

THERMAL CHARACTERISTICS OF LIGHT RAIL TRANSIT FRICTION MATERIALS

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ABSTRACT

Material specification and operating temperatures are very important parameter for product performance especially for transportation application such as brake pad material. For light rail brake pad, other than strength, wear and friction, thermal stability is also an important property to investigate due to temperature variation during operation. Hence, four sample differences of laboratory formulation are used to study the thermal stability of brake pad material. The thermal characteristic is determined by thermal gravimetric and differential thermal analyser which are initially heated from 30 °C to 1000 °C and are used in combination with the evolved gas analysis. Scanning electron microscope equipped with energy dispersive x-ray are used to determine the element present in formulated brake pad samples. A brake pad is basically a mixture of iron (Fe), carbon (C), fibre, lubricant material and binder system. Some vapours and gases (water, carbon dioxide, methanol, phenol and ammonia) are detected which contributed to the results in the weight change of each sample. These gases are from the phenolic binder. Sample S1 shows a low percentage of weight loss and more stable product compared to other samples. The stability of product materials plays a main role in determining the thermal stability of the samples at the high temperatures.

Keywords: *Brake pads, friction materials, light rail transit, semi-metallic materials, thermogravimetry.*

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1.0 INTRODUCTION

Thermal analysis refers to any technique for the study of materials which involve thermal control. Measurements are usually made with scanning mode for variation or fixed temperature as in isothermal mode. In thermal analysis, changes in weight form the basis of Thermogravimetry (TG) while measurement of energy changes form the basis of Derivative Thermogravimetry (DTG). Both techniques are frequently used to evaluate material properties. TG tells how much the weight sample is lost while DTG tells the reaction is exothermic or endothermic [1]. There is a series of different types of friction materials in the market, which can be classified into the following categories: Semi Metallic (SM), Non Asbestos Organic (NAO) and Sinter Metal. The materials are mainly composed of a relatively large amount of iron powder, some graphite, abrasives, lubricant and filler. The mixture is bonded together with resin. Production of brake pads is typically performed in four steps: mixing of the ingredients, hot pressing of the powder onto the metal back plate, post-curing in a batch oven and finishing by painting, grinding and slotting [2].

When a brake pad is applied, it rubs against the surface of a brake disc and leads to a diminution of the speed of the vehicle. Activation of a brake system on a moving vehicle converts the kinetic energy of the vehicle into heat created by the friction force between the surface of the friction material and the brake disk. The high temperature implies degradation of the material on the surface of the brake pad, with gas evolution as a consequence [3]. During application, a braking system must exhibit high frictional forces and must be capable of absorbing large amounts of energy. High stresses and much heat are produced. The matrix or binder is usually a matrix thermosetting polymer viz., epoxy, phenolic, polyimide or unsaturated polyester. A polymer composite has metallic fibres and carbon fibres which provide strength, friction and thermal conductance. For maximum friction, soft metals with high coefficients of friction are preferred. To avoid gross seizure between brake friction pad and the brake disc surface, lubricants are added. While lubricants prevent gross seizure, they do not prevent local welding and metal transfer. To minimize these, abrasives (often called the frictional component) are added. Since these abrasive components also produce wear, the amount added depends on how much wear can be tolerated in a specific application. An important requirement is thermal stability, which means that the coefficient of friction and the wear rate do not appreciably change up to a specific temperature. Maximum temperatures are around 500°C for PMC (Polymer Matrix composites). The wear-resistant components are added essentially for dry applications. Some of these components such as metal oxides may be formed during manufacturing. Fillers are used to improve manufacturability and to modify properties [4].

Therefore, it is very important to obtain an information and understanding of the physical and thermal properties changes when a friction material is exposed to severe thermal condition. Thermogravimetry analyses with derivative thermogravimetry have been used to evaluate thermal stability of materials.

2.0 METHODOLOGY

This research mainly emphasize on the characteristics of four different laboratory formulated light rail transit brake pad compounds developed by SIRIM. This study will focus on testing of material for thermal and elemental analysis. Analysis from thermal and elemental data will be used to relate with friction and wear behaviour of each formulated light rail transit brake pad. The laboratory of four different formulated light rail transit brake pads must be identified. The two main techniques used are the Scanning Electron Microscope equipped with Energy Dispersive X-Ray Analysis (SEM – EDX) and the thermogravimetry analysis using thermogravimetric analyzer (TG-DTG) which is equipped with mass spectrometric, TG-MS [5].

3.0 EXPERIMENTAL

The technique used in getting the physical property of a substance is measured as a function of temperature while the substance is subjected to a controlled temperature programme. Individual raw material thermal analysis instrument is the Perkin Hammer TGA 7 which gives the result of the TG-DTG curve. The second equipment used for the analysis of the four formulated light rail transit brake pads is Mettler Toledo TGA/SDTA851^o with a small furnace (up to 1600^oC) which gives the result in TG-DTG plus mass spectrometric (MS) curve. This is used to identify the possibility of vapours and gases discharge during the temperature rise. Combination of thermal and evolved gas analysis is highly recommended and used extensively in a variety of materials research initiatives. The thermogravimetric temperature program ran dynamically from 30^oC to 1000^oC at a heating rate of 10 ^oC/min with 50 ml/min flow rate of nitrogen gas.

4.0 RESULTS AND DISCUSSION

4.1 Elemental Analysis

Table 1 shows summary of elements detected Energy Disperse Analysis. In EDX analysis for the sample S1, eleven elements were detected. Carbon (C) and iron (Fe) was found to be dominant in this sample, 41.53 %-wt and 31.36%-wt as compared to other elements copper (Cu) 7.61%-wt, metal antimony (Sb) 2.14 %-wt and barium (Ba) 1.88%-wt. It was observed that the iron powders are in the shape of short fibres and well distributed in the sample. Sb is used in the form Sb₂S₃ that serves as a lubricant to reduce vibrations and to improve friction stability [6]. On the other hand the barium powders are not well dispersed in this formulation and also to other element such as zinc oxide, sulphur, antimony, and copper [7]. Different particles are introduced to stabilize the friction coefficient around a given value: abrasive mineral particles, metallic particles and solid lubricant.

