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Thermal Analysis of Rubberwood

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The thermal behaviour of rubberwood in an inert environment was examined using differential thermal analysis (DTA). The resultant DTA trace contains a number of peaks, both endothermic and exothermic, attributed to degradation of the individual wood components. The effects of particle size of the wood, the gas flow rate, the heating rate and fungal infestation on the thermal response of the material were investigated. Reducing the gas flow rate has almost the same effect as increasing the particle size of the wood. A comparison was also made between the thermal behaviour of rubberwood and those of other wood species.

Rubberwood is perhaps the most common fuelwood in Malaysia. It is widely used as a domestic fuel in the rural areas and as a source of energy for the drying of agricultural commodities (including rubber), bricks and related products, sawn timber and other materials. It is also converted into charcoal for use in steel production and in certain areas, as a source of fuel.

Although rubberwood has long been used as fuel, very little information was available on its thermal degradation characteristics. These were investigated in a study using differential thermal analysis (DTA) and thermogravimetry (TG), in both oxidative and non-oxidative environments. The analyses were carried out in a self-designed thermal analyser, which allows testpieces of up to around 20 mm in diameter to be used, compared with only a few millimetres in the case of most of the ready-made apparatus. For comparison, a few local wood species were included in the study. Results of DTA and TG analyses in an oxidative environment have been reported[1]. In this paper, the results of DTA of rubberwood in an inert atmosphere are presented and discussed.

EXPERIMENTAL

Apparatus

This has been described in an earlier publication[1].

Materials

The wood of four old rubber trees, one each from clones Tjir 1, PR 107, RRIM 605 and RRIM 623 were used in the investigation. They were obtained from the RRIM Experiment Station at Sungai Buloh, in the form of trunk sections of approximately 50 mm thick. These trunk sections were divided into two similar lots, one of which was immediately oven-dried at around 80°C while the other lot was kept in a poorly ventilated and humid place for two weeks (during which time the wood became mouldy) before being oven-dried. For ease of reference, the first lot is referred to as 'fresh' wood and the second lot as 'mouldy' wood. The densities of the various wood pieces at 8% moisture content are shown in *Table 1*.

For particle sizes below 4 mm in diameter, the test samples were prepared by passing chips through a hammermill and separating the wood particles into various fractions with a set of Endecotts test sieves. Testpieces of larger diameters were cut out directly from a disc of around 13 mm thick using the appropriate hole saws and were cylindrical in shape.

The wood from the Tjir 1 tree was used in the main investigation while those from the other trees were used for comparison. Unless otherwise stated, the test samples were derived from the fresh wood sections and were oven-dried at 105°C overnight before use.

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TABLE 1. DENSITIES OF FRESH AND MOULDY WOOD

Tree	Density (kg/m^3) Fresh wood	Mouldy wood
Tjir 1	647	619
PR 107	694	624
RRIM 605	655	581
RRIM 623	627	571

The local hardwood species used for comparison were obtained from the Forest Research Institute of Malaysia (FRIM) at Kepong, in the form of hand samples measuring 75 × 100 × 18 millimetres.

Procedure

The experimental set-up is shown in *Figure 1*. The reference material consisted of 5 g of aluminium oxide of particle size −100+240 mesh. After the test sample had been dropped onto the sample bed, nitrogen (of 99.995% purity) from a gas cylinder was passed through the two tubes with a combined flow rate of 0.8 litre per minute. Following this, the reactor was heated at a rate of 5.5°C per minute. While the reference material was fluidised from the outset, the test sample remained stationary throughout the run. The reference bed temperature and the temperature difference between the two beds were measured using 1-mm diameter chromel/alumel thermocouples and were recorded continuously on a hot-pen recorder. The experiment was stopped when pyrolysis of the wood had been completed, as indicated by the DTA trace levelling off. This usually occurred above 500°C.

RESULTS AND DISCUSSION

Shape of a Differential Thermal Analysis Curve

Figure 2 shows the DTA curve of the fresh wood of Tjir 1 of particle size −36+72 mesh. The sample size used was 3 grammes. The curve exhibits the following features:

- A weak endotherm between 130°C and 155°C *(A)*
- Part of an endotherm from around 155°C to 240°C *(B)*
- An exotherm peaking at 320°C *(C)*
- An endotherm peaking at 360°C *(D)*
- An exotherm peaking at 400°C *(E)*
- A week endotherm at around 490°C *(F)*.

Only *Endotherms A* and *F* exist as individual peaks; the rest overlap to a certain degree with the adjacent peaks.

Interpretation of the DTA curve had been made by Tan[2], based on the results of further investigations and also on the information available in the literature[3-7]. *Endotherm D* and *Exotherm E* were shown to be due to the thermal decomposition of cellulose and lignin respectively. *Endotherms A* and *F* seemed to be attributed to the extractives while *Endotherm B* and *Exotherm C* appeared to be due to a combination of the extractives and the hemicelluloses.

Visual observations of the sample were made in a repeat run. It was noted that:

- An aromatic odour was emitted from the sample tube from about 145°C.
- Smoke began to be emitted from around 190°C and its intensity increased with increasing temperature.
- At 220°C, the sample had turned slightly brownish and the aromatic odour was still present.
- The sample became darker with increasing temperature. At around 290°C, its view was totally blocked by the smoke.
- Visibility was partially restored at 390°C, by which temperature the sample had turned into char.

Thus, it is quite clear that charring of the wood took place mainly between 290°C and 390°C, which corresponds to degradation of cellulose and part of lignin.

