INFLUENCE OF THERMAL TREATMENT ON THE SEPARATION PROPERTIES OF POLYAMIDE MEMBRANE

by

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Progress report submitted in partial fulfillment of
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CERTIFICATION OF APPROVAL

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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FATHULLAH BIN SHAMSUDDIN

ABSTRACT

Membranes have a huge application in the industry nowadays. Their cost effective and efficiency further make the demand for membranes increases. In ensuring the effectiveness of membrane applications, continuous monitoring enhancement had to be done. Integrally-skinned asymmetric polyamide-6 membranes were prepared by casting various solution of polymer concentration from 10wt% to 20wt%, in 85wt.% formic acid onto a glass plate and precipitation in inert atmosphere before it would be then immersed in cold distilled water until it peeled off.. The obtained membranes were dried for 1 h at different temperatures ranging from 40 to 130°C. The pervaporation separation properties of such treated membranes were studied with binary water-ethanol mixtures consisting of 40wt% ethanol. Experimental tests procedure will be using the GC equipment, which suitable for typical laboratory-scale testing. The selectivity was improved by thermal treatment of the membranes at temperatures above the glass transition temperature Tg of polyamide-6 (≈60°C). However, the permeate flux decreased with increasing drying temperature due to the removal of entrapped water from amorphous domains, the formation of new crystalline domains and the decrease of the pore size and of the specific surface area during the thermal treatment. The study of the physical properties of the polyamide-6 membrane will be characterized by using the assistance of existing physical and mechanical testing equipments available such as SEM and DMA.

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INTRODUCTION

1.1 BACKGROUND OF STUDY.

Membrane separation processes have been studied extensively as promising energy-saving separation techniques. Among them, pervaporation is used for liquid mixture separation such as azeotropic mixtures or organic mixtures with close boiling points. In today modern era, membranes had played a major and important role in the industrial sector. It is used widely in separation and purification process fields extending from liquid-liquid to liquid-gas separation processes.

The effect of thermal treatment on pervaporation separation properties of polyamide membranes could be explained by the rearrangement of polymer chains in the amorphous domains and therefore with the growth and formation of crystalline domains. It was predicted that the fast removal of entrapped water in the polymer material during the drying process hinders the collapse of pores and voids.

1.1.1 Membrane Overview.

Membranes are thin films of porous material which can be used for a number of chemical separations. Although many membranes are made from polymer films, membranes can be formed from ceramics, carbon fiber, and porous metal substrates. The pores can range from atomic dimensions (<10 angstroms) to 100+ microns.

The small pores of the membranes can serve as a physical barrier, preventing passage of certain materials such as salt, bacteria and viruses while allowing the free passage of water and air. The desalination of water using reverse osmosis is a well known use of membranes as a filter. The membrane structure and chemistry can also serve to carry out other separations. Membranes provide a high surface area material where chemical reactions or diffusion can take place, where bundles of hollow fiber membranes (membranes in a thin tubular form) are used in dialysis to purify the blood by removing certain toxins. Membranes can also be used to carry out solvent extraction and catalysis while also serving to separate the reactants. Hydrophobic membranes can be used to prevent passage of liquid water but allow vapor to pass. This property has been exploited in membrane distillation where brackish water is heated using solar power and the pure water vapor passes through the membrane and condensed to produce very high quality water. It uses less energy than boiling and utilizes bountiful but low value energy in remote areas.

A number of different techniques are available in preparing synthetic membranes. Some of these techniques can be used to prepare organic as well as inorganic membranes and are shown in the table below. For this project, the membrane preparation process that is chosen to be used to prepare the membrane samples was the phase inversion process. In fact, about 80% of the membranes used and practiced nowadays in various industries are by using this process.

Table 1 : Membrane preparation techniques.

Membrane Preparation Process	**Types of Membranes Prepared**
Phase Inversion	Gas, reverse osmosis, dialysis membranes, ultrafiltration, and microfiltration.
Phase Separation/Leaching	Na2O-B2O3-rich phase SiO2-rich phase (glass/ceramics).
Sol/Gel Techniques	Metal alkoxied hydrolyzed, polymerized (alumina, titania, zirconia).
Sintering	0.1 - 20 microns pore size (fibrous mats, PTFE, PE, PP, etc.).
Controlled Stretching	0.1 – 5 microns pore (Goretex® and Celgard®).
Extrusion/Activation	Silicone rubber, NAFION®.

Controlled Pyrolysis	Base organic membranes pyrolyzed to form silica or carbon molecular sieve membranes.
Track-etching	Radioactive source exposure, then etch with acids, (0.1 – 1 nm, NUCLEOPORE®).
Thin Film Deposition	Sputtering plating, vapour deposition (transition metals alloys, i.e. Al/Ag, Cu/Zr, Ni/Pd).
Anodic Oxidation/Etching	Oxidation from one face; etching by strong acids of metal.
Coating	Composite membranes.

1.1.2 Pervaporation Overview.

Pervaporation, is an energy efficient combination of membrane permeation and evaporation. It is considered an attractive alternative to other separation methods for a variety of processes. Requiring low temperatures and pressures during pervaporation, it often has cost and performance advantages for the separation of constant-boiling azeotropes. Pervaporation is also used for the dehydration of organic solvents and the removal of organics from aqueous streams. Additionally, pervaporation has emerged as a good choice for separation heat sensitive products.

The process involves the separation of two or more components across a membrane by differing rates of diffusion through a thin polymer and an evaporative phase change comparable to a simple flash step. Concentrate and vapor pressure gradient is used to allow one component to preferentially permeate across. A vacuum applied to the permeate side is coupled with the immediate condensation of the permeated vapors. Pervaporation is typically suited to separating a minor component of a liquid mixture, thus high selectivity through the membrane is essential. For this research project, batch pervaporation process were done due to its simplicity and energy saving. Batch processing was chosen rather than continuous pervaporation process due to its ability to yield quantitative that is required for the project analysis.

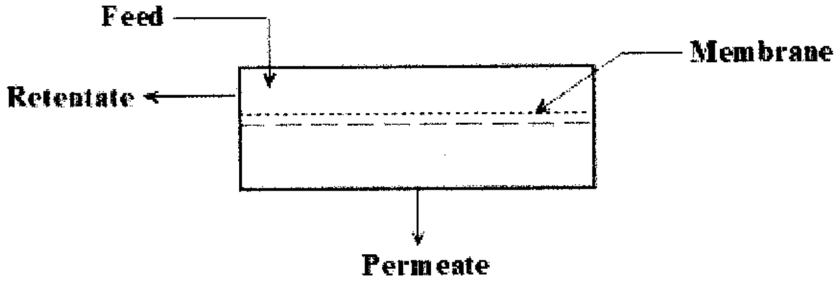

Figure 1 : Overview of the Pervaporation Process.

Pervaporation can used for breaking azeotropes, dehydration of solvents and other volatile organics, organic/organic separations such as ethanol or methanol removal, and wastewater purification. Characteristics of the process include:

i) Low energy consumption.
ii) No entrainer required, means no contamination.
iii) Permeate must be volatile at operating conditions.
iv) Functions independent of vapor/liquid equilibrium.