Table 1: Summary of sample elemental compositions detected by EDX

S1		S2		S3		S4	
Element	% Wt						
C K	41.53	C K	34.76	C K	36.34	C K	48.01
O K	11.02	O K	20.3	O K	12.84	O K	16.33
Al K	0.29	Al K	2.24	Al K	0.64	Al K	0.61
Si K	0.44	Si K	3.55	Si K	0.52	Si K	3.7
S K	1.18	S K	2.7	S K	1.43	S K	1.75
Ca K	0.47	Ca K	1.93	Ca K	-	Ca K	2.84
Fe K	31.36	Fe K	18.68	Fe K	38.3	Fe K	16.46
Cu L	7.61	Cu L	2.19	Cu L	2.71	Cu L	-
Zn L	2.06	Zn L	-	Zn L	2.46	Zn L	2.92
Sb L	2.14	Sb L	-	Sb L	1.04	Sb L	-
Ba L	1.88	Ba L	12.43	Ba L	3.72	Ba L	7.39
Mg K	-	Mg K	1.21	Mg K	-	Mg K	-

Ten elements were detected in sample S2. There is no significant difference observed between S2 and S1 except for the weight percentage difference. Carbon is still the dominant element and probability its content in sample S2 is equal to that of sample S1. The content of iron is less in sample S2. The metal antimony (Sb) and zinc (Zn) are not detected in sample S2. The significant difference observed in sample S2 is the presence of magnesium (Mg) which low percentage weight. The existence of copper in the material could be in the form of magnesium oxide is command use Reinforcement fibres to control fade and increase braking effectiveness [7]. The percentage of silica (Si) and sulphur (S) are more compared with sample S1, but their peaks high are different. The percentages of copper (Cu) is less in sample S2. The usage of calcium (Ca) is appearing in EDX spectrum in sample S1 and S2 is coming from Wollastonite.

Calcium (Ca) element is not detected in sample S3. Probably Wollastonite powders are not well dispersed or not used in this formulation. Iron (Fe) and carbon (C) are still dominant element. It is also observed that the percentages of Ba, Sb, and Cu are also more or equal in both sample S2 and S3 but their peak height are different. The Ba content in sample is less in sample S3 compare in S1 and S2. The percentages of Al and Si are higher than those in sample S1 and less than sample S2.

In sample S4, again magnesium (Mg) not detect in EDX spectrum. Copper (Cu) and antimony's sulphide (Sb) also not detected. Carbon (C) and iron (Fe) still dominant in sample. Its content in sample S4 is less compares other samples. The percentage of barium (Ba) is more compare with sample S3 but less compare with sample S2. The percentages of aluminium (Al) still less or equal with sample S3.

4.2 Thermal Analysis (TG-MS) on Different Samples Brake Pads

Figure 1 shows the TG and DTG traces of the sample S1 which was heated from room temperature to 1000°C at 10°C per min in a nitrogen atmosphere. It can be seen clearly in DTG curve, the sample S1 has six different regions of weight loss at certain temperature range during the heating process (Table 2). The first region (30°C - 115°C) corresponding to a weight loss of 0.07% representing the loss of the absorbed water. The second region (115°C - 230°C) representing the volatility from adhesive and degradation of sulphur content in a sample. Sulphur will be decomposing after boiling temperature achieved around 200°C (Periodic Chemical Table). During the second region, the weight loss was very low is about 0.01%. The third region (230°C - 403°C), there is a decrease in mass of around 2.3%. The maximum rate of degradation is found at temperature 363°C. On this region only organic materials such as binder is degrade [8]. Probability it is happen because of the reaction the gasses evolved between organic materials itself.

Between the second and third region, oxidation process was detected. In the fourth region (403°C - 488°C) another decrease of mass are detected which is around 1.6%. The quick of decomposition rate occurred between 230°C to 403°C is cause by intermediate unstable product form. In this temperature region, two possible molecules are release as shown in Figure 2. The possible molecules released are water and carbon dioxide. Base on data obtained by Chang and Tackett (1991) in gases evolved during the phenolic resin TG-MS experiment, they are found at temperature 420°C - 580°C gases from carbon dioxide and water are released. The data was supported by Ramousse *et al.* (2001). Ramousse *et al.* (2001) found two gases which carbon dioxide and water are released on his experimental data. From his explained, the emanation of carbon dioxide and water is corresponding to a weight loss in mass and an endothermic peak.

The emanation of water is from 230°C and 450°C. The emanation of water is corresponding to the decomposition of binder. The emanation of carbon dioxide is due to the combustion of carbon. The combustion of carbon seems to take place between 250°C and 850°C [9]. The emanation of carbon dioxide also corresponds to the decomposition of phenolic binder [8]. The fifth regions (488°C - 688°C) there were found another decrease of mass around 1.5%, accompanied by large exothermic peaks. In this temperature range, the probability the weight loss is degradation of the phenolic resin by oxidation into volatile elements and degradation of Sb₂S₃. Different reactions such as cracking, dehydrogenation and dehydration are involved in the resin degradation process [7]. In this region also the two possible molecules which is water and carbon dioxide are release [8, 9]. The gases is continues release until in six region. The six regions (688°C - 1000°C) another decrease of mass is around 5.3%. The degradation of ingredient such as graphite is activated around 750°C to 800°C [10] at the same time as continues activity of thermal degradation of Sb₂S₃. Presenting of iron powder is not shown in TG and DTG curve even in EDX analysis shown is element is detected. On this region, a stable of product form is can be seen on TG and DTG

curve. According Bulthé *et al.* (2008) at higher temperature, the decreases of water production is underlines degradation of the resin and it will finish. Therefore the carbon dioxide production is revealing the degradation of the other constituents of the material. The total residues for sample S1 is about 89.7% and still undergo to degrade above 1000°C.