Effect of Particle Size

The effect of particle size on the thermal behaviour of rubberwood was examined using

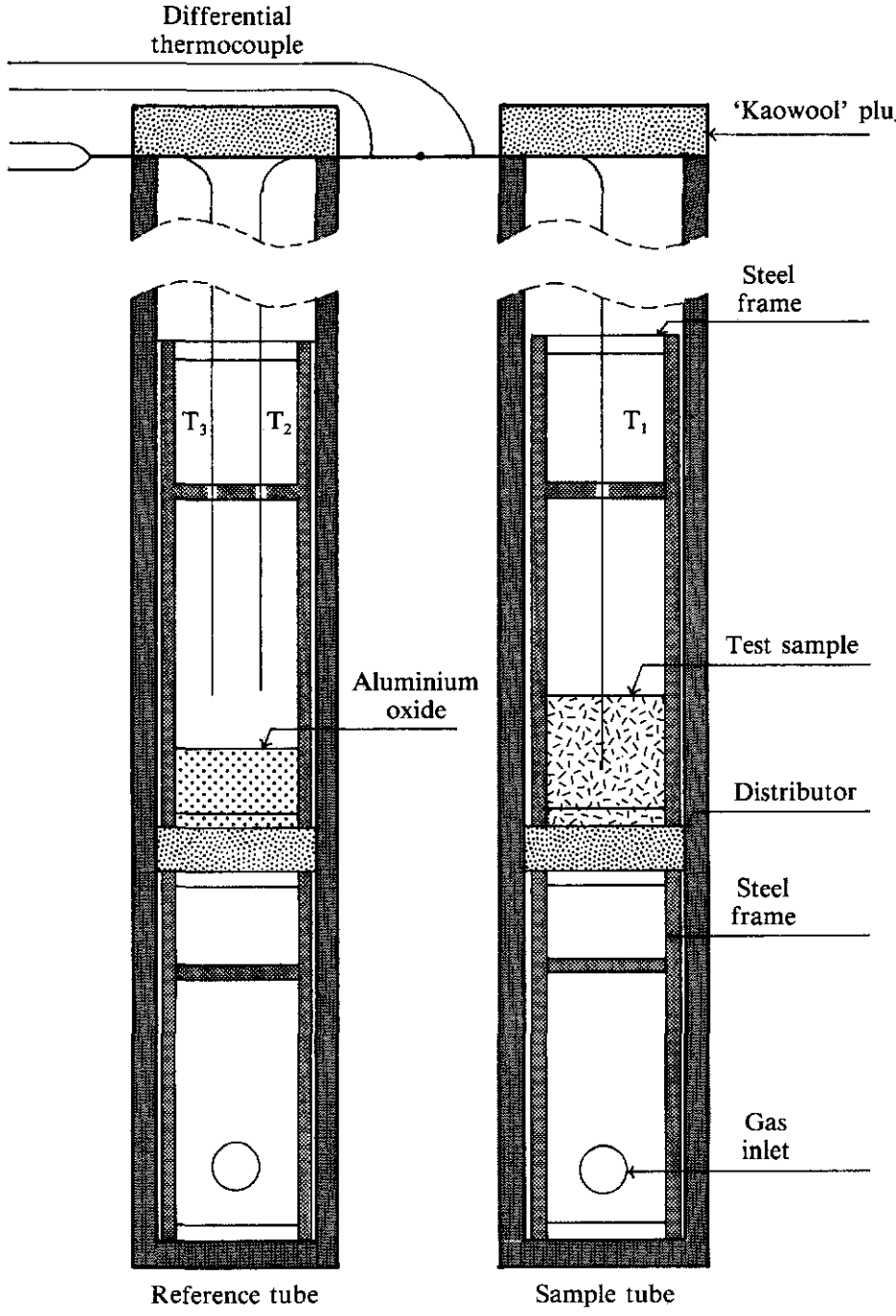


Figure 1. Experimental set-up.

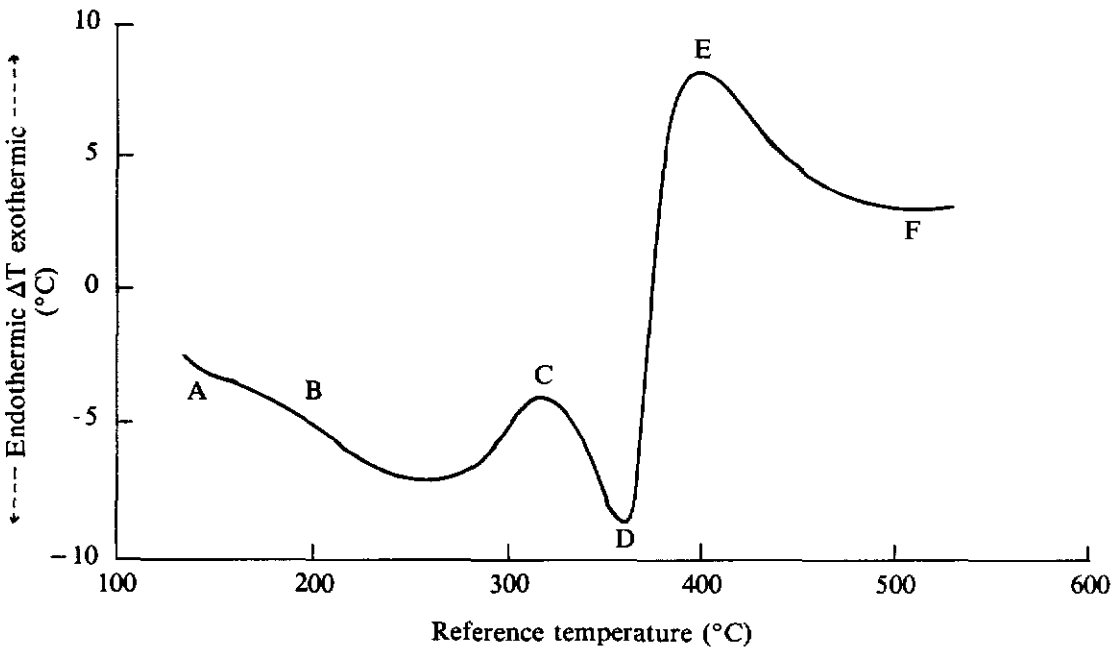


Figure 2. DTA curve of rubberwood of particle size −36+72 mesh.