1.1.3 Ethanol.

Ethanol (CH_3CH_2OH) is used as an automotive fuel by itself and can be mixed with gasoline to form what has been called "gasohol" FUEL ETHANOL- the most common blends contain 10% ethanol and 85% ethanol mixed with gasoline. Over 1 billion gallons of ethanol are blended with gasoline, which is used by about 9% of cars every year in the United States. In fact, 50% of cars in Brazil use pure ethanol as fuel with modified engines. Because the ethanol molecule contains oxygen, it allows the engine to more completely combust the fuel, resulting in fewer emissions. Since ethanol is produced from plants that harness the power of the sun, ethanol is also considered a renewable fuel. Therefore, ethanol has many advantages as an automotive fuel.

membranes. Frequently, the applied modification techniques include thermal treatment of the membrane.

The expectations of ethanol's degradability rely on experiments that use microcosms of groundwater and soil mixtures to demonstrate that ethanol is rapidly degraded both aerobically (100 mg/l in 7 days, Corseuil *et al.*, 1998) and anaerobically (100 mg/l in 3 to 25 days, depending on conditions Corseuil *et al.*, 1998; 96 mg/l within 30 days, Suflita and Mormile, 1993; 100 mg/l within 14 days, Yeh and Novak, 1994). In these experiments, ethanol generally delays degradation of benzene, toluene, and xylenes, up to double, and some investigators (Corseuil *et al.*, 1998) caution against generalizations about ethanol's effect.

1.3 OBJECTIVES AND SCOPE OF STUDY.

The objectives and scope of the project are :-

i) To study the effect of thermal treatment on the properties of the membrane.
ii) To investigate the effect of polyamide concentration on the properties of membrane.
iii) To study the separation of ethanol from binary water-ethanol mixture.
iv) To characterize the polyamide membrane prepared.

CHAPTER 2

LITERATURE REVIEW AND/OR THEORY

Literature reviews were focused on the methods or means in obtaining the best result for the experiment that will be done and also including previous journals related to the subject. Since the project is experimental based research project, hence the accuracy and precision of results are critical and important [1, 2, 3, 4]. The standards APHA method was review, which is typically used in Quality Control Departments in continuous monitoring of products quality. It is suitable for laboratory scale water tests but it is quite complex and time consuming.

A study of water-ethanol mixtures pervaporation by dense polyamide-6 membranes was done by Kujawski *et al.* [14] and showed that the selectivity parameters in pervaporation were improved for membranes obtained at higher polymer content and dried at higher temperatures, but with a loss of lesser permeation. The cast membranes were dried at different temperatures from 25 to 80°C. The performance of the membrane would be monitor by substituting trifluoroethanol by formic acid as the solvent for casting solutions of polymer.

Besides that, Meier-Haack *et al.* [15] had investigated the composite pervaporation membranes composed of an asymmetric polyamide-6 membrane and an ultra thin self-assembled polyelectrolyte separating layer. A high end group concentration was found to be advantageous for sufficient adhesion of the multilayer systems on the supports. Performance of these membranes depends strongly on the layer number and on the type of polyelectrolytes.

Variation of polyelectrolyte concentration of the dipping solution and adsorption time had only a minor effect on the membrane performance. On the other hand, high molecular weight polyelectrolytes had a high impact on membrane performance. This condition was studied and proved by Lenk *et al.* [16]. More importantly, besides of that it was observed that membranes prepared at high temperatures yielded a lower swellability and therefore a higher selectivity and a lower permeate flux.

As been studied by Cheng *et al.* [17], aliphatic polyamides (Nylon-66 and a Nylon-6, -66, -610 terpolymer) were isothermally precipitated from formic acid solution. Under certain conditions, crystallization of Nylon-66 will be resulting in a membrane with a uniform skinless microporous structure that was rapidly wetted by water. In contrast, liquid-liquid phase separation produces a polyamide film with largely unconnected cellular voids that is as a result not wetted by water.

In his other study, a novel microporous membrane was synthesized from a polymeric nano-composite material, mica-intercalated-Nylon 6, by isothermal immersion-precipitation in a pure water bath [18]. By contrast, pure Nylon 6 precipitated under the same condition yielded a skinned asymmetric membrane. In addition, porosity of the membrane was found to increase with increasing water content. This was evidenced by the tensile strength and the water permeability measurements.

Pervaporation experiments to measure steady state permeation and separation characteristics by using the polymeric membranes were done by Doguparthy *et al.* [19]. Composite membrane showed good selectivity to water in both methanol–water and ethanol–water mixtures. The most important observation is that there was good separation of ethanol–water is possible with this membrane.

Besides of that, the important information about the principles and function of membranes were also done [5]. This was done to determine the suitable form and properties of membrane for separation processes besides to enhance understanding of the transport phenomena. Membrane is practiced in various fields ranging from, petrochemical processes, water treatment, pharmaceutical and pollution control [6, 7].

According to the solution-diffusion model, higher fluxes can be obtained with an increased thermal motion of the polymer chains and the diffusing species. Properties of the polymers that affect diffusion include the "backbone" material, degree of cross-linking, and porosity. Molecular-level interactions between membranes and the diffusing species are expressed via a permeability constant used in the Arrhenius relationship [12].

The most common method for thermal characterization of polymeric matrix composites is the dynamic mechanical analysis (DMA) method. All DMA techniques produce curves of dynamic storage and loss modulus and loss tangent (tan δ) as a function of temperature. Tan δ is proportional to the ratio of the loss modulus (E" or G") to the storage modulus (E' or G') [10]. At the glass transition, these parameters go through a peak value. Similarly to other methods of measuring δ, the interpretation of the curves is aided by software and the value of δ may be determined as the temperature at the onset or the midpoint of the transition based on the storage modulus curve at the maximum in tan δ.

The Scanning Electron Microscope, or SEM, is an incredible tool for seeing the unseen worlds of microspace. Conventional light microscopes use a series of glass lenses to bend light waves and create a magnified image [13]. The SEM creates the magnified images by using electrons instead of light waves. It shows very detailed 3-dimensional images at much higher magnifications than is possible with a light microscope. The images created without light waves are rendered black and white. Samples have to be prepared carefully to withstand the vacuum inside the microscope.

CHAPTER 3

METHODOLOGY/PROJECT WORK

3.1 PROCEDURE IDENTIFICATION.

The project procedure could be divided into four major stages, which are membrane preparation, pervaporation of ethanol experiment, analysis of the membrane pervaporation separation capability, and characterization of the membrane. Before further steps could be taken, materials required throughout this study were identified and prepared beforehand. The materials and procedures are further described below.

3.1.1 Materials.

Polyamide-6 (PA-6; M_n 22.600 g/mol; Technyl® Polyamides) was purchased from Rhodia Engineering Plastics Co. Ltd. (Taiwan). Meanwhile, formic acid and pure ethanol both are analytical grade and were made by Systerm and Merck (Germany) respectively. Each was used without any purification.

