stresses besides other considerations such as heat dissipation and noise absorption.

The application of friction materials in braking systems may occur at temperature as high as 800°C albeit 500°C is more common [2]. Chemical reactions and molecular disintegration at elevated temperatures cause decomposition of constituent materials. The physical and chemical changes at interfaces of friction components during braking and the cooling characteristics have been studied extensively [3, 4]. The raw materials composition governs the friction performance of friction materials [5].

In any effort to produce novel friction material, therefore, it is necessary to detect and compile the temperature – weight loss profile of the constituent materials and formulations to be able to predict the braking system performance and response over time. The TG method is apt for this purpose as it is a procedure for measuring changes in sample mass (or weight) with temperature. DTG represents weight loss per minute i.e. rate of reaction.

The weight loss is primarily caused by decomposition of components as heat is supplied to it although the reverse may also occur due to, for example, oxidation. At many instances, the TG/DTG method should be complemented with other techniques for specific reasons [6, 7]. This paper presents the TG/DTG analysis of several formulations of novel brake friction materials for light rail transit brake system.

2 Experimental

The equipment used for TG is a Perkin-Elmer TGA 7. The temperature range used was 50°C - 1000°C at a scan rate of 10°C/min. Sample in powder form weighing not less than 10mg was used. Liquid nitrogen is used to maintain the cooling rate on the furnace.

3 Results and discussion

3.1 Raw materials

Graphite has good resistance to high temperature but begins to oxidise at 500°C [8]. Referring to fig. 1, the tested sample exhibited high resistance to temperature increment up to 785°C, after which the sample begins to loss weight. The rate of weight loss increases constantly and reached 8% per minute at 990°C while the actual weight loss amounted to 34,5%. It has been reported that combustion of graphite leads to the weight loss [9]. The test result shows that the combustion process occurs in a single step between 785°C and 990°C.
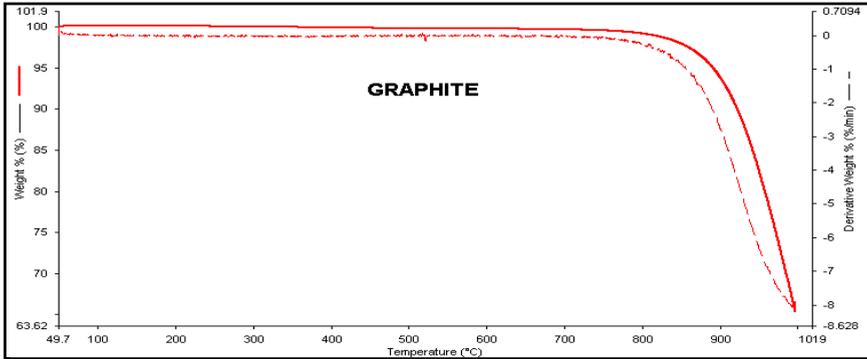
Figure 1:   TG/DTG curve of graphite (raw material).

Fig. 2 shows the TG/DTG thermogram of barium. At 180°C, the sample gained weight to a maximum of 100,065% at rate of 0,25%. The weight gain could be a result of reaction with the surrounding atmosphere but lasted up to 380°C. Above this temperature, weight loss was registered in three stages. The first stage persisted till 650°C while producing sharp peaks on the DTG curve at 546,3^{o}C with a maximum loss at a rate of 0,15%.
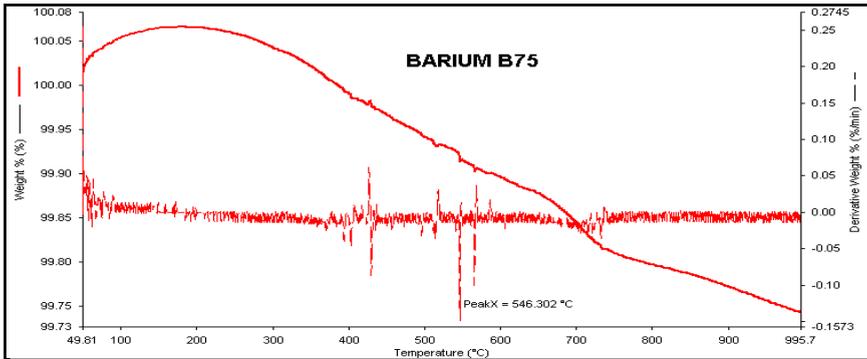
Figure 2:   TG/DTG curve of barium (raw material).

