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# Influence of Internal Pressure Developed During Carbonization by Sealed-Tube-Technique of Pyrolysis

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T. Hossain, N. Zaman,  
S. T. Jahan, Jiban Podder,  
M.A. Rashid.

Dept. of Physics, BUET,  
Dhaka-1000, Bangladesh.

**Abstract:** During carbonization of a pure organic compound inside a sealed tube, a heavy pressure is developed inside the tube due to emission of hydrogen and various hydrocarbon gases and in the present work the influence of this high pressure on the overall carbonization process was studied. It was found that the increasing internal pressure caused by the pyrolysis gases increases the carbon yield, the vapor pressure gradually decreasing as more and more carbons are produced. The pressure attains a steady value when the carbon yield becomes maximum.

**Keywords:** Carbonization , Pyrolysis, Mesophase, Graphitization.

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## INTRODUCTION

By pyrolysis, carbon containing organic compounds are gradually converted to solid carbon as the main product and to different volatile compounds as by products. Some organic compounds are found to be evaporated leaving no carbonaceous residue in open crucible. Pyrolysis of the organic compounds in an inert atmosphere does not even produce significant amount of carbon. In the sealed-tube-technique of pyrolysis, heating of the sample is first carried out under normal pressure, then under increasing pressure caused by the pyrolysis gases. It is presumed that this increasing pressure may accelerate the coke formation. Keeping the above points in mind, the sealed-tube-technique of pyrolysis has been adopted in the present study to investigate the influence of internal pressure on carbonization.

## EXPERIMENT

A small amount ( $\sim 1.32 \times 10^{-3}$  kg) of the sample under investigation was held in a heavy-walled Pyrex tube about  $18.6 \times 10^{-2}$  m in length and  $12.8 \times 10^{-3}$  m internal diameter. The tube containing the sample was sealed at both the ends and placed inside a steel bomb fitted with screw-caps at both the ends. The sample was then

heat-treated inside a solenoidal furnace at a fixed power input to the bomb up to the temperature of 813<sup>o</sup>K (540<sup>o</sup> C) at which the sample was kept for a long duration. A heating rate of 10<sup>o</sup>C per minute was adopted by the temperature controller. During heating, the pressure inside the sealed tube increased primarily because of the presence of volatile materials, such as hydrogen and other hydrocarbon gases which are evolved. After allowing the tube to cool to room temperature the sealed tube with the carbonized sample was opened inside a specially designed safety box to avoid blast due to heavy pressure inside. The carbon residue was then separated from inside the tube, dried and measured to see the amount of carbon produced. Using the ideal gas equation, the inside pressure was then calculated from the experimentally obtained value of carbon product and assuming that the main components of the pyrolysis gases that are emitted are methane and hydrogen, the presence of which have been chemically tested earlier [1]. A model calculation of the inside pressure developed due to emission of hydrogen and methane is shown in Table 1.

**Table 1** Pressure developed due to emission of hydrogen and methane.

Heat treatment during (hrs)	Initial			Final		
	Temp. T <sub>1</sub> (°K)	Volume V <sub>1</sub> (lit)	Pressure P <sub>1</sub> (kPa)	Temp. T <sub>2</sub> (°K)	Volume V <sub>2</sub> (lit)	Pressure P <sub>2</sub> (kPa)
10	813	28.39	100	305	0.024	4.44x10 <sup>4</sup>

## RESULTS AND DISCUSSION

The amount of carbon produced by sealed-tube-pyrolysis of the samples, namely benzene, naphthalene, anthracene, phenanthrene, chrysene and hexane at 813<sup>o</sup>K (540<sup>o</sup>C) for different heat-treatment duration are graphically presented in Fig. 1. The internal pressure as a function of heat-treatment duration for the different samples is also shown in fig. 2. The observed fall in pressure with the increase in heat-treatment duration might be due to the reason that organic samples undergo changes in structure with the evolution of hydrogen and other hydrocarbon gases until a non-volatile carbon remains as residue in which case the pressure attains a steady value. The observed fluctuations in the initial portion of the DTA traces for the samples indicates such dehydrogenation and emission of volatile products [2,3,4].

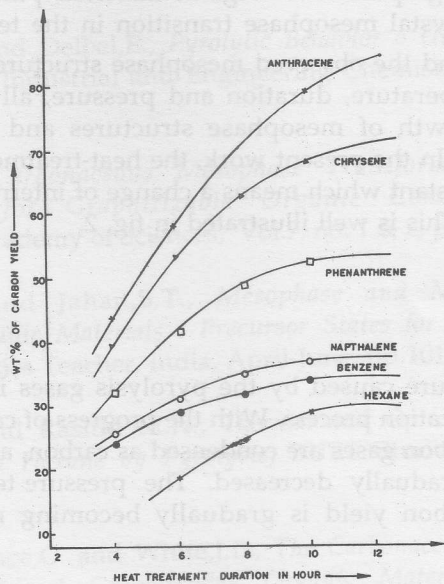


Fig. 1 Percentage of carbon yield at 813°K for different heat treatment duration.

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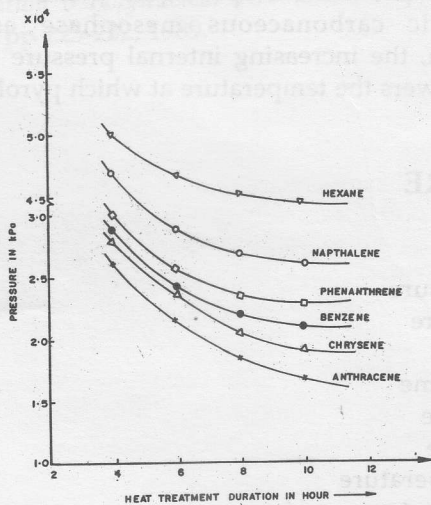


Fig. 2 Variation of internal pressure with heat treatment duration.

It is known that all graphitizable organic materials pass through an optically anisotropic liquid crystal mesophase transition in the temperature range 573-873°K(300-600°C) and the observed mesophase structures arise from a unique combination of temperature, duration and pressure, all the three parameters promoting the growth of mesophase structures and subsequent chemical condensation [5,6,7]. In the present work, the heat-treatment temperature of the sample was kept constant which means a change of internal pressure with heat-treatment duration. This is well illustrated in fig. 2.

## CONCLUSION

The increasing pressure caused by the pyrolysis gases inside the sealed tube enhances the carbonization process. With the progress of carbonization more and more of the hydrocarbon gases are condensed as carbon, as a result of which the inside pressure is gradually decreased. The pressure tends to attain a steady value when the carbon yield is gradually becoming maximum with heat-treatment time.

It is also well-known that pressure usually increases the viscosity of a liquid. So increase in viscosity with pressure reduce the tendency of bubble formation and hence reduce disorder produced by convection currents within the system. Thus the increasing internal pressure caused by the pyrolysis gases enhances the molecular condensation reactions as well as the viscosity of the system. The enhanced viscosity affects both diffusion and coalescence rate. With the increase in temperature and time, the isotropic liquid phase is gradually changed to optically anisotropic carbonaceous mesophase as a prerequisite to graphitization. Again, the increasing internal pressure not only increases the coke yield but also lowers the temperature at which pyrolysis is completed.

## NOMENCLATURE

P	:	pressure
P <sub>1</sub>	:	initial pressure
P <sub>2</sub>	:	final pressure
V	:	volume
V <sub>1</sub>	:	initial volume
V <sub>2</sub>	:	final volume
T	:	temperature
T <sub>1</sub>	:	initial temperature
T <sub>2</sub>	:	final temperature



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