3.1.2 Membrane Preparation.

The membranes are to be prepared from casting solutions containing various content of PA-6 wt.% in formic acid/water (85/15wt./wt.). PA-6 content would be varied at 10wt.%, 15wt.%, and 20wt.%. It would then be prepared by phase inversion process technique whereby the polymer solution would be cast on a glass plate using a casting knife (Sheen Instruments, England). The drawdown thickness was set to be ≈200μm. The PA-6 membranes were then would be dried for an hour at different temperature ranging from 40°C to 130°C in the drying oven (Thermal Engineering).

The overall procedure for membrane samples preparation could be viewed in the flowchart as illustrated below.

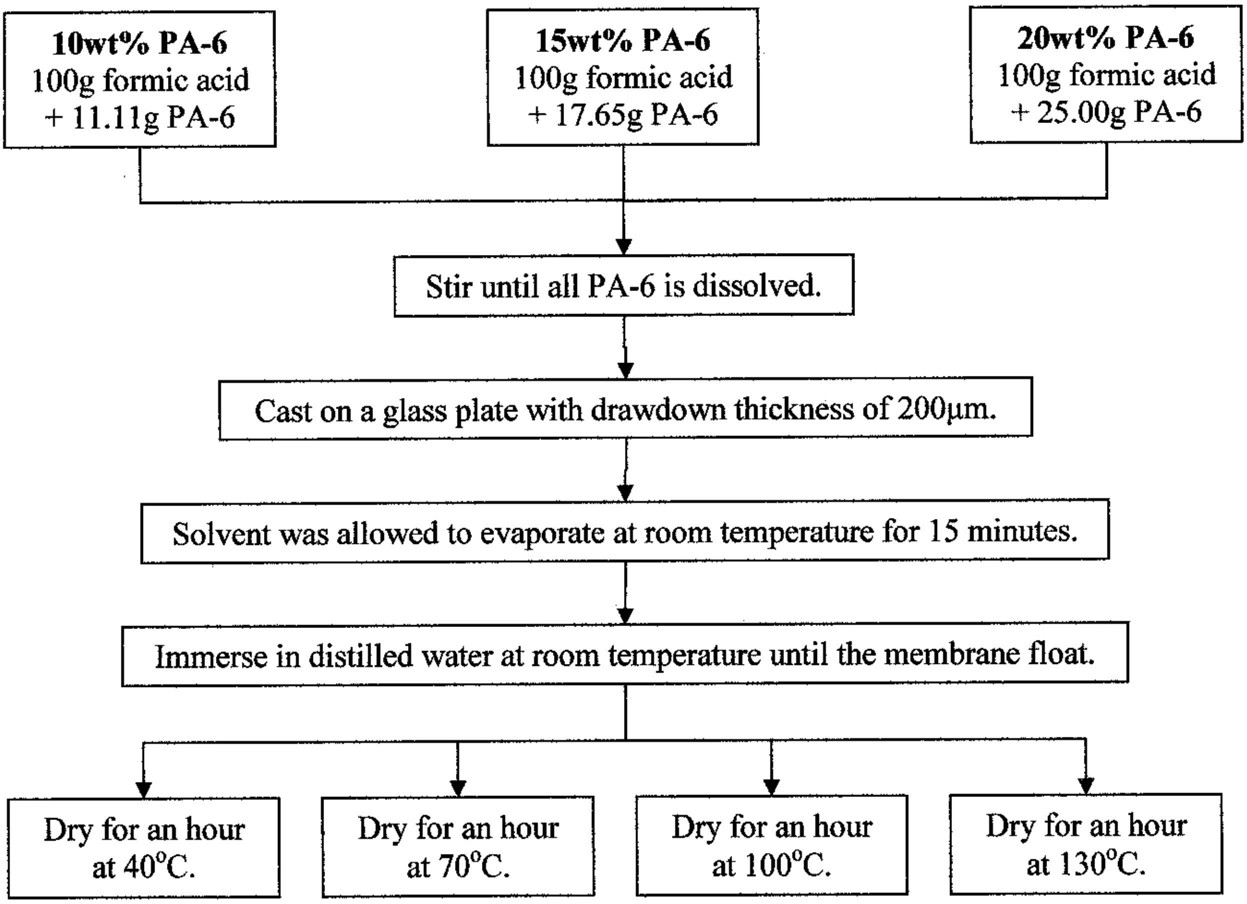

Figure 3 : Flow of membranes preparation process.

3.1.3 Pervaporation Separation of Ethanol.

Equal volume of 100mL initially binary water-ethanol mixture would be placed in the test tubes. The binary water-ethanol mixture prepared beforehand consist of 60wt.% water and 40wt.% ethanol. The mouth of the test tubes would be covered with a PA-6 membrane sheet, which later would be placed upside-down on a shaker with the aid of retort stand for four hours at a speed of 130. This experiment was done at room temperature. Therefore, the driving force involved would be the gravitational force.

The test tubes experiment position for the pervaporation separation of ethanol could be viewed in the illustration below.

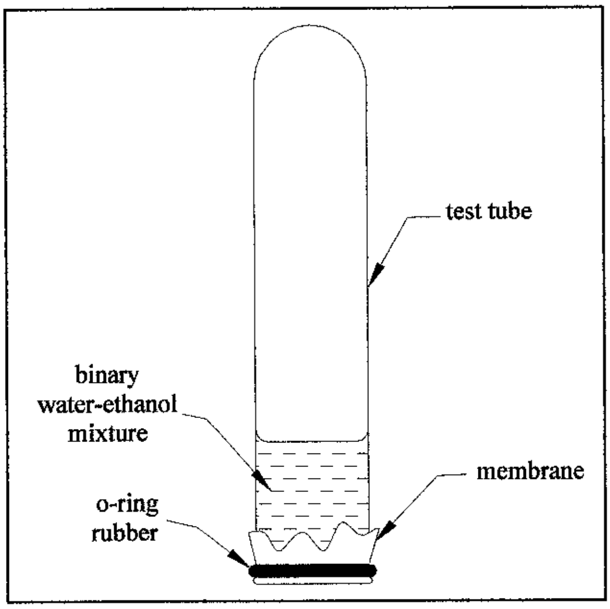

Figure 4: Layout of pervaporation experiment.

3.1.4 Analysis of Separation Capability.

The analysis of ethanol content in the experiment solution would be done by using the gas chromatograph (GC; *Shimadzu GC-2010*) equipment, equipped with flame ionization detector (FID) and a 30 meters round capillary column (*SGE BP20, Shimadzu*) with polyethylene glycol as its mobile phase. This capillary column is compatible to measure ethanol or any other volatile components in water solution. The parameters for main components of GC-FID equipment for the analysis are as listed in the table below.

Table 2 : Parameters of GC-FID equipment components.

SPL (Injection)	**Column (SGE BP20)**	**FID**
Temperature : 200°C Total Flow : 107.3 mL/min Column : 1.47 mL/min Purge : 3.0 mL/min Split Ratio : 70	Temperature : 40°C Inner Diameter : 0.22 mm Film Thickness : 0.25 μm Rate : 15°C/min	Temperature : 230°C Sampling Rate : 40 msec Stop Time : 5 mins Makeup Flow : 30 mL/min Air Flow : 400 mL/min

The FID is capable of detecting and measuring compounds from saturated, unsaturated and chlorinated hydrocarbons, aromatics, ketones and alcohols. Concentration measurement using the GC-FID was chosen due to its capability to produce accurate and precise results, simple, fast and easier to be operated compared to typical laboratory practices and other analysis equipments available.