The TG/DTG curves for rubber (fig. 3) represent single stage decomposition. This sample was observed to be stable under heating but only before 245°C. Above this temperature, the sample decomposes in one-step until the temperature reached 550°C after which no weight loss was registered. The DTG graph also indicates that the sample is capable of decomposing at a maximum of 11% per minute at 426,8^{o}C.

The TG/DTG results for Nipol (a proprietary polymer-based material) indicate a 3-stage decomposition as the sample is heated from 49,58^{o}C to 995,8^{o}C as shown in fig. 4. The significant loss of weight is at the second stage for which the temperature bandwidth is between 355°C and 535°C. This sample

can decompose at a rate as high as 32% per minute. The total loss of weight is about 93%. Above 725°C there was no weight loss recorded.
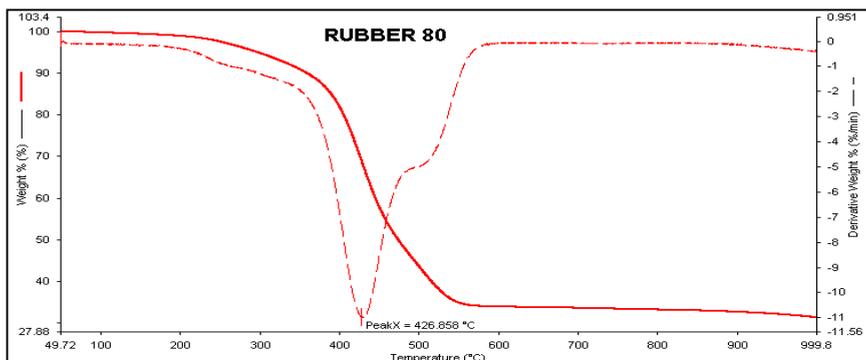

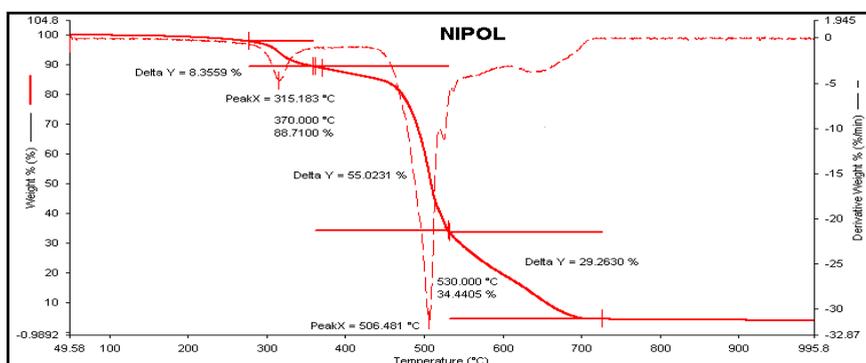Figure 3:   TG/DTG curve of rubber (raw material).

Figure 4:   TG/DTG curve of Nipol (raw material).

Fig. 5 shows the TG/DTG plot for iron oxide. It is observed that at 49.78°C the sample gains weight up to 0.05%. Possible reason for this addition of mass could be the oxidation process during initial heating. The decomposition process of this sample begins at 221.2°C and can be divided into two stages. The first stage commenced at 221.2°C up to 345,6°C. Above this temperature, the sample underwent gradual increment of weight loss (in %; DTG) up to 0.24% at 996.3°C. Based on the DTG curve, there is no complex decomposition of the sample that occurs in this temperature domain (345.6°C - 996.3°C) although the curve indicates a minute step-wise decomposition slightly above 700°C. Sharp peaks (maximum weight loss) were evident between 500°C and 900°C.

3.2 Formulations

Three decomposition stages were detected from the TG/DTG curves as shown in fig. 6 below. This sample decomposed steadily from the onset of heating until

468,2°C. During this period, the total loss of weight is 11,8%. Decomposition of two possible raw materials i.e. barium or rubber may have caused the increment of rate of weight loss between 200°C and 300°C. These possibilities are based on the results of raw materials. This is not conclusive but subject to verification by other thermal analysis technique(s).
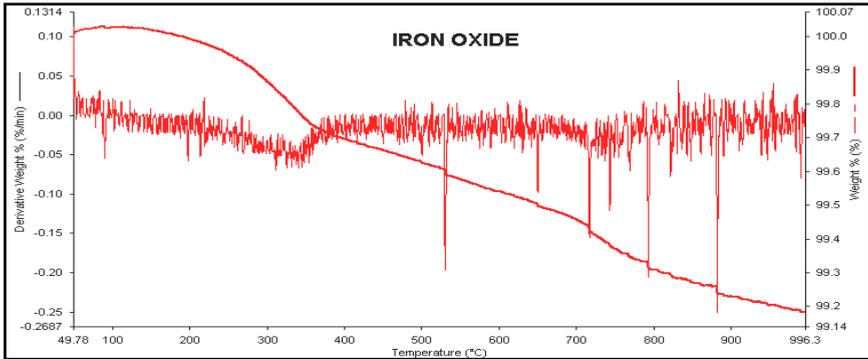