The TG and DTG curve shows in Figure 3 for sample S2 also experience six different regions. The first (30°C - 128°C) and second region (128°C -148°C) gives a low of weight loss. This region eliminated because of water adsorbed and volatility of adhesive. The third region (148°C - 402°C), the weight loss is high compared to sample S1 in this temperature range which is around 2.6%. Again, the different of decomposition rate is probability related during sample preparation. An intermediate unstable product is form on this temperature range. It can be seen clearly through TG and DTG curve. On the fourth region (402°C - 502°C) water and carbon dioxide gases are detected as shown in Figure 4, which corresponding of weight loss about 1.76%. Again, the water and carbon dioxide gases detected on fifth region (502°C - 613°C), corresponds of weight loss is about 1.11%. The weight loss is due of decomposition of binder and carbon. The higher decomposition rate for sample S2 is occurred at region's six (613°C - 1000°C) which is around 10%. It may be due of decomposition of aluminium and carbon which detected on EDX analysis. The trend of curve also shown the sample S2 still undergo to degrade above 1000°C. There is still no having metal compound presence to show the oxidation process until of heating process. The total of weight loss in mass is around 15.68%.

Sample S3 shows the TG and DTG in Figure 5, traces show the trend of curve as in sample S1. At between temperatures 100°C - 200°C, the exothermic peak is detected. In sample S3, the four different regions can be clearly seen. The maximum degradation is occurred at temperature 438°C. The first region (30°C - 128°C) gives a low of weight loss about 0.02% due to volatility of adhesive. The second region (185°C - 513°C) is corresponding of the weight loss about 6.26%. In this region, a stable product is found and can be seen clearly on TG and DTG curves. It is probability only one element is degraded. It was happen probability the organic material which can easily decompose below 600°C. Phenolic binder is decompose between 230°C and 450°C and combustion of carbon is take place between 250°C and 850°C [9]. The data is supported by two of gases evolved which is water and carbon dioxide as shown in Figure 6. About 1.5% of weight loss is detected on third region (513°C - 653°C), accompanied by endothermic peak. Water and carbon dioxide gases are detected again corresponds due of the combustion of carbon, binder and other polymers material can be decompose at this temperature. At fourth region, about 8% of weight loss is detected. The TG and DTG curve shown that the stable of product form. Sample S3 shown that the elements are contribution is stable. The total residue for sample S3 is about 84%.

Figure 7 is shown the sample S4 experienced with intermediate unstable form. The rate of degradation is occurred in a short period compare with sample S1, S2 and S3. From TG and DTG curve, the sample S4 experiences six different regions. The first region (30°C - 197°C) corresponds to a weight loss of 0.39% representing the loss of the absorbed water and curing of the phenolic resin. The

second region (197°C - 409°C) with the weight loss 1.94%, signifies the continue curing phenolic resin and the thermal degradation of polymer. The third region (409°C - 484°C) with the weight loss is about 1.2%. The fourth region (484°C - 654°C) with the weight loss is about 2.6% corresponding of decomposition of binder and combustion of carbon as shown in Figure 8. The water and carbon dioxide gases are detected around this temperature. The intermediate unstable form was occurred from second region until fourth region. The fifth region (654°C - 789°C) with the weight region is about 4.7% and also corresponding of the emanation of water from binder and combustion of carbon. The six regions (789°C - 1000°C) with the weight loss are about 8.4%. The total residue is about 81.1%.

The TG and DTG results for sample S1, S2, S3 and S4 showed the production of water was due to the reaction of chemical degradation of phenolic resin. The production of water and carbon dioxide is a due to the degradation of the phenolic resin, carbon compound and others ingredients. Hence, this degradation is brought about the local temperature reached in the contact and the mechanical action in friction [7]. The detection of the gases released did not give any information regarding of oxidation of iron. According Ramousse *et al.* (2001), the gases released did not show any differences between coal and graphite. The mass losses cannot be due respectively to the coal and graphite as the mass losses do not corresponding to any appropriate amount.

5.0 CONCLUSIONS AND RECOMMENDATION

- i. The combination use of scanning electron microscopy plus energy dispersive spectrometry (SEM + EDX) and thermogravimetry - mass spectrometry (TG -DTG-MS) are carried out.
- ii. In EDX result show, carbon are dominated in sample S4 compare to other sample S1, S2 and S3. However iron is detected dominate in sample S3 in EDX spectrum. Due of limitation factor, XRF analysis also show the inorganic elements are constitutes of the brake pads. Base on composition elements detected by EDX, the brake pad materials are categorized as a semi metallic type of friction material.
- iii. The thermal results indicate the oxidation process is not occurring on four different samples until the end operating temperature. It will be occurred when the operating temperature are exceed than 1000°C. The materials start decomposed at 100°C. It is due of the extraction of water compounds in samples. After extraction of water, the binder materials start to decompose and followed by others materials until end of temperature tested. Some of gases (water, carbon dioxide, methanol, phenol and ammonia) are detected evolved to contribution in results of weight change in each samples. These of gases are from phenolic binder. Sample S1 is show low percentage of weight loss and more stable of product form compare to other sample. The stability of product materials is main role to determine the thermal stability of the samples which can established at the high temperature.

Table 2: Table below shows TG-MS result of samples S1, S2, S3 and S4 heating in N2 from 30°C to 1000°C at heating rate of 10°C/min

TGA & DTG											
	1 st Weight Loss		2 nd Weight Loss		3 rd Weight Loss		4 th Weight Loss		5 th Weight Loss		Residue
	T _r and T _{min} (°C)	Wt Loss (%)	T _r and T _{min} (°C)	Wt Loss (%)	T _r and T _{min} (°C)	Wt Loss (%)	T _r and T _{min} (°C)	Wt Loss (%)	T _r and T _{min} (°C)	Wt Loss (%)	Residue (%)
S1	T _r = 30-115	0.07	T _r = 115-230	0.01	T _r = 230-403	2.27	T _r = 403-488	1.61	T _r = 488-688	1.49	89.7
	T _{min} = 79		T _{min} = 210		T _{min} = 363		T _{min} = 428		T _{min} = 568		T _r = 688-1000
S2	T _r = 30-128	0.03	T _r = 128-145	0.05	T _r = 145-402	2.61	T _r = 402-502	1.76	T _r = 502-613	1.11	84.62
	T _{min} = 82		T _{min} = 137		T _{min} = 367		T _{min} = 427		T _{min} = 557		T _r = 613-1000
S3	T _r = 30-185	0.02	T _r = 185-513	6.26	T _r = 513-653	1.49					84.53
	T _{min} = 106		T _{min} = 438		T _{min} = 573						T _r = 653-1000
S4	T _r = 30-197	0.39	T _r = 197-409	1.94	T _r = 409-484	1.22	T _r = 484-654	2.55	T _r = 654-789	4.67	81.13
	T _{min} = 76		T _{min} = 374		T _{min} = 439		T _{min} = 559		T _{min} = 759		T _r = 789-1000