It responds to any molecule with a carbon-hydrogen bond, but not at all, or poorly to compounds such as hydrogen sulphide (H_2S), carbon tetrachloride (CCl_4), or ammonia (NH_3). The carrier gas effluent from the GC column is mixed with hydrogen (H_2) and then routed through an unbreakable stainless steel jet. The H_2 supports a flame at the tip of the jet, ionizing the analyte molecules.

A collector electrode attracts the negative ions to the electrometer amplifier producing an analog signal which is connected to the data system input. The FID is the most commonly used GC detector, responding linearly from its minimum detectable quantity of about 100 picograms to almost 100%. The FID response is very stable from day to day, and is not susceptible to contamination from dirty samples or column bleed.

3.1.5 Characterization of Membrane.

The membrane characterizations that will be studied throughout the period are the membrane surface and its mechanical properties. The characterization of the membranes will be based on two influences, PA-6 wt% content in the membrane and of course the thermal treatment effect on the membranes. Among the equipments that would be involved in this stage are the Scanning Electron Microscopy (SEM) and Dynamic Mechanical Analysis (DMA) equipments (*Mettler Toledo, Germany*).

Electron microscopy from the SEM takes advantage of the wave nature of rapidly moving electrons, where visible light has wavelengths from 4,000 to 7,000 Angstroms, electrons accelerated to 10,000 KeV have a wavelength of 0.12 Angstroms. Optical microscopes have their resolution limited by the diffraction of light to about

1000 diameters magnification. Electron microscopes, so far, are limited to magnifications of around 1,000,000 diameters, primarily because of spherical and chromatic aberrations. Scanning electron microscope resolutions are currently limited to around 25 Angstroms, though, for a variety of reasons.

The scanning electron microscope generates a beam of electrons in a vacuum. That beam is collimated by electromagnetic condenser lenses, focused by an objective lens, and scanned across the surface of the sample by electromagnetic deflection coils. The primary imaging method is by collecting secondary electrons that are released by the sample. The secondary electrons are detected by a scintillation material that produces flashes of light from the electrons. The light flashes are then detected and amplified by a photomultiplier tube.

Meanwhile, DMA is a technique used to study and characterize materials. It is most useful for observing the viscoelastic nature of polymers. An oscillating force is applied to a sample of material and the resulting displacement of the sample is measured. From this, the stiffness of the sample can be determined, and the sample modulus can be calculated. By measuring the time lag in the displacement compared to the applied force it is possible to determine the damping properties of the material.

By definition, viscoelastic means the materials that have of both viscous as well as elastic properties. Viscous is usually meant for liquids, which means that the liquids have a relatively high resistance to flow or having the properties of glue. Elastic materials meanwhile, which typically meant for solids, are capable of resuming to its original shape after stretching or compression.

For both SEM and DMA analysis, sample preparation must be done beforehand. Since the membrane is thin and considerably brittle, extra caution must be practice to avoid the sample from damages and thus causing the result to be inaccurate. Therefore, assistance from the related technician is needed throughout the process. Sharp knife or scissor would also be required for sample cutting.

3.2 TOOLS.

The main equipments and their respective purpose of function that are required for assistance throughout the study period for completion are as listed below.

i) Casting knife – membrane formation.
ii) Drying oven – membrane preparation.
iii) SEM – membrane surface visualization.
iv) DMA – mechanical properties.
v) GC – concentration measurements.

Previously, BET equipment was also included as one of the equipments for membrane characterization but due to the equipment's current problem, porosity measurement of the membranes by using this equipment was not possible.

Since laboratory experiments is also involved throughout the process, common laboratory apparatus and glassware will also be required, especially during binary water-ethanol mixture preparation and testing in determining its pervaporation separation quality of the membranes.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 CHARACTERIZATION.

It is known that as a result of thermal treatment, the morphology changes and shrinkage of the membranes is observed. These findings are commonly explained by reorientation of polymer chains and the formation of intermolecular from intramolecular hydrogen bonds. It is obvious that similar changes in the morphology and structure occur in polyamide membranes during the thermal treatment. This is further proved during the preparation process of the membranes. By the observation made throughout the process, the physical appearances of the membranes after the thermal treatment were seen to be corrugated and shrink.

4.1.1 Surface and Structure Effect.

The effect of ethanol pervaporation separation properties of polyamide membranes was mainly caused by the rearrangement of polymer chains in the amorphous domains and therefore with the growth and formation of crystalline domains. Fast removal of entrapped water in the polymer material during the drying process hinders the collapse of pores and voids. The higher flux and lower selectivity of membranes dried in vacuum is contributed to the remaining 'defects' in the membranes, caused by slow removal of entrapped water.

As mentioned in *Chapter 2* previously, membranes prepared by the phase inversion process would have an asymmetric structure consisting of a thin top layer and a highly porous sublayer as could be seen in *Figure 12* of *Appendix D.* The separation process takes place mainly in the top layer whereas the sublayer acts as a mechanical support of the membrane. The small dimension or thickness of the top layer, which is less than 1 μm would ensures a high permeation rate because of short diffusion pathways through the separating layer (top layer).

Crystallization of polyamide resulted in a membrane with a uniform skinless microporous structure that was rapidly wetted by water. In contrast, phase separation produces a polyamide film with largely unconnected cellular voids that is as a result not wetted by water. In other words, fast removal of water helps to form a non-defected membrane structure that has well-built pores and voids for separation. This is due to the hindered collapsing of pores and voids.

The top surface would consist of polyamide spherulites which have grown together to form polygonal plates that would enhance the separation process capability. As for the bottom surface of the membranes, it exhibits a highly porous structure and also represents the morphology of a 'truncated cellular structure' which is found inside the membranes. Although the SEM equipment is capable of showing the highly porous support layer with its cellular pore structure, it could not detect the changes in membrane structure upon thermal treatment.

But the pore sizes of thermally treated membrane could be clearly seen from *Figure 9* and *10* in *Appendix D*. The pores in *Figure 9* could not been perfectly formed although after been thermally treated. 10°C above T_g of PA-6 could not provide the membrane physics to form well-arranged pores and voids, and thus it was called 'had collapsed'. Thermal treatment further exceeding T_g of PA-6 would promote the formation of desired pores and voids.

In addition, porosity or pores size of the membrane was found to increase with increasing water or decreasing PA-6 content in the casting solution. This was evidenced by comparison of the SEM images of *Figure 10* and *11* in *Appendix D*. Membrane pores in *Figure 11* that has lower PA-6 content are seen has larger pores size of the membrane in *Figure 10*, which has higher PA-6 content. Both membranes were thermally treated at equal temperature, which were at 100°C, but have different PA-6 content.