3-g samples. The particle sizes used ranged from <150 mesh to 10 mm in diameter. As shown in *Figure 3,* the DTA curves of the <150 mesh, −36+72 mesh and −10+18 mesh particles are almost similar in shape, except for the magnitude of *Endotherms A* and *F*. They are, however, quite different from those of the 5 mm and 10 mm diameter fractions, which contain an extra exotherm between 340°C and 380°C *(G)* and whose *Exotherm C* is relatively large in size.

Additional runs were carried out using a single testpiece with the thermocouple's sensing tip being placed within the wood through a 1.5-2 mm diameter hole of depth 7 millimetres. The DTA traces of 5 mm, 10 mm and 19 mm diameter pieces are fairly similar in shape *(Figure 4);* the most glaring difference being in the relative size of *Peak G* which increases with particle size, apparently at the expense of the exotherm to its right, *i.e. Peak E*. There is hardly any difference between the DTA curve of the 5 mm diameter piece and the one obtained earlier using a 3-g sample. In the case of the 10 mm diameter size group, the resolution of the reaction peaks is much better using this technique that the one used earlier (in which the thermocouple is surrounded by the testpieces), although there are no basic differences between the two curves obtained. Likewise, the reaction peaks of the 19 mm diameter piece are well-defined. Thus, in the DTA of wood of larger particle sizes, there is a clear advantage in using a single testpiece with the thermocouple tip placed inside it.

Further analyses were performed on 10 mm and 19 mm diameter single testpieces with a portion of their water-soluble extractives removed by boiling or soaking in water. From the DTA curves obtained *(Figure 5),* it is evident that *Exotherm G* is attributed to the water-soluble extractives, directly or indirectly. In other words, the peak could either result from the thermal degradation of the extractives or reactions between the pyrolysis products of the extractives and those of the other components. What is clear is that it masks part of

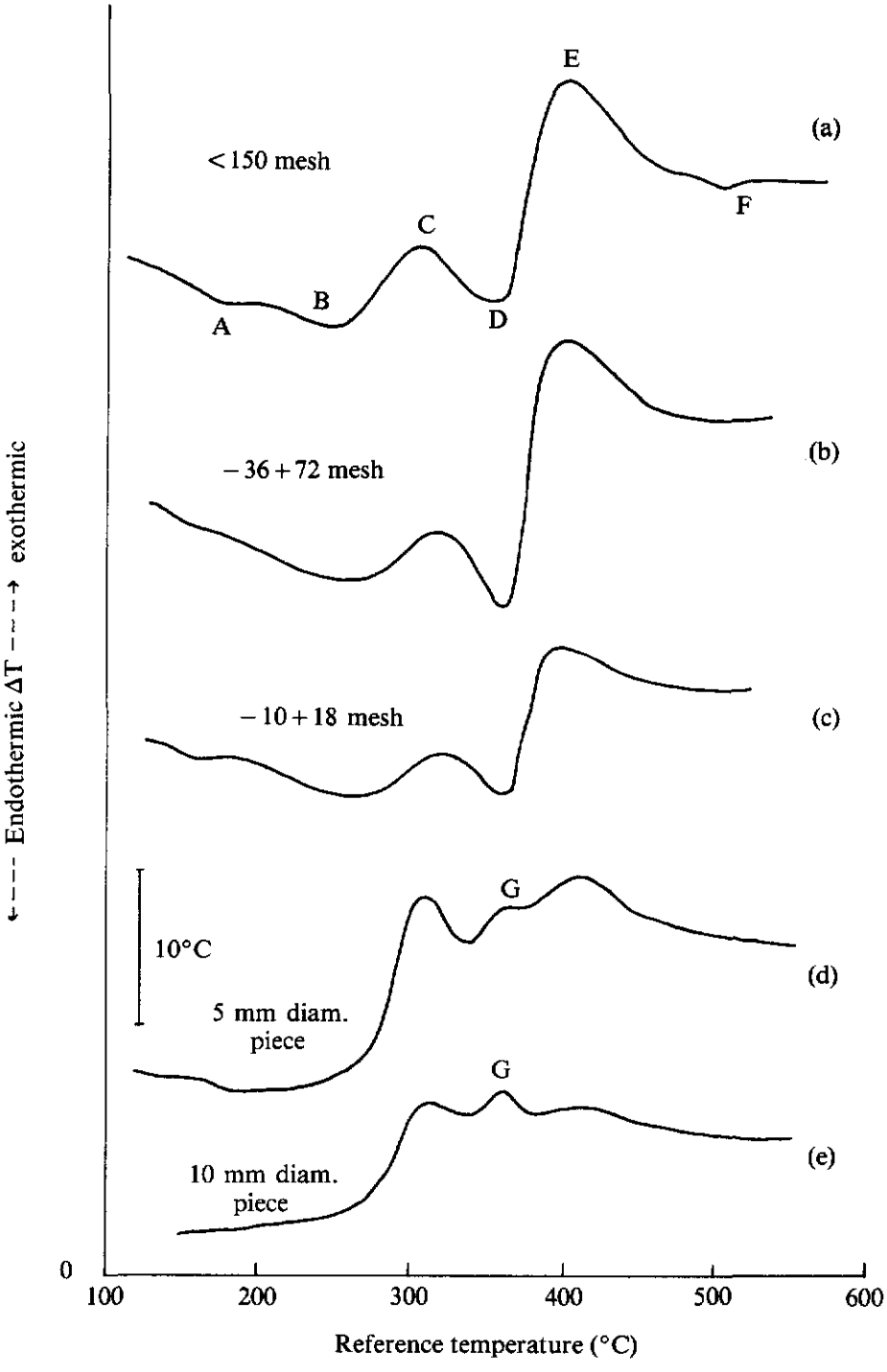


Figure 3. DTA curves of rubberwood of different particle sizes.