T_r: Weight Loss Temperature Range

T_{min}: Min Temperature Range (DTG)

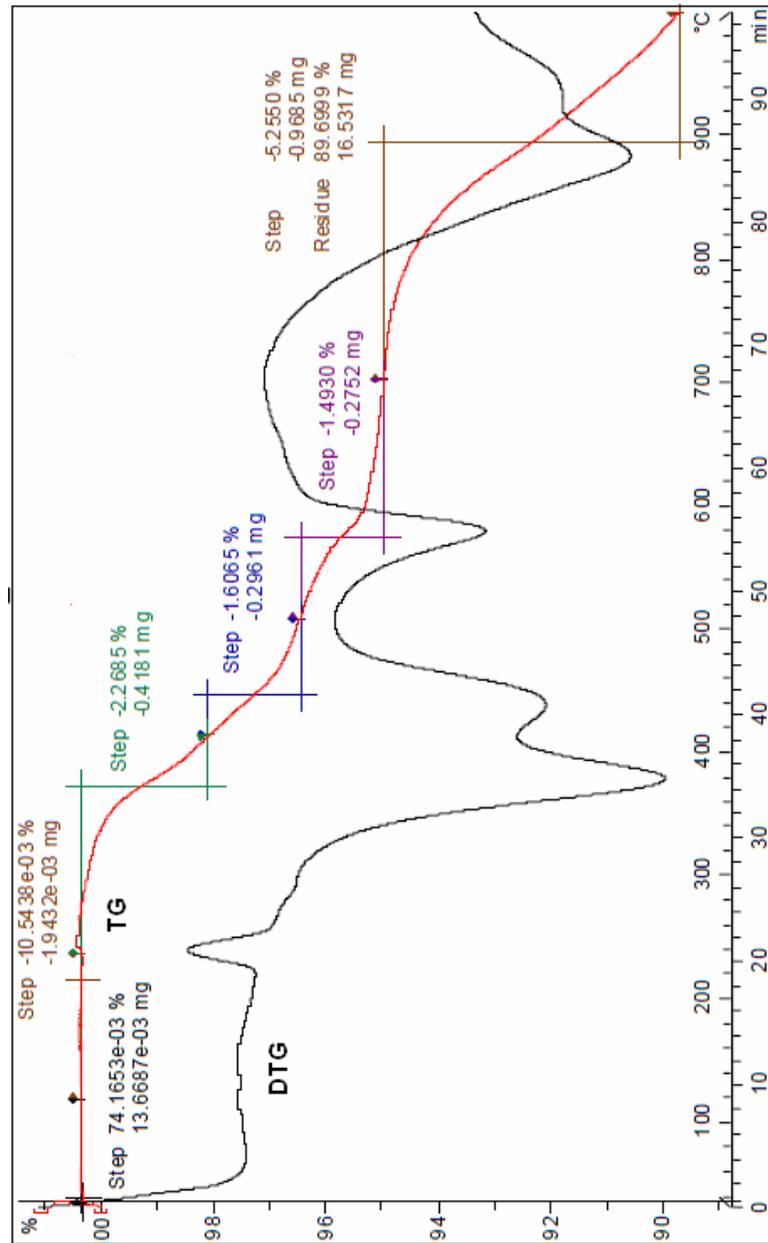


Figure 1: TG and DTG weight loss curve of sample S1

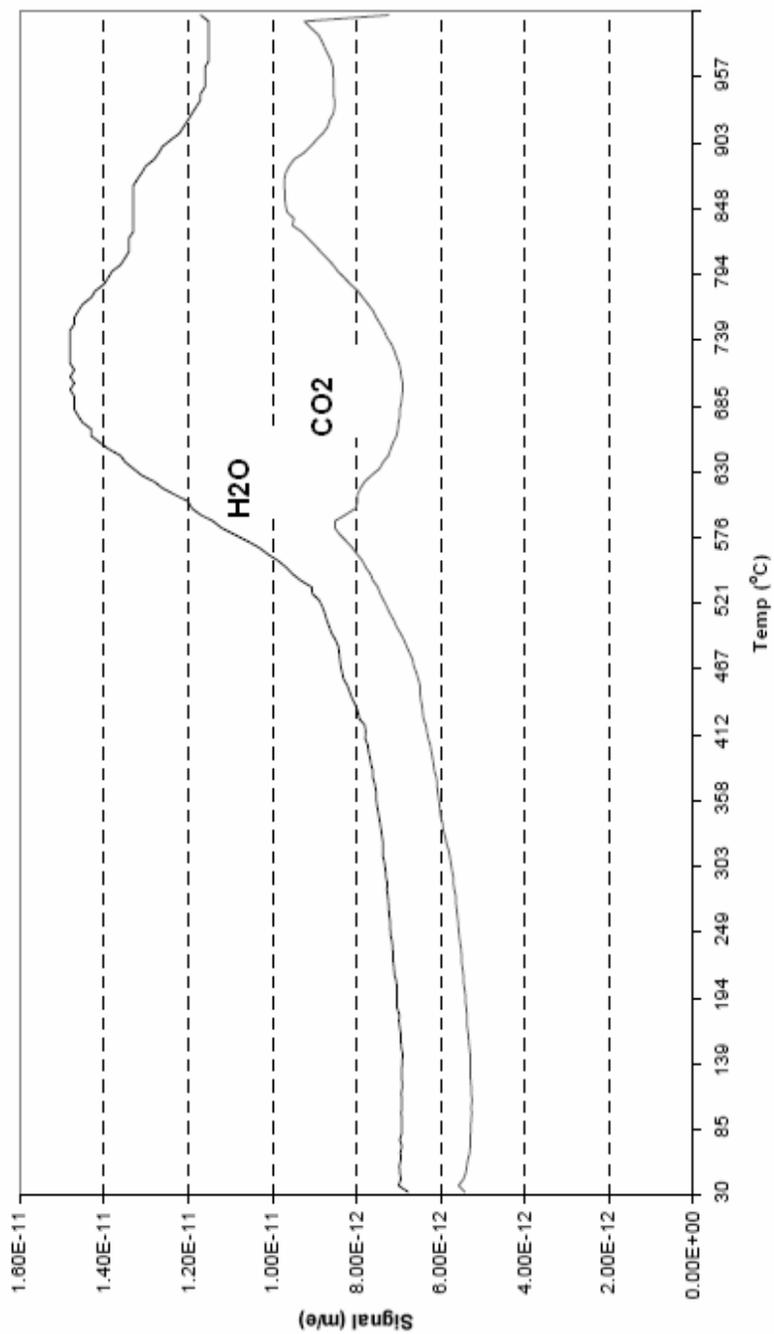


Figure 2: MS signal of sample S1 heating in N2, from 30°C to 1000°C at heating rate of 10°C/min.