4.1.2 Mechanical Properties.

From *Table 2* below and figures in *Appendix C* obtained from the DMA equipment, it is shown that the membrane lost its viscoelastic properties at around 100°C for the first sample (20wt% PA-6 dried at 70°C) and at around 130°C for the second sample (20wt% PA-6 dried at 100°C). But the reason why the second sample have a higher temperature where it lose its viscoelastic properties is because of having thicker value (200μm) compared to the first sample (50μm). This shows that the membrane thickness have an influence in maintaining to its viscoelastic property at higher temperature. But with thicker membrane, the pervaporation rate to the membrane would certainly be decreased due to the increment of diffusion pathways through the separating layer.

Table 3 : Lost of viscoelastic property for two samples.

Sample	**Temperature**
1	100°C
2	130°C

Besides the thickness factor that made the second membrane to lost its mechanical properties at higher temperature may also caused by the thermal treatment made onto it. This is because, at 100°C, which is 40°C above T_g of PA-6, the membrane polymer has a higher chance of rearranging its structure to form a stronger ordered arrangement. Although at 70°C is also above T_g of the polymer, but the difference of 10°C could not cause and made the membrane polymer to be well-arranged.

But an error also occurred in this evaluation. The membrane should have a maximum loss modulus at its T_g due to its condition in rearranging the polymer molecules. This would be mainly due to their thicknesses that were relatively thin. Furthermore, at the end of the DMA evaluation, the samples were observed to be deformed or break, that is shown towards the end of the plotted graphs.

4.2 PERVAPORATION SEPARATION CAPABILITY.

Generally, the results obtained from the pervaporation separation experiment and analyses done for this project are as predicted theoretically. Explanations regarding the factors that influenced the effect of the separation are further described.

4.2.1 Effect of Temperature.

The compiled results shows that as the thermal treatment of the membranes are inclined, the permeate flux or percentage of ethanol loss by evaporation through pervaporation separation decreases. The decrements of ethanol loss were found to be constant for all three different polyamide content used for membranes preparation with the increment in thermal treatment temperature. It also tells us that at higher thermal treatment temperature, although there were decrements in pervaporation flux, the selectivity of the polyamide membranes in ethanol pervaporation separation process was improved for all three different polyamide content of the membrane.

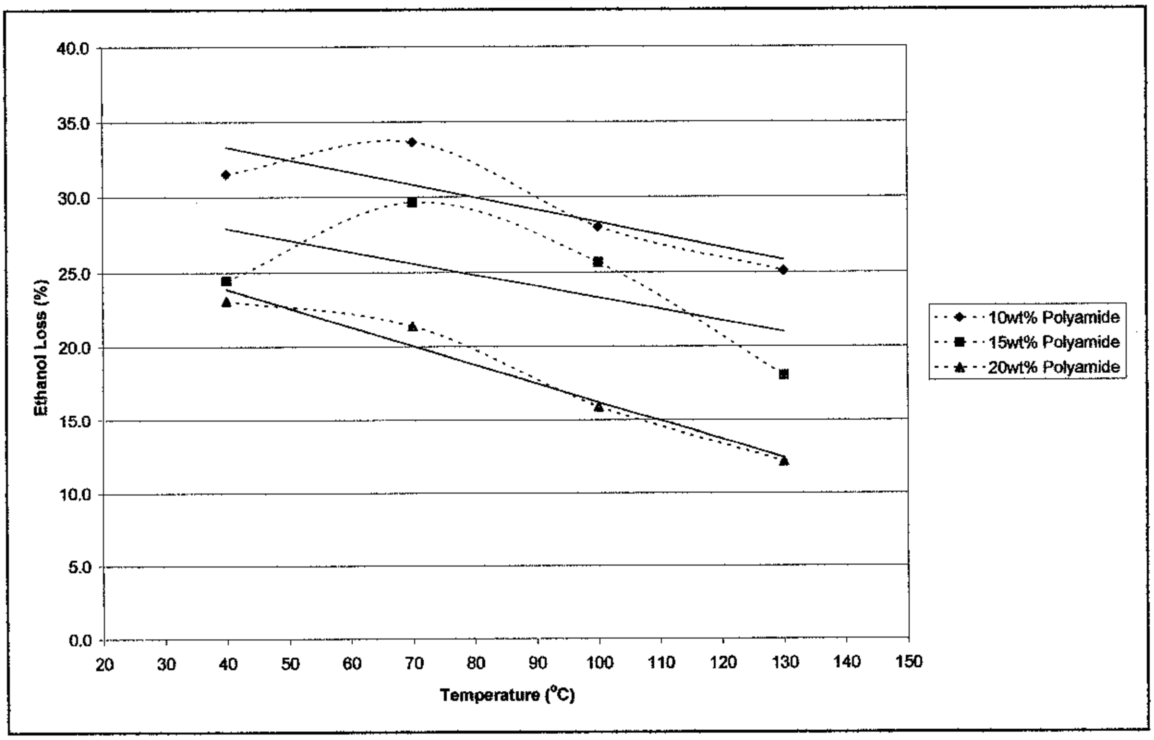

Figure 5 : Effect of thermal treatment on the percentage loss of ethanol.

This situation is further proven by the characterization of the membranes that were thermally treated as described in the section before. It was evidenced by the ethanol permeability and flux measurements throughout the study. The fast removal of water had caused the pores and voids in the membrane had been avoided from collapsed and thus forming appropriate and desired membrane structure for good separation. Higher thermally treated membrane gives a better result than lower ones.

4.2.2 Effect of PA-6 Content.

Besides of the temperature effect, comparison was also made based on the polyamide content of the membranes that were prepared. From the figure below, it obviously shows that the thermal treatment on the membranes has a greater and constant pervaporation effect at higher temperature compared to lower temperature. Thermal treatment at temperatures above T_g of polyamide, at 60°C, gives a greater reduction in ethanol loss for pervaporation separation process. This is proved by similar trendlines' slope of the thermal treatment temperature at 70°C, 100°C and 130°C.