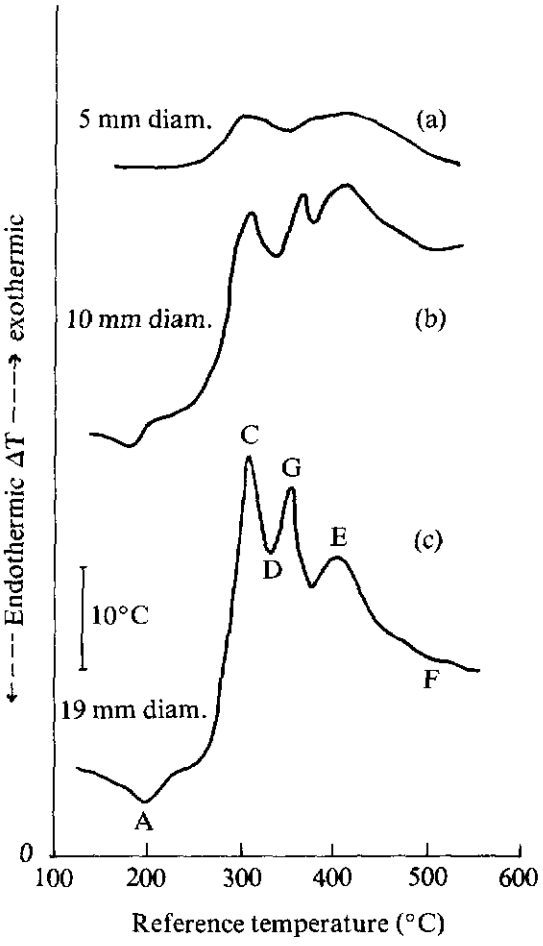


Figure 4. DTA curves of rubberwood of various diameters (single piece).

the cellulose endotherm in the DTA curves of the larger wood pieces.

The above experiments show that the thermal response of rubberwood is dependent on particle size. From the shapes of the DTA curves obtained, it appears that the pyrolysis of the larger wood pieces has an overall exothermic effect. The picture is not so clear in the case of wood of smaller particle sizes.

Effect of Gas Flow Rate

The effect of gas flow rate on the shape of the DTA curve was investigated using −36+72 mesh particles with a sample size of 3 grammes. As shown in *Figure 6,* reducing the gas flow rate from 0.8 litre per minute to 0.05 litre per minute resulted in (a) the formation of an extra peak between 350°C and 390°C, similar to the one present in the DTA curves of the larger wood pieces and (b) pyrolysis of the wood being more exothermic. In essence, lowering the gas flow rate has a similar effect as increasing the particle size of the wood. This could perhaps be explained by the fact that with a lower gas flow rate, the retention time of the pyrolysis products is longer, thus giving them more time to react, as happened within the larger testpieces. It is interesting to note that *Curve (c)* has almost the same shape as the DTA curves of the 5 mm diameter size group obtained earlier.