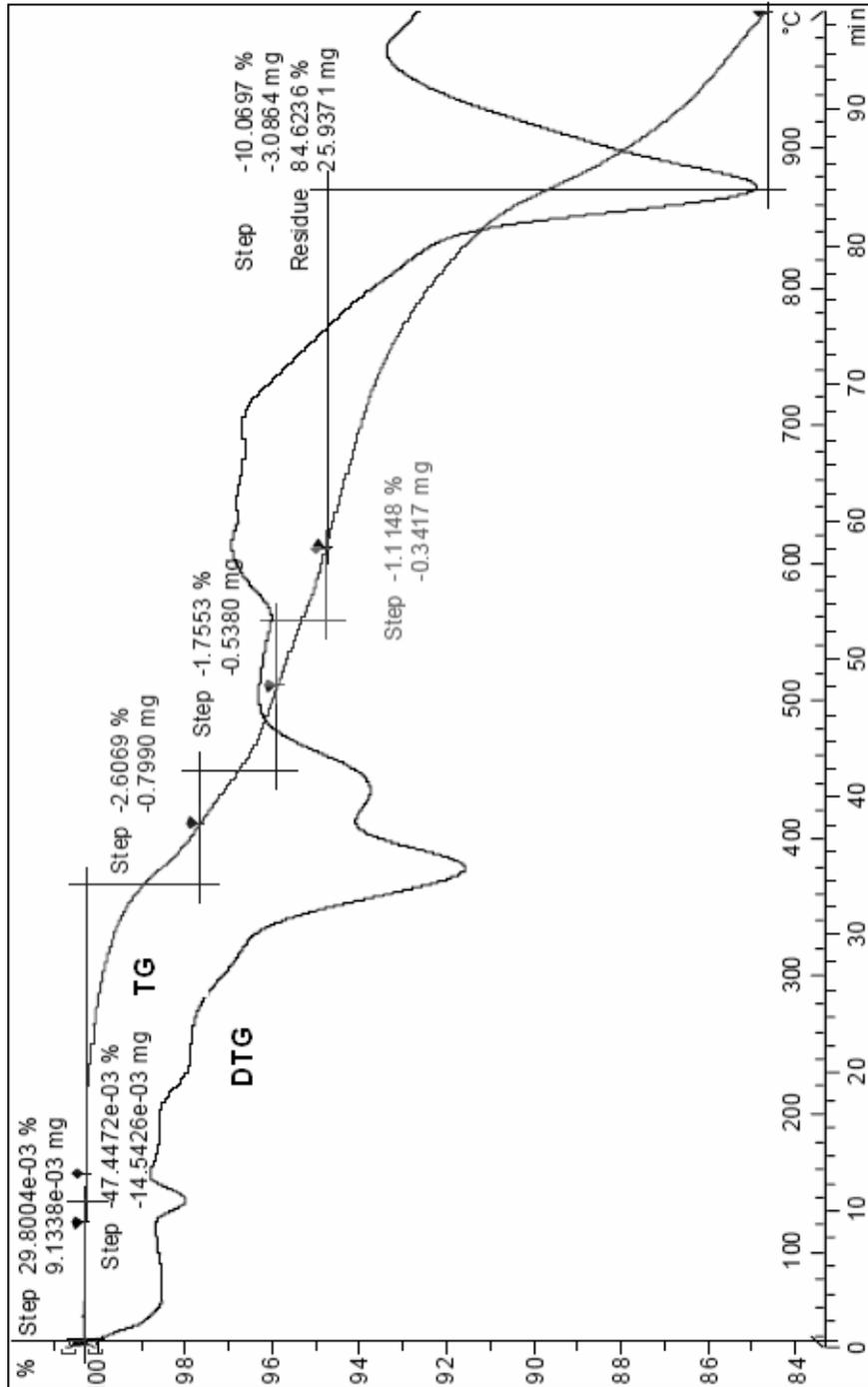


Figure 3: TG and DTG weight loss curve of sample S2

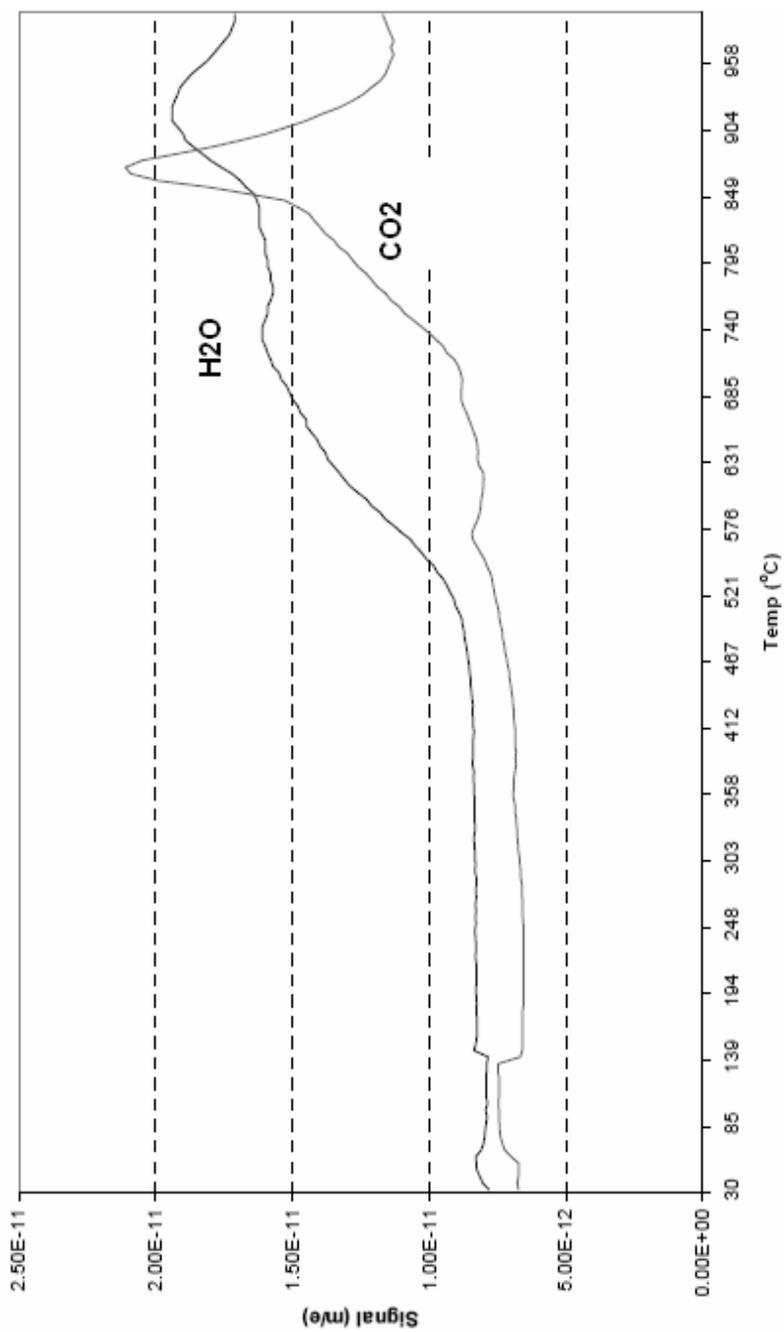


Figure 4: MS signal of sample S2 heating in N2, from 30°C to 1000°C at heating rate of