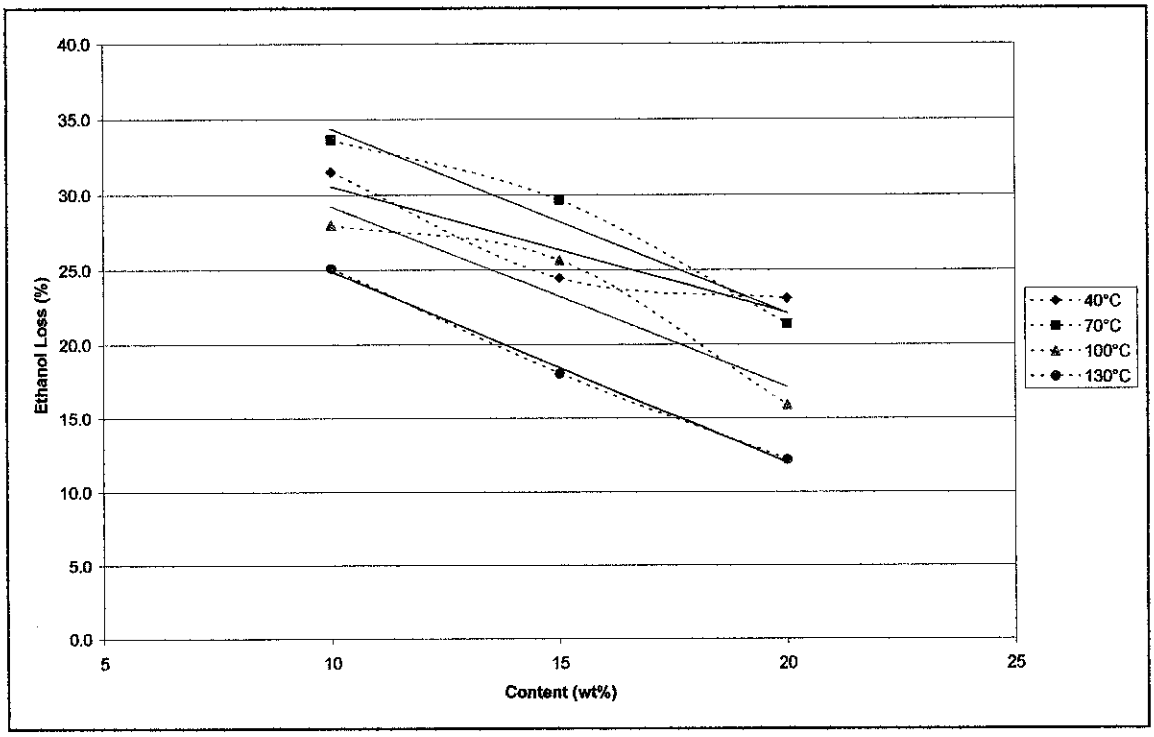

Figure 6: Effect of polyamide content on the percent loss of ethanol.

Membranes treated at 40°C gives lower reduction in percentage of ethanol loss mainly due to its thermal treatment that was below T_g of PA-6. Its permeation flux factor only depends on its PA-6 content whereas the others depend on two factors, which were the thermal treatment above T_g and PA-6 content of the membrane.

As been proven and evidenced from the characterization section previously, the pores and voids also influenced by the PA-6 content in the casting solution. Higher PA-6 content would give a higher resistance to the permeation of ethanol through the membrane compared to membrane with lower PA-6 content. With higher resistance, it could be proven that the permeation flux of ethanol during pervaporation separation process would be decrease as proved by the analysis and compiled in the figure above.

But thermal treatment at temperatures above the maximum temperature applied in this research project, which is 130°C, was not recommended because it may cause the structure of the polyamide membrane to be deformed or oxidized. With this condition occurred, a fair comparison at each thermal treatment temperature could not be made and summarize. The situation would be worse with the physical properties of the membranes prepared, which are due to its thin sheet and brittleness. It would cause the result from the pervaporation separation process to be inaccurate and inconsistent.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS.

From the experiments, analyses, and studies that were conducted throughout this project, a few conclusions could be outlined. The conclusions that are related and could be made are as listed below.

i) Asymmetric membranes from polyamide-6 prepared by the phase inversion process were suitable for pervaporation separation process of binary water-ethanol mixtures.

ii) Drying temperature of membranes above the T_g of polyamide-6 ($\approx 60^{o}C$) led to an increase in selectivity although the permeation flux is lowered.

iii) Fast removal of entrapped water in the polymer material during the drying process hinders the collapse of pores and voids.

iv) The higher flux of membranes dried is contributed to the remaining 'defects' in the membrane.

v) Thicker membrane contributed to higher resistance to the lost of viscoelastic property of the membrane.

5.2 RECOMMENDATIONS.

For this research project, a few recommendations for the sake of future enhancement in project analysis could be proposed. Among of the recommendations that could be outlined are as listed below.

i) Reduce the experiment thermal treatment temperature range to yield a more accurate result of behavior from the effect of thermal treatment changes.

ii) Change the reference unit for pervaporation separation experiment from volume to mass of the solution since the ethanol concentration was fixed using the weight percent for consistency.

iii) Literature review and membrane samples preparation could be done simultaneously at the same time to avoid the loss of time for pervaporation separation experiment later.

iv) Heating should not be involved during polyamide dissolving, which would cause solvent evaporation and the polymer solution would turn into gel when stored for usage later.

v) Specific and suitable place for pervaporation separation experiment is prepared, having sufficient ventilation as safety measure.

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APPENDIX A

Analytical equipments used throughout
the study.

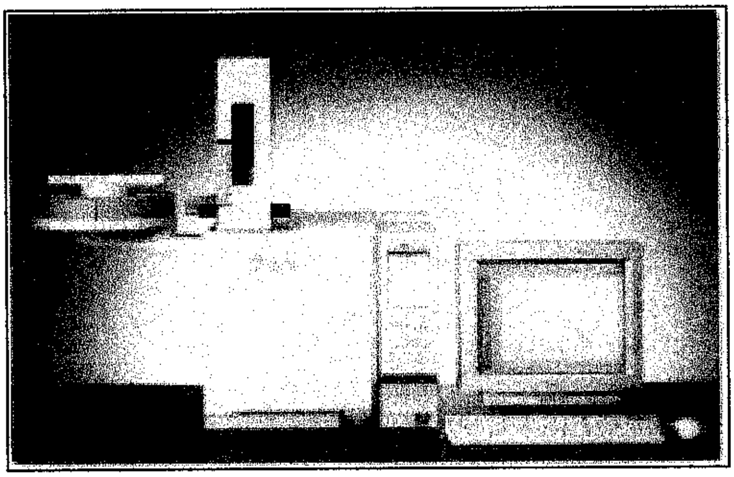

Figure 7: Shimadzu GC-2010 Gas Chromatograph

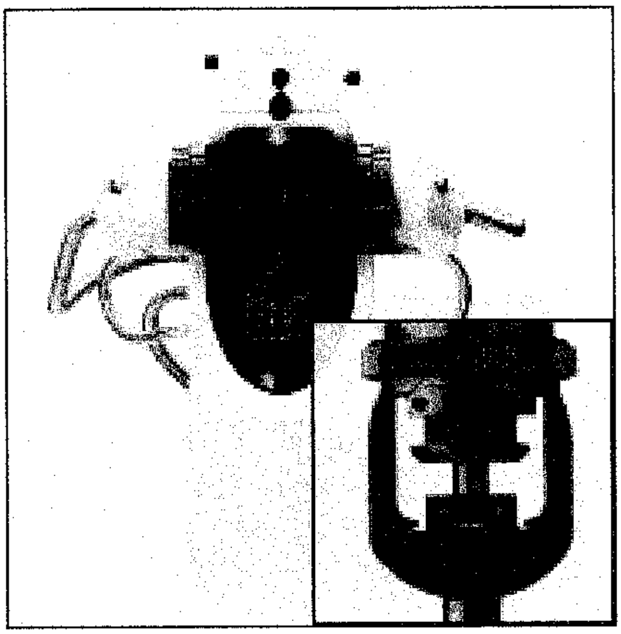

Figure 8 : Mettler-Toledo Dynamic Mechanical Analyzer (DMA).

APPENDIX B

Data from pervaporation separation
capability by using GC-FID.

Table 4 : Data for pervaporation separation experiment.