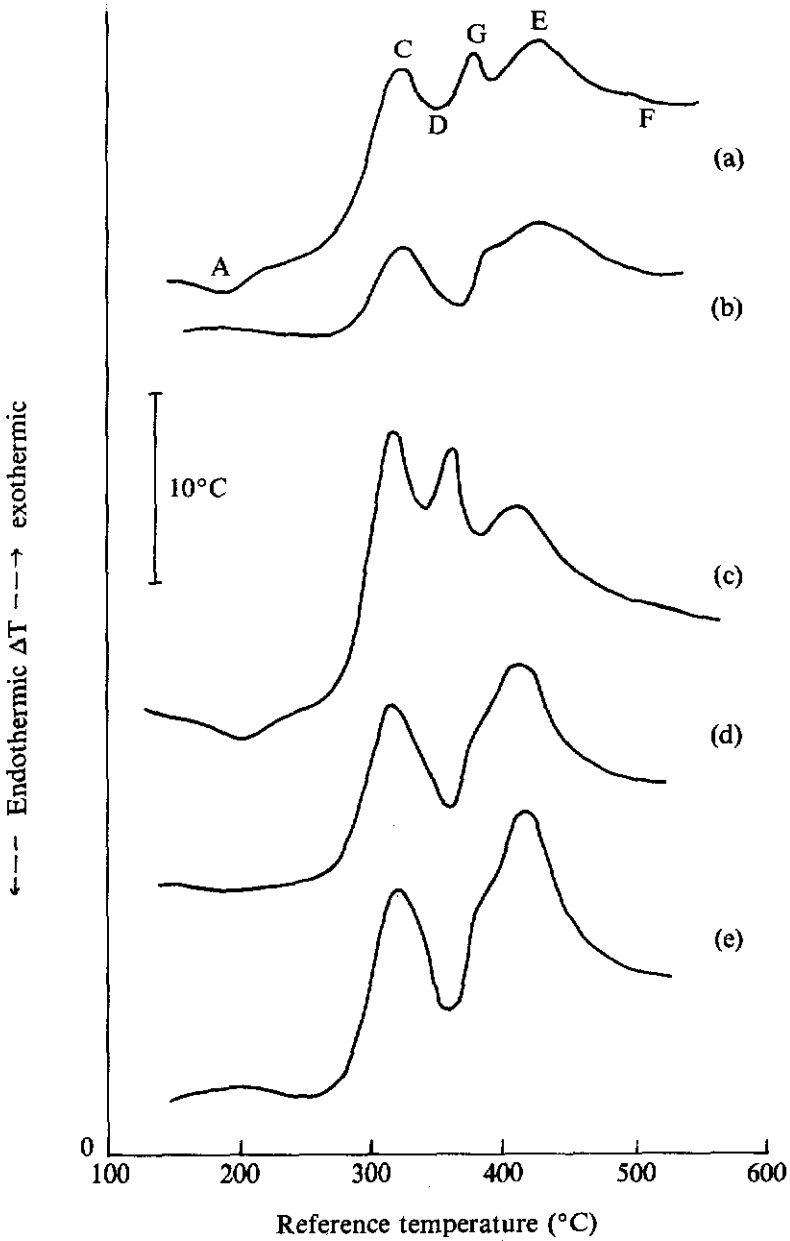
Effect of Heating Rate

To examine the effect of heating rate on the thermal response of rubberwood, runs were carried out on 19 mm diameter single testpieces using a heating rate of 3.6°C per minute. Owing to the relatively large thermal capacity of the reactor (around 20 kJ/K), the study could not be extended to heating rates above 6°C per minute. As demonstrated in *Figure 7,* reducing the heating rate from 5.5°C per minute to 3.6°C per minute resulted in the second exotherm, *i.e. Peak G,* increasing in size. This appears to be the only noticeable difference, as there was very little change in the individual peak temperatures.

Comparison of Wood from Different Trees

The thermal behaviours of rubberwood from the four trees were compared using three particle sizes, *viz.* −36+72 mesh fraction, 5 mm diameter pieces and 19 mm diameter single testpieces. A sample size of 3 g was used for the first two particle sizes while the weights of the single testpieces varied from 2.4 g to 2.7 grammes.

As shown in *Figure 8,* there is no noticeable difference between the DTA curves of rubberwood from the four trees for the −36+72 mesh fraction. In the DTA curves of the 5 mm diameter size group *(Figure 9), Exotherm G* is more conspicuous for the wood from the PR 107 tree than for those from the other three trees. This is also the case for the 19 mm diameter



(a) 10 mm diameter piece
(b) 10 mm diameter piece, boiled in water for 8 h
(c) 19 mm diameter piece
(d) 19 mm diameter piece, boiled in water for 14 h
(e) 19 mm diameter piece, soaked in water for four days

Figure 5. Comparison of DTA curves of rubberwood with and without a portion of its extractives removed.

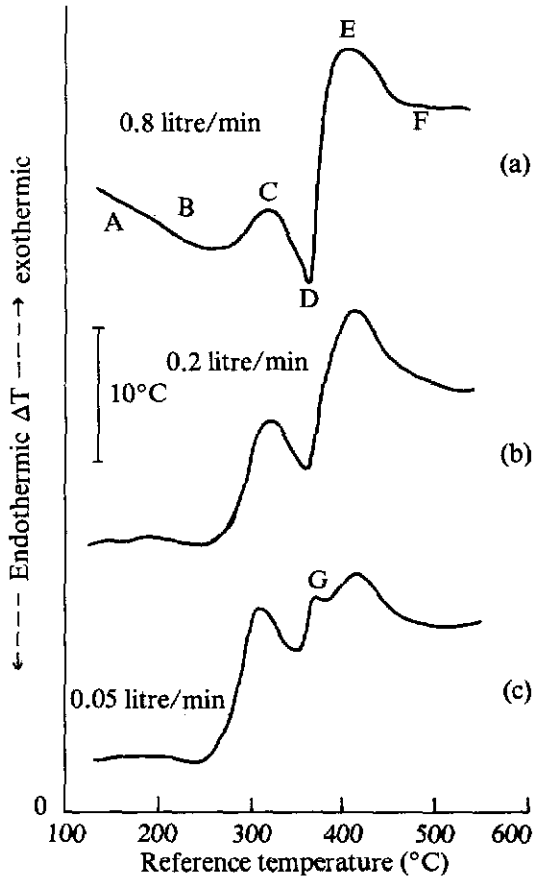


Figure 6. DTA curves of rubberwood (−36+72 mesh) from runs using different gas flow rates.

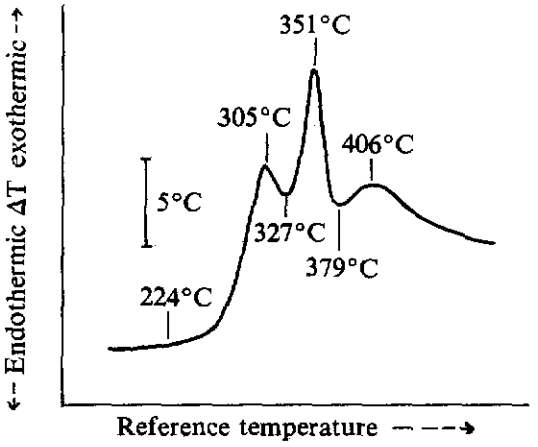


Figure 7. DTA curve from a run using a heating rate of 3.6°C per minute.

