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# Thermal Decomposition of Decomposed Woods under Argon Atmosphere

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**Abstract**

Weight losses in the thermal decomposition of the decomposed woods have been studied by means of dynamic experiments, carried out under argon atmosphere. The thermal pyrolysis of decomposed woods has been studied from ambient temperature up to $1000^0$C by thermogravimetric analyzer (TGA/DTA) analysis. Following an intial plateau region, the decomposed woods rapidly decompose in a narrow temperature range of 300-$400^0$C. After this sharp weight loss, there is a gradual but constant weight loss up to $900^0$C. The experiment indicates that decomposition occurs in discrete stages of hemicellulose evolution, cellulose decomposition and lignine degradation.

*Keywords: Pyrolysis, decomposed wood, argon, TGA/DTA*

## 1.0 Introduction

The thermal decomposition of decomposed wood samples *Aquilariella malaccensis* or Karas have been studied in this paper. The samples clearly produced the similar profile like other living tree but the most concern is on the sudden weight loss with respect to the time and temperature for each sample. A good practice of pretreatment for each sample has helped in producing a comparable result. Elimination of resin and wax during Soxhlet extraction has contributed in explainable profile of graphs which adhere to Browne [1] who has done comprehensive literature review on the thermal decomposition of wood. Browne has divided the pyrolysis processes into four zones or temperature ranges.

Zone A below $200^0$C, only noncombustible gases, primarily water vapor, with traces of carbon dioxide, formic and acetic acids and glyoxal exist. Dehydration of sorbed water is complete. Zone B, from $200^0$C to $280^0$C the same gases as in zone A present but with trace of water vapor left and some carbon monoxide. The reactions are endothermic and the gaseous products are largely noncombustible. Zone C, from $280^0$C to $500^0$C active pyrolysis takes place under exothermic conditions leading to secondary reactions among the largely combustible gases evolved notably carbon monoxide, methane, formaldehyde and higly flammable tars in the form of smoke particles and exothermic. The residue left in zone C becomes charcoal and catalyzes secondary reactions. Zone D above $500^0$C, the residue consists of primarily charcoal provides an extremely active site for secondary reactions.

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## 2.0 Materials & Method

### *2.1 Preparation of sample*

Karas woods were cut into smaller pieces and milled then wash with distilled water prior to oven dried. About 50g of sample underwent hot-solvent Soxhlet extraction to get rid of resins and wax.The ratio of 2:1 benzene and ethanol were used to eliminate wax and resin followed with 95% ethanol and distilled water respectively. Sample was refluxed for 8 hours for each solvent before oven dried.

### *2.2 Thermogravimetric-TGA/DTA*

The experiments were carried out in a thermogravimetric analyzer, model SETARAM TGA92. The atmosphere used for pyrolysis was pure argon (99.99%) at a low pressure of 150kPa with a flow rate of around 35ml $min^{-1}$, according to the specifications of the equipment. In order to obtain accurate results, the TG analyzer was calibrated using $CuSO_4$. Thermogravimetric Analysis (TGA) measures the changes in weight of a sample with increasing temperature. Moisture content and presence of volatile species can be determined with this technique. The dynamic experiment has been carried out with different heating rates namely 5, 10 and 15 $^0C$ $min^{-1}$ which were kept constant throughout the experiments. Whereas, Differential Thermal Analysis (DTA) measures the difference in temperature between a sample and a thermally inert reference as the temperature is raised. The plot of this differential provides information on exothermic and endothermic reactions taking place in the sample.

## 3.0 Results and discussions

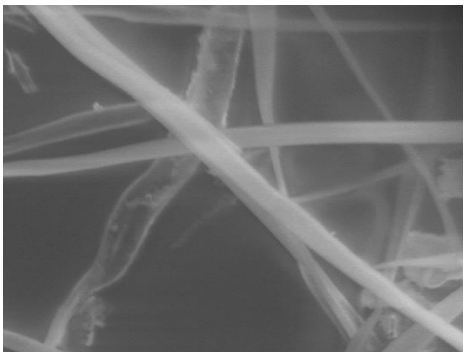

Figure1 Scanning Electron Microscope SEM of cellulose microfibrils from *Aquilariella malaccensis* or Karas wood, showing the crystalline nature of cellulose microfibrils