Sample	Run	Weight (g)		Average Thickness (μm)	Ethanol Concentration (wt%)	Average Ethanol Concentration wt(%)
		Initial	Final			
10wt% @ 40°C	1	34.70	32.26	*	30.719	27.392
	2	34.78	32.83	100	24.064	
15wt% @ 40°C	1	34.72	32.74	*	31.181	30.231
	2	34.55	32.37	*	29.281	
20wt% @ 40°C	1	34.48	32.33	*	31.166	30.772
	2	34.65	32.79	80	30.378	
10wt% @ 70°C	1	34.76	32.71	50	26.031	26.539
	2	34.67	32.37	40	27.046	
15wt% @ 70°C	1	34.52	32.78	110	26.212	28.145
	2	34.75	32.83	60	30.078	
20wt% @ 70°C	1	34.46	32.77	*	33.899	31.447
	2	34.30	32.39	*	28.994	
10wt% @ 100°C	1	34.55	32.65	80	26.687	28.796
	2	34.65	32.19	70	30.905	
15wt% @ 100°C	1	34.65	32.52	*	31.955	29.742
	2	34.86	33.21	50	27.529	
20wt% @ 100°C	1	34.94	33.17	*	34.845	33.620
	2	34.90	32.84	*	32.394	
10wt% @ 130°C	1	34.95	33.10	70	25.527	29.961
	2	34.80	33.13	75	34.395	
15wt% @ 130°C	1	34.42	31.86	70	30.494	32.775
	2	34.78	32.87	75	35.056	
20wt% @ 130°C	1	34.58	32.73	60	35.609	35.128
	2	34.55	32.86	95	34.647	

* No thickness value due to unavailability of micrometer at the moment of experiment.

Sample Information

alysis Date & Time : 4/29/2005 4:19:43 PM
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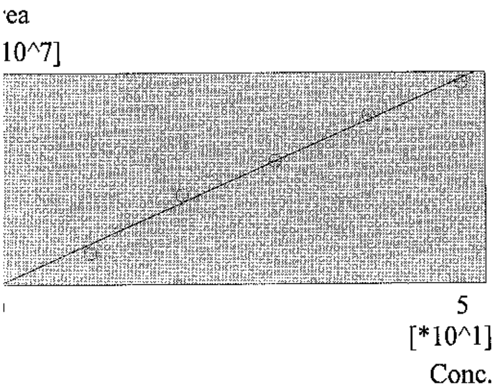
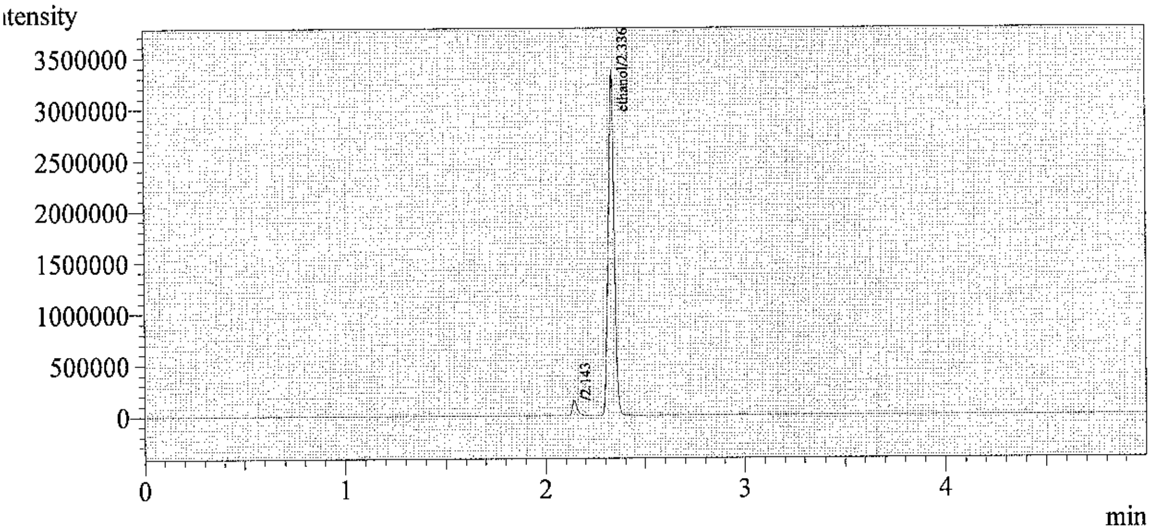
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 10wt%@40C C:\GCsolution\Data\FYPjan2005\fathullah\10wt%@40C.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.143	240357	143558	0.000		
2	2.336	6976008	3330113	30.719	%	ethanol
Total		7216364	3473671			

Sample Information

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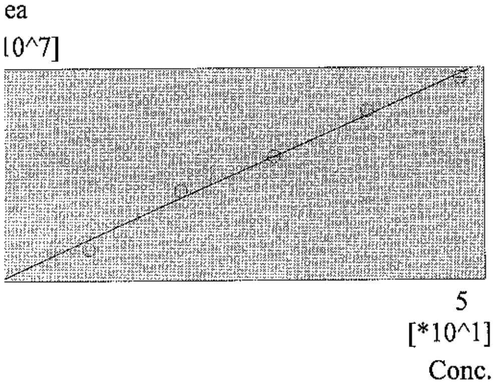
Calibration Curve - Analytical Line 1 - Channel 1

.me:ethanol



No.	Conc.	Area
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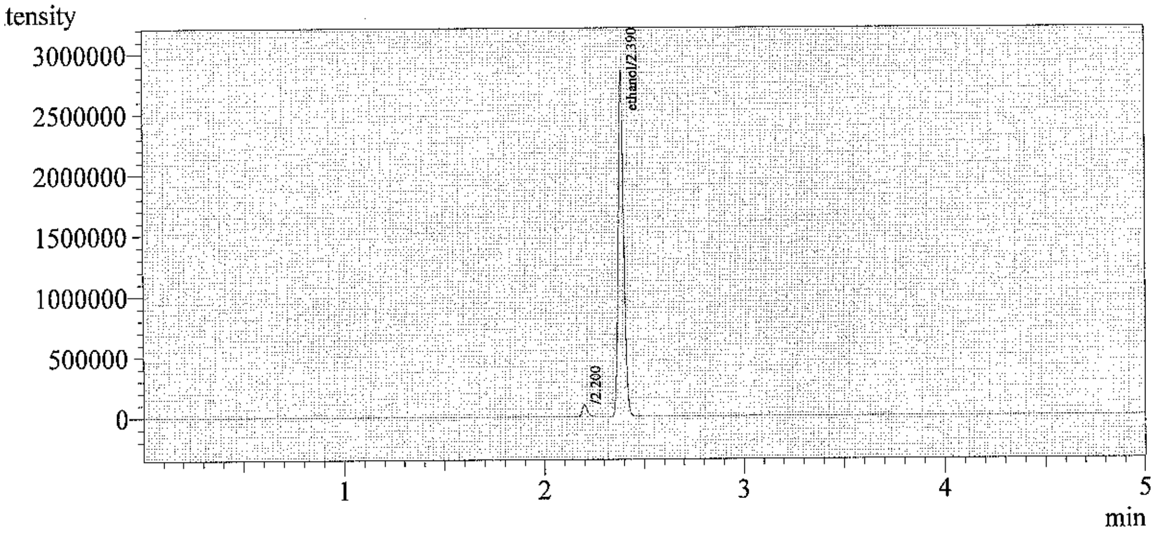
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 10wt%@40C C:\GCsolution\Data\FYPjan2005\fathullah\10wt%@40C-2.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
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2	2.390	5464597	2809763	24.064	%	ethanol
Total		5632594	2907491			