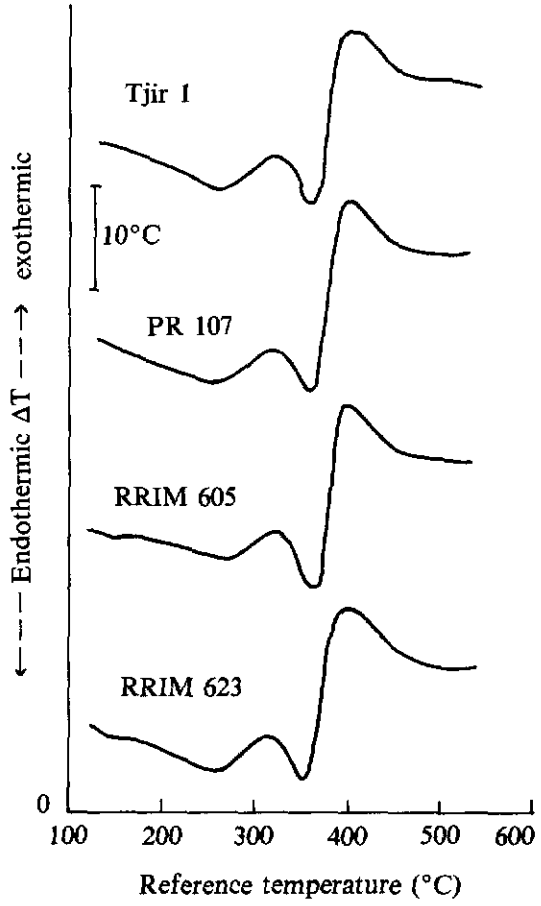


Figure 8. DTA curves of rubberwood from different trees (−36+72 mesh particles).

single testpieces *(Figure 10)*. The results obtained indicate that there is a slight difference in the thermal behaviour of wood from the four trees and that the difference is only brought about by the use of larger testpieces. As *Exotherm G* is mainly attributed to the water-soluble extractives, it would appear that the wood from the PR 107 tree contains a higher proportion of these materials than those from the other three trees. This view is supported by the fact that the PR 107 wood is 6%–11% denser than the wood from the other trees.

Comparison between Fresh and Mouldy Wood

A comparison was made between the thermal behaviours of the fresh and the mouldy wood

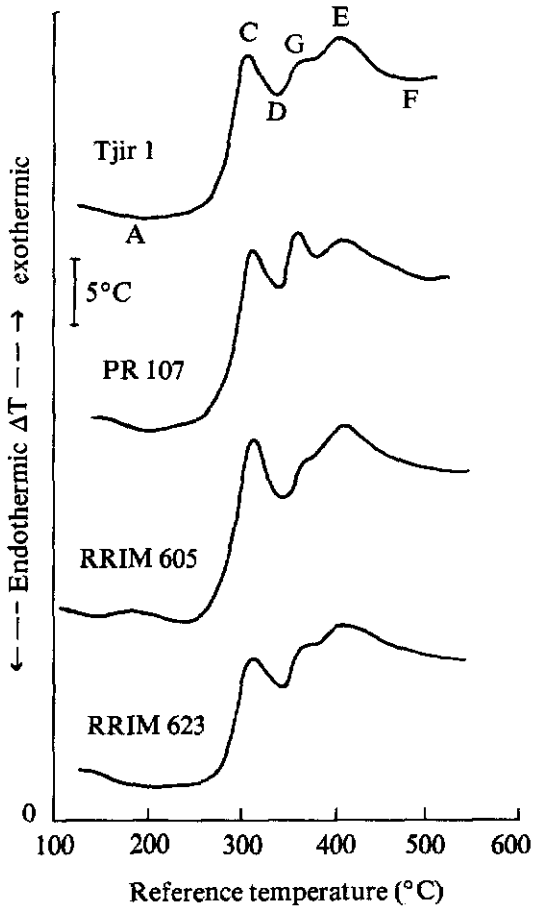


Figure 9. DTA curves of rubberwood from different trees (5 mm diameter pieces).

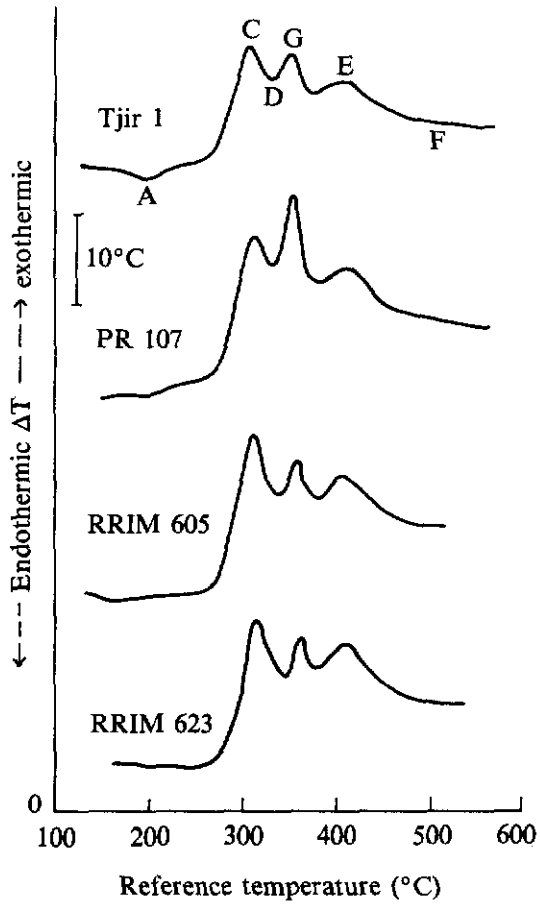


Figure 10. DTA curves of rubberwood from different trees (19 mm diameter pieces).

from each tree using 19 mm diameter single testpieces. As demonstrated in *Figures 11* and *12, Peak C* is smaller while *Peak G* is larger in the DTA curve of the mouldy wood than in that of the fresh wood from the same tree. This could be due to a change in the chemical composition or physical properties of the wood as a result of fungal infestation. The fact that the density of the mouldy wood is 5%–11% lower than that of the corresponding fresh wood indicates that some changes have taken place within the wood.

Comparison with Other Wood Species

The comparison of the thermal behaviours of rubberwood and other hardwood species was made using 19 mm diameter single testpieces. The resultant DTA curves are shown in *Figure 13*. With the exception of *Endotherms A* and *F*, all the reaction peaks which are present in the DTA curve of rubberwood are also present in those of the other wood species. There was only a slight variation in the individual peak temperatures; *Peak C*, for example, varied from 293°C to 316°C, *Peak G* from 351°C to 367°C and *Peak E* from 406°C to 429°C. The difference was greater in the relative sizes of the individual peaks. It will be noted that *Peaks E* and *G* are more prominent in the DTA curves of the other wood species than in that of rubberwood. It thus appears that pyrolysis of the other wood species above 320°C is more exothermic than that of rubberwood. If the

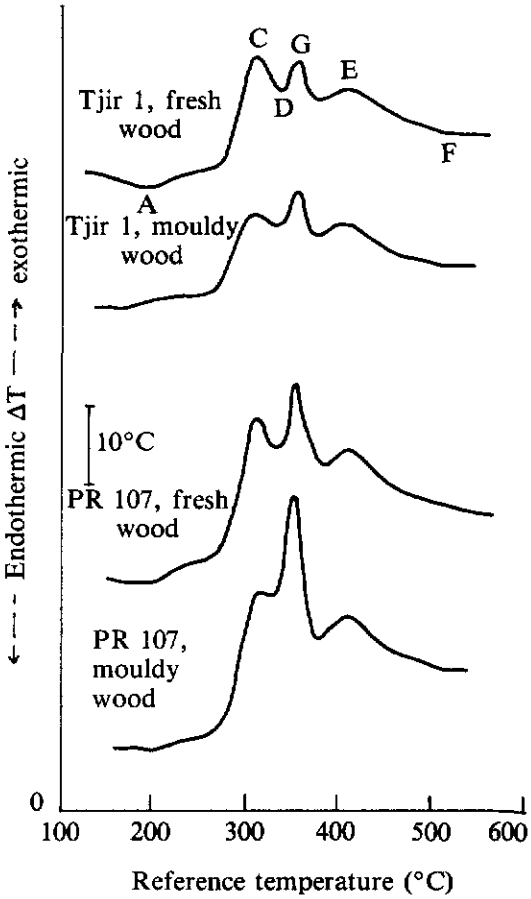


Figure 11. Comparison of DTA curves of fresh and mouldy wood (from Tjir 1 and PR 107 trees).

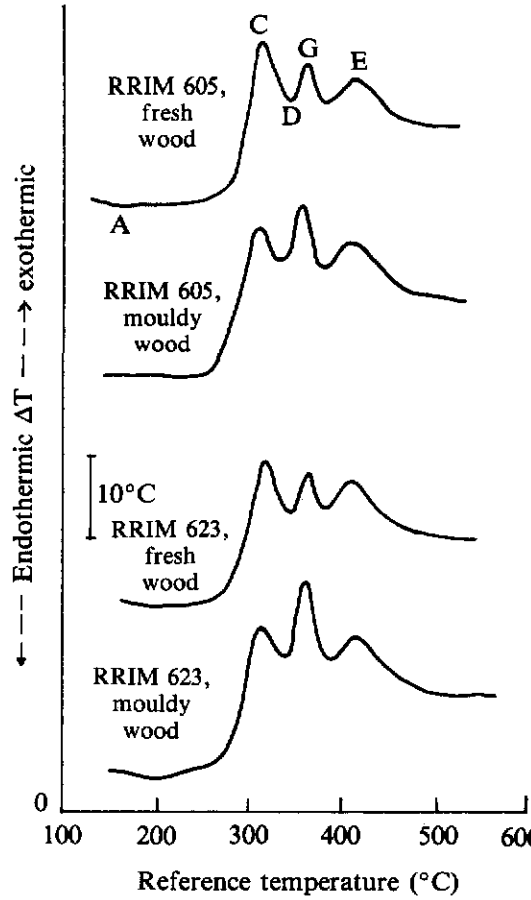


Figure 12. Comparison of DTA curves of fresh and mouldy wood (from RRIM 605 and RRIM 623 trees).

wood components which are responsible for *Peaks E* and *G* are the same for all the wood species, an inference which may be made from the results obtained is that the other wood species contain a higher percentage of lignin and the extractives than rubberwood, which is not surprising since the rubber trees from which the test samples were obtained were only between twenty to twenty-five years old compared with the forest trees which were over fifty years old at the time of felling.

Besides the tropical hardwoods, comparison of the thermal behaviour of rubberwood was also made with those of its bark, coconut wood and oil palm wood. Again 19 mm diameter single testpieces were used, except for the bark whose test sample consisted of a square piece of dimensions 16 × 16 × 6 millimetres. As shown in *Figure 14,* the DTA curves of coconut wood and oil palm wood are fairly similar in shape. Each of them appears to contain two strong and one weak exotherms, the first peaking at around 275°C (compared with around 300°C in the case of rubberwood), the second at around 340°C and the third at around 400°C. As the third exotherm is attributed to lignin, the results obtained indicate that the lignin contents of coconut wood and oil palm wood are lower than that of rubberwood. The DTA curve of the bark sample differs considerably from that of rubberwood but both of them

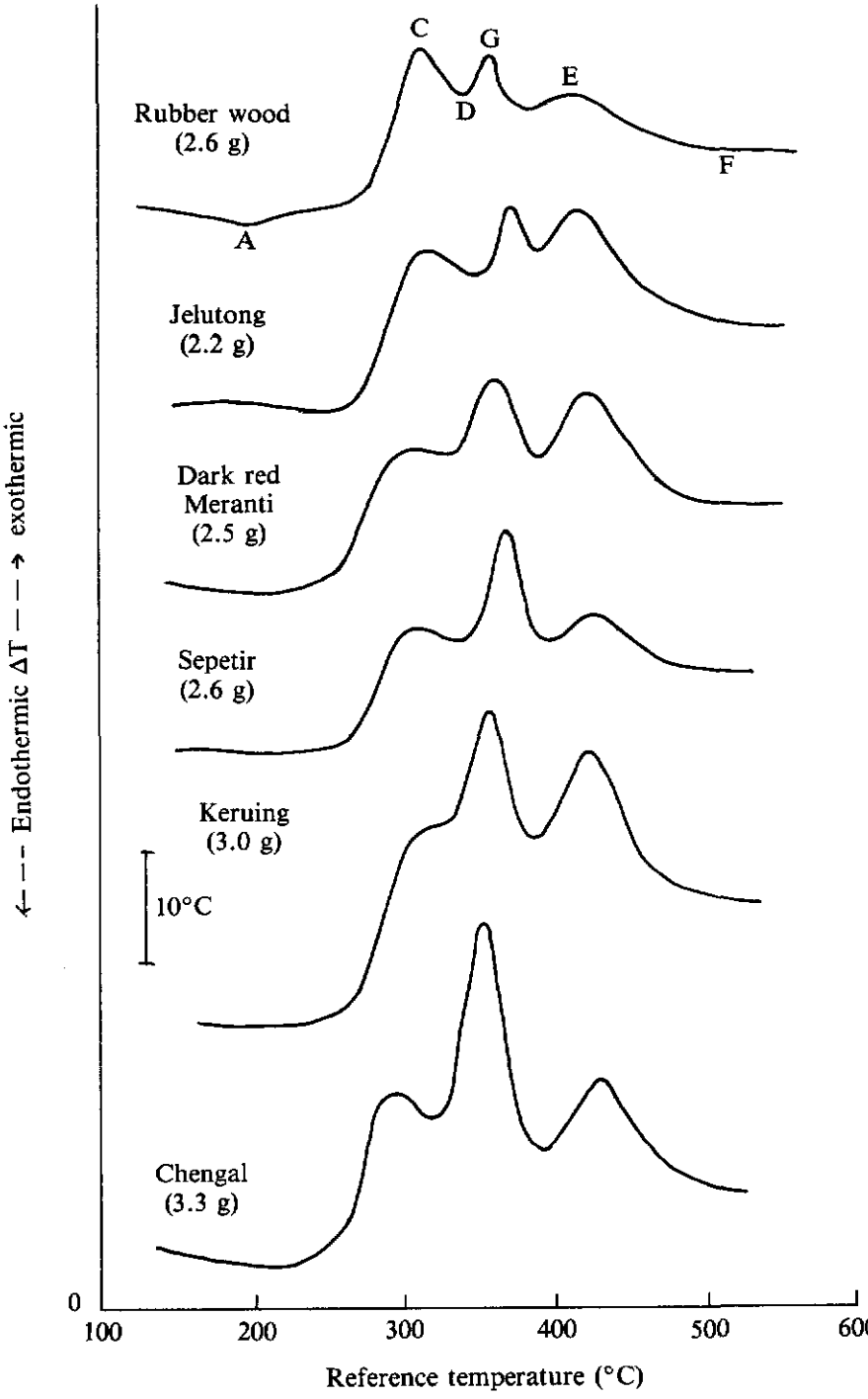


Figure 13. Comparison of DTA curves of rubberwood and some hardwood species.

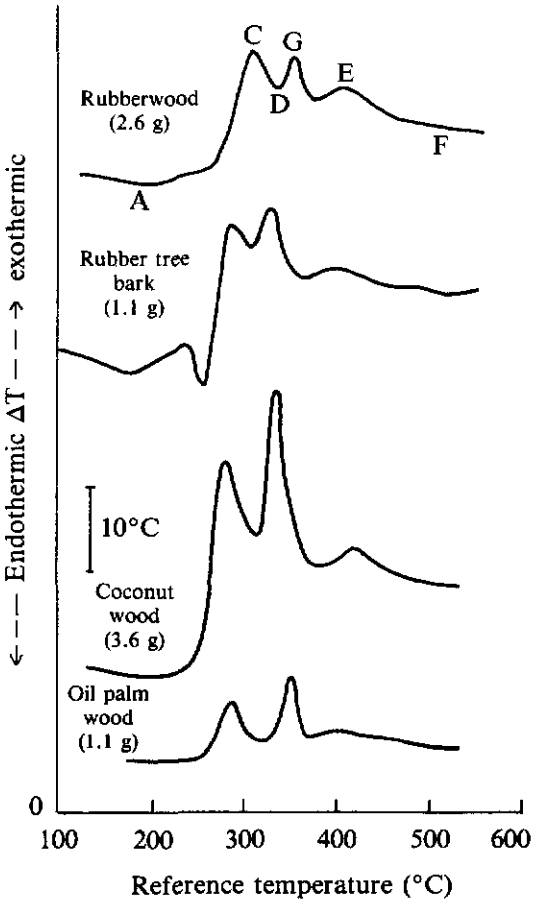


Figure 14. Comparison of DTA curves of rubberwood and some agricultural by-products.

contain certain common features, namely an endotherm below 200°C, an exotherm peaking at around 400°C and a weak exotherm at around 500°C. The lignin content of rubber tree bark also appears to be relatively low.

CONCLUSIONS

The pyrolysis of rubberwood in nitrogen begins at around 130°C and continues until around 500°C, with the major reactions occurring between 250°C and 400°C. The shape of the DTA trace is influenced by the particle size of the wood, the gas flow rate, the heating rate and fungal infestation. Thus, for comparison of the thermal behaviours of different wood species, it is important that the same experimental conditions are used. The same reaction peaks which are present in the DTA curve of rubberwood are also present in those of the tropical hardwoods, except for a difference in size which is believed to be due to a difference in the chemical compositions of the various wood species.

ACKNOWLEDGEMENT

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