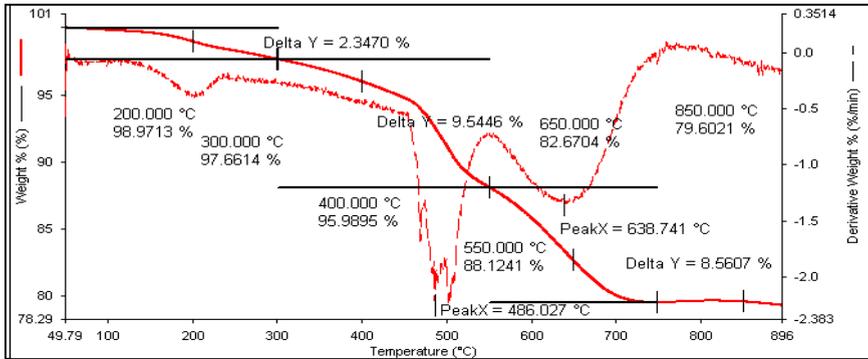Figure 5:   TG/DTG curve of iron oxide (raw material).

Figure 6:   TG/DTG curve of sample S1.

Rapid rate of weight loss occurred between 468,2°C and 486°C. As indicated in the DTG curve, the peak at 486°C corresponds to the derivative weight of -2,3% per minute, being the highest rate registered for this sample across the temperature domain. The rate of decomposition then improved to -1,25% at 550°C. The final stage of weight reduction commenced at 550°C resulting in 8,5% weight loss compared to the preceding stage. Significant weight loss occurred while heating the sample between 300°C and 700°C, contributing to 18,1% reduction. Decomposition eventually ceased at 750°C.

The TG/DTG plot for sample S2 is shown in fig. 7. This sample revealed two stages of decomposition at a rate not more than -0,5% per minute at initial heating. Significant degradation began at 455°C and registered a peak rate of -

0,83% per minute corresponding to 499,6°C. Above this temperature, the decomposition occurred at a slow rate not exceeding -0,5% per minute until the temperature reached 825°C where the rate became -1,05% per minute with continuous lost of weight.

The final stage of decomposition climaxed at 900 with a total reduction of 87% weight compared to the original weight of the sample. In general, the sample decomposed steadily over a temperature range between 400°C and 800°C with rate of weight loss less than -1,5% per minute.

The TG/DTG results for this sample, as shown in fig. 8, indicate somewhat a similar pattern to that of sample S2 but the temperature range where most of the material decomposed is rather narrower compared to sample S2. The first part of decomposition occurred up to 400°C. This was followed by a sudden rise in the weight loss beginning at 450°C. The weight decrement continued steadily until 500°C where the derivate weight achieved a peak -1,05% per minute and began to decrease (i.e. slowing down of the rate of weight loss) until 580°C. This observation corresponds to a weight loss of 4,31%, being the highest loss in a single stage between 450°C and 580°C.
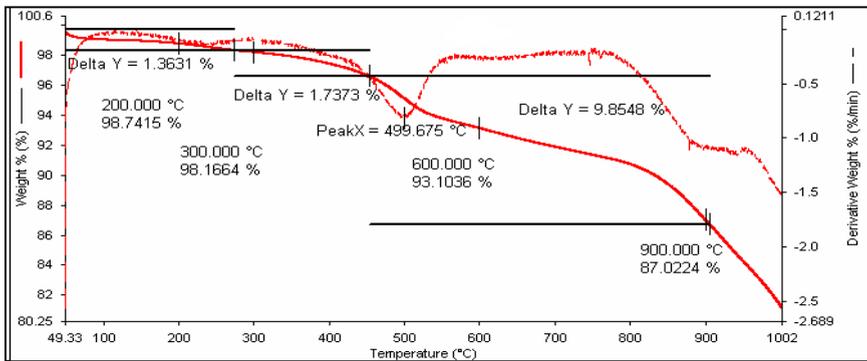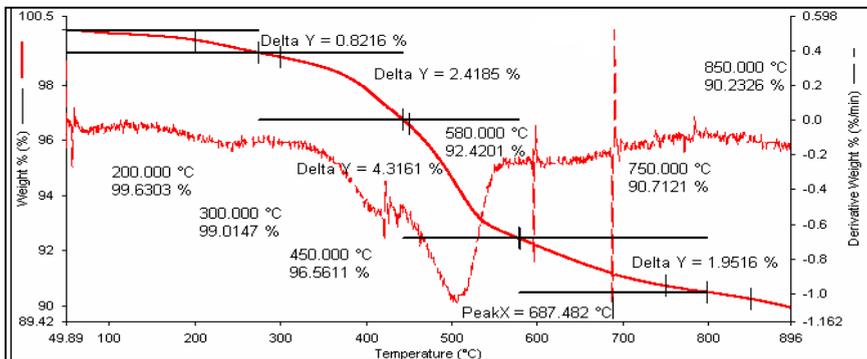

Figure 7:   TG/DTG curve of sample S2.

Figure 8:   TG/DTG curve of sample S3.

The final segment of disintegration of the material was continuous weight loss but at rates that vary unexpectedly at 600°C and 687,4^{o}C. By 750°C, the sample has lost 9,28% weight from its original value. Above that, the combustion of graphite may have probably contributed to further loss of weight.

This sample also performed similarly to samples S2 and S3. It could not be concluded whether the decomposition occurred in one-step based on the TG/DTG curves. But the fact that the rate of weight loss peaked at several temperatures suggest that sample S4 is sensitive to temperature changes especially at lower range (up to 200°C) and around 500°C, the second being very similar to sample S1. Significant increase of derivative weight at 180°C suggests the degradation of the barium content. The second peak at 250°C corresponds to the decomposition of Nipol. A major portion of the sample decomposed between 400°C and 600°C.
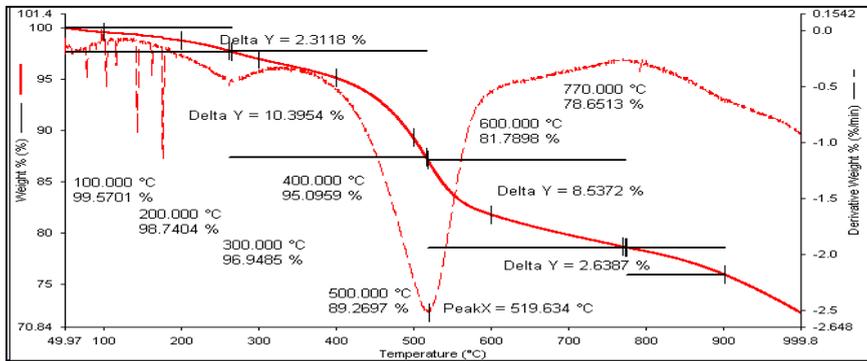

Figure 9:   TG/DTG curve of sample S4.

The maximum degradation rate of this sample is 2,5% per minute at 519,6^{o}C, surpassing the rate for all other samples i.e. S1, S2 and S3. With reference to the raw materials' results (refer figs. 1, 2, 3, 4 and 5 above), this could be due to the decomposition of barium, Nipol and/or iron oxide. Decomposition continued at slower rates up to 900°C signalling the combustion of graphite. Total weight loss amounted to 23,8%, also the highest of all samples mentioned earlier.

4   Summary

Sample S1 decomposed in three stages. Samples S2, S3 and S4, however, displayed recognisable TG curve pattern (two stage decomposition) although the rate of decomposition and reaction occurred at different rates and temperatures. Sample S4 registered the maximum total weight loss of 23,8% at 1000°C while decomposition of sample S2 achieved 13% at the same temperature. S4 is sensitive to temperature changes especially at lower range up to 200°C and around 500°C, the second being very similar to sample S1. Decomposition of samples S1 and S3 attained completion at 900°C with the former registering 11,8% weight loss while the later registered 9,28% loss, both at a rate about –

2,5% per minute. In spite of this, sample S3 exhibited high decomposition rate within a narrower temperature range. The TG results revealed that all samples inclined to decomposition between 450ºC and 580ºC, corresponding to the degradation of certain ingredients such as barium, Nipol and iron oxide. Coupling TG to other thermal analysis procedures such as the EGA is necessary to detect and ascertain exactly which component is decomposed at each stage and temperature. Selection of suitable friction material depends on the braking requirement of particular applications. Future work would include a comprehensive analysis involving determination of braking temperature and desired performance besides comparison of current results against those obtained for the commercial brake pads in tandem with techniques such as the EGA.

Acknowledgements

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