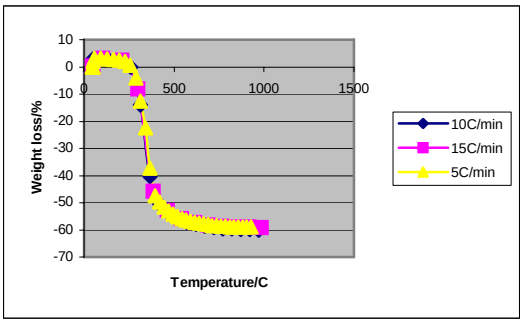


Figure 2 TG curves of decomposed karas wood in argon atmosphere

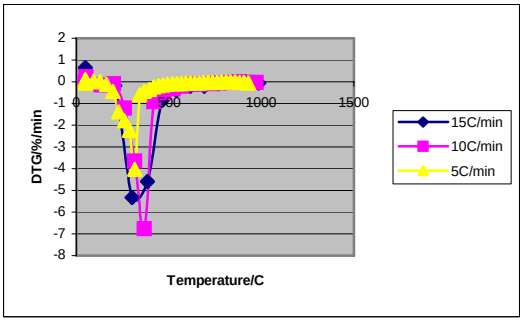


Figure 3 DTG curves of decomposed karas wood in argon atmosphere

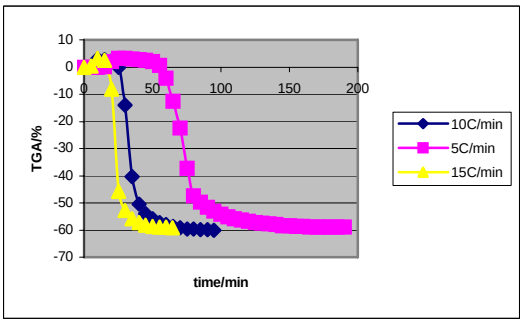


Figure 4 weight loss/%= f(t) of pyrolysis of karas wood

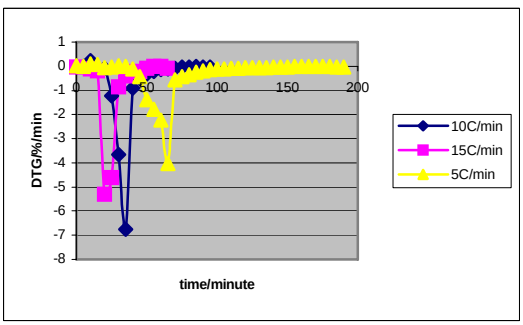


Figure 5 First derivative DTG curve= f(t) of pyrolysis of karas wood

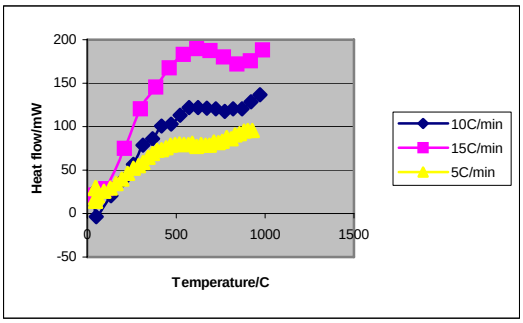


Figure 6 Heat flow as a function of temperature for different heating rates

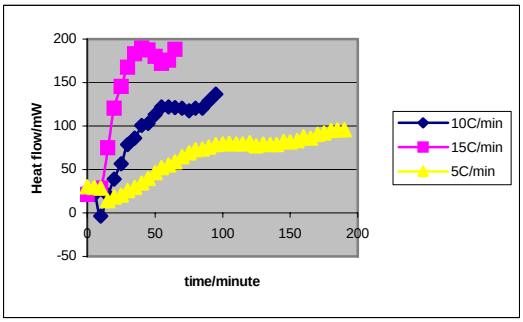


Figure 7 Heat flow as a function of time for different heating rates

## 4.0 Results and Discussions

The dynamic gravimetric (TG and DTG) curves of karas woods are shown in Figure 2 and 3 with respect to temperature $^0$C and time in minute. They are similar to the typical dynamic gravimetric curves of other cellulosic materials. The rate of weight loss of this Karas wood is very high at a narrow range of temperatures. The homogeneity of samples can be seen from the Figure 2 as shown above. Following a plateau region to 230$^0$C, the Karas wood rapidly decomposes between 257$^0$C and 417$^0$C, corresponding to a weight loss of around 60% at 442$^0$C, 571$^0$C and 691$^0$C for heating rate of 5$^0$Cmin$^{-1}$, 10$^0$Cmin$^{-1}$ and 15$^0$Cmin$^{-1}$ respectively. The first derivative plot indicates that the maximum rate of weight loss is at 313$^0$C, 367$^0$C and 300$^0$C respectively. Normally, lignin is decomposed at this range of temperature. The 'smooth' dynamic gravimetric curves indicate that the transition from char dominating to tar dominating reaction is gradual. In the pyrolysis of cellulosic materials, char formed at lower temperatures have a higher aliphatic content, and are easily decomposed [3]. In chemistry, aliphatic compounds are non-aromatic organic compounds, in which carbon atoms are joined together in straight or branched chains rather than in rings. The simplest aliphatic compound is methane ($CH_4$) [4].This is a possible explanation of the slow but constant weight loss observed in Fig 2 and 3 after the main decomposition step. These figure also show no distinct effect on the subject yield when the heating rate varies for gradually increasing temperature. For DTG graph, it indicates that as the temperature goes up, decomposition of Karas wood is more perfect and steeper for 10$^0$C/min heating rate. From figure 3 for heating rate 10$^0$C/min, the decomposition of wood starts at temperature 260.2$^0$C after 25 minutes and the absorbed energy is 56.58mW. For figure 4 and 5, it can be clearly seen that decomposition of

decomposed Karas wood is faster at the higher heating rate that, is $15^0$C/min. Figure 6 and 7 indicate the energy used for the decomposition as a function of temperature and time

## 5.0 Conclusions

1. The heating rate doesn't affect the profile of weight loss = f(T) to decomposed Karas wood.
2. Heating rate at $10^0$C/min gives perfect decomposition profile and clearly described peak
3. The faster the heating rate the steeper is the slope of the weight loss curve and the sharper is the first derivative (DTG), thus the decomposition rate is faster

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