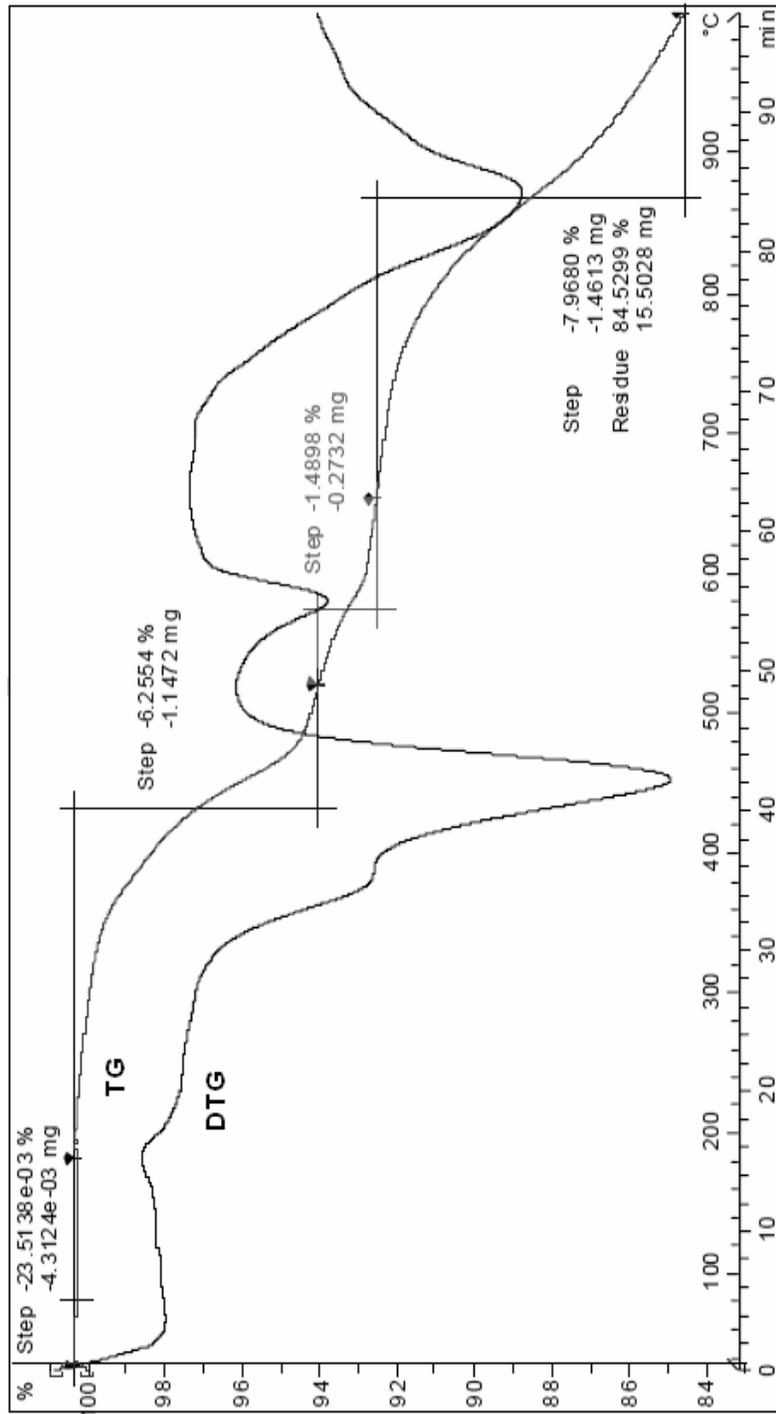


Figure 5: TG and DTG weight loss curve of sample S3

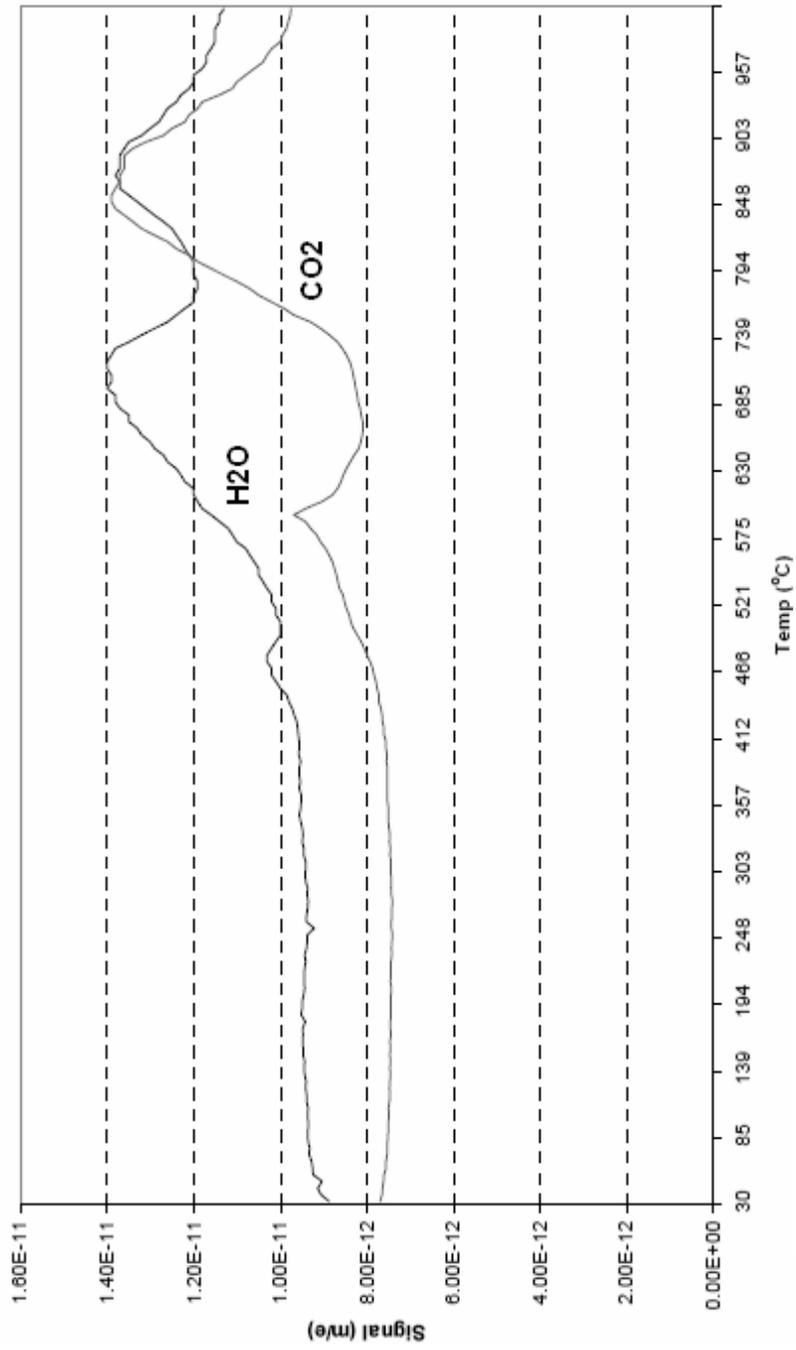


Figure 6: MS signal of sample S3 heating in N₂, from 30°C to 1000°C at heating rate of 10°C/min.