Sample Information

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Calibration Curve - Analytical Line 1 - Channel 1

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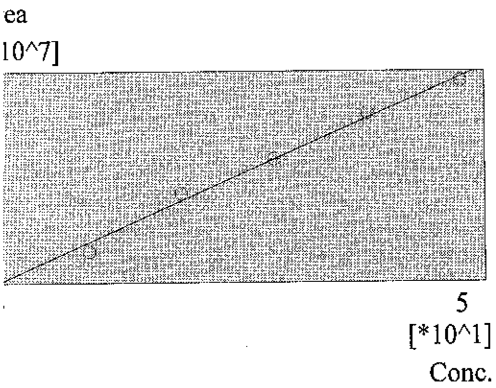

No.	Conc.	Area
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3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

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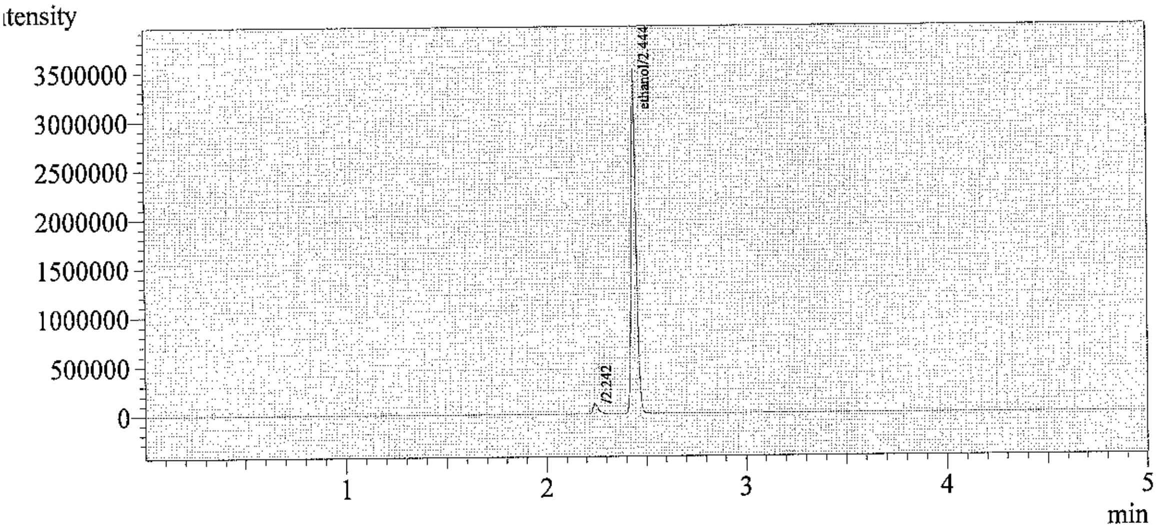

Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.242	189487	102774	0.000		
2	2.444	5911393	3434362	26.031	%	ethanol
Total		6100879	3537136			

Sample Information

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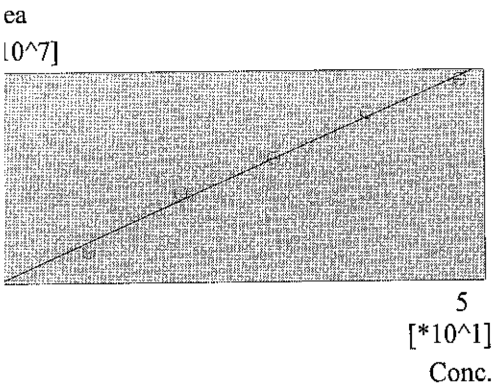
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



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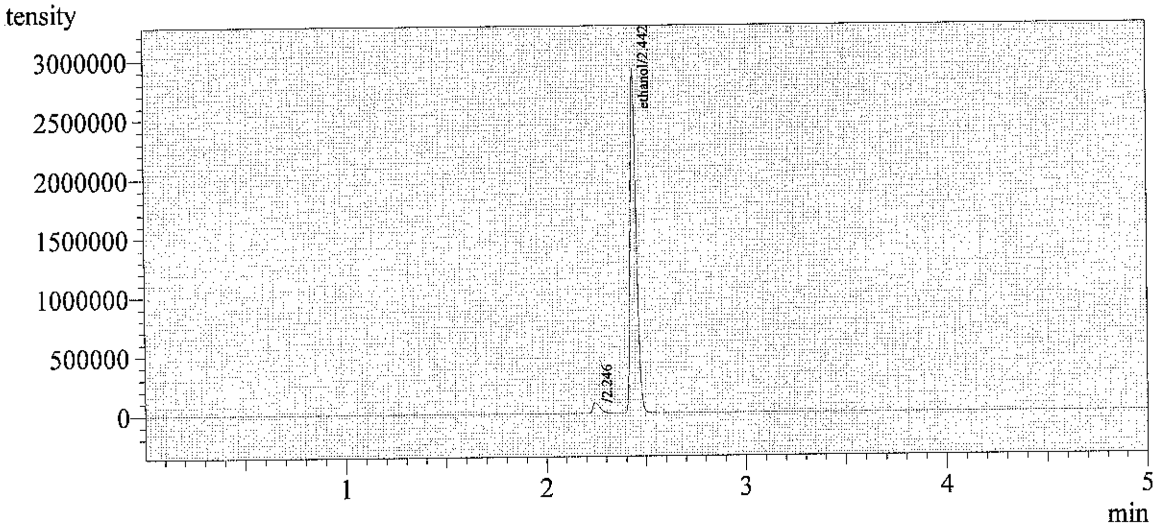
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 10wt%@70C C:\GCsolution\Data\FYPjan2005\fathullah\10wt%@70C-2.gcd



Peak Table - Channel 1

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1	2.246	216459	88957	0.000		
2	2.442	6141803	2839293	27.046	%	ethanol
Total		6358262	2928250			

Sample Information

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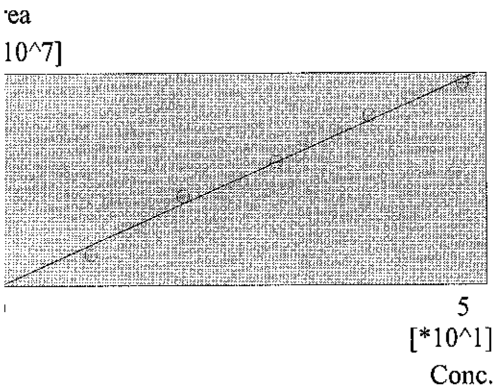
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
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