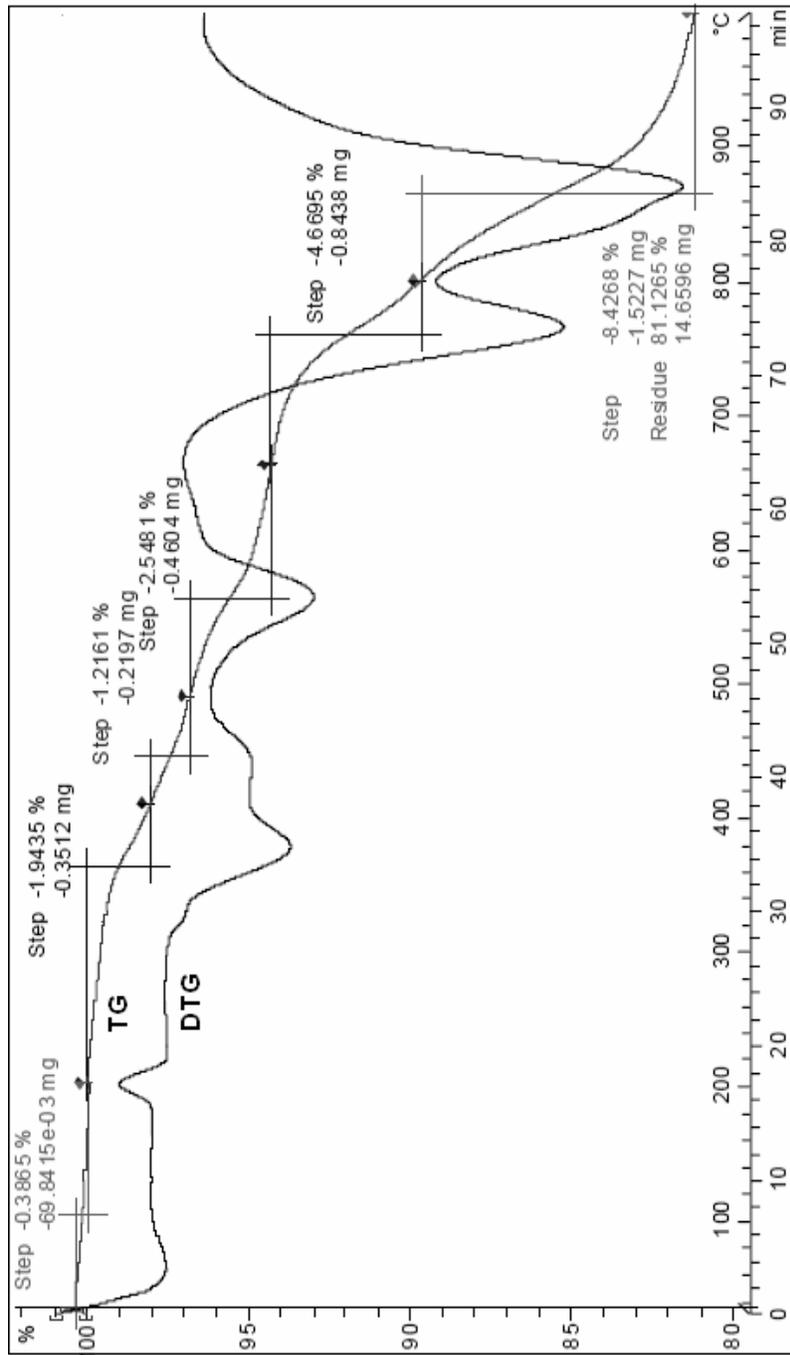


Figure 7: TG and DTG weight loss curve of sample S4

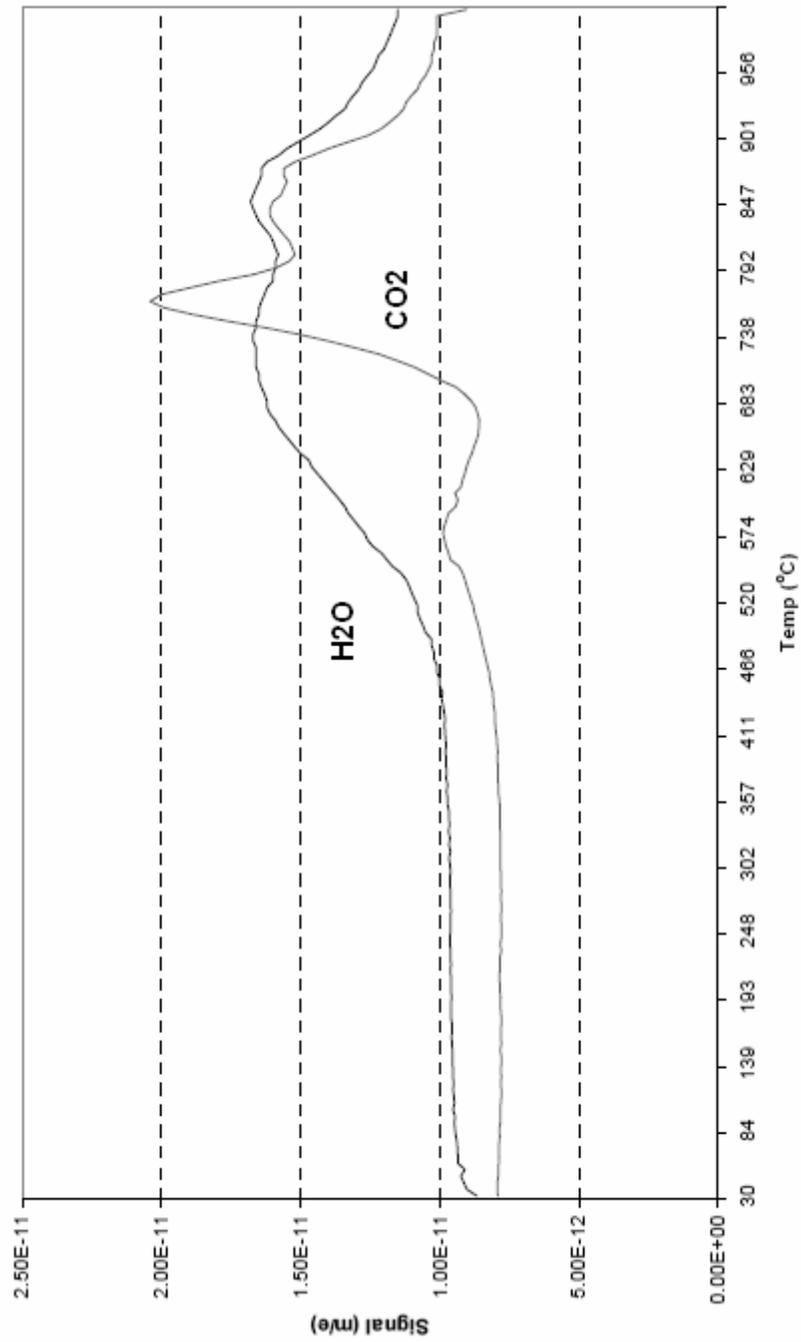


Figure 8: MS signal of sample S4 heating in N₂, from 30°C to 1000 C at heating rate of 10°C/min.

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REFERENCES

1. Keattch, C. J. and Dollimore, D., 1975, *An Introduction to Thermogravimetry*, Heyden, London, (2nd Ed).
2. Selamat M. S. *et al*, 2006, Comparison Method for Producing Friction Material Pad Through Powder Metallurgy Technique, Brake Friction Material Book, *Pusat Penerbitan Universiti (UPENA) Universiti Teknologi MARA*, 29 - 34.
3. Giri N.K., 2004, Automotive Technology: *Braking System*. Khanna Publisher, 28, 1269 – 1349.
4. Eriksson, M., Bergman, F and Jacobson, S., 2002, On the Nature of Tribological Contact in Automotive Brakes, *Journal of Wear*, 252, 26–36.
5. Ramousse S., Hoj J.W. and Sorensen O.T., 2001. Thermal Characterisation of Brake Pads. *Journal of Thermal Analysis and Calorimetry*. 64, 933–943.
6. Üexkull *et al.*, 2005, Antimony in Brake Pads - A Carcinogenic Component. *Journal of Cleaner Production*, 13, 19–31.
7. Bulth'e *et al*, 2008, Mechanical and Chemical Investigation of the Temperature Influence on the Tribological Mechanisms Occurring in OMC/Cast Iron Friction Contact. *Journal Wear*, 264, 815–825.
8. Blau, P., 2006, Compositions, Functions and Testing of Friction Brake Materials and Their Additives. *Technical report ORNL/TM-2001/64*. Oak Ridge National Laboratory, 29; available through NTIS, Springfield, Virginia.
9. Cherng Chang and Tackett Juanita R., 1991. Characterization of Phenolic Resins with Thermogravimetry - Mass Spectrometry. *Thermochemica Acta*. 192, 181-190.
10. Cho *et al.*, 2006. Tribological Properties of Solid Lubricants (graphite, Sb₂S₃, MoS₂) for Automotive Brake Friction Materials. *Journal Wear*, 260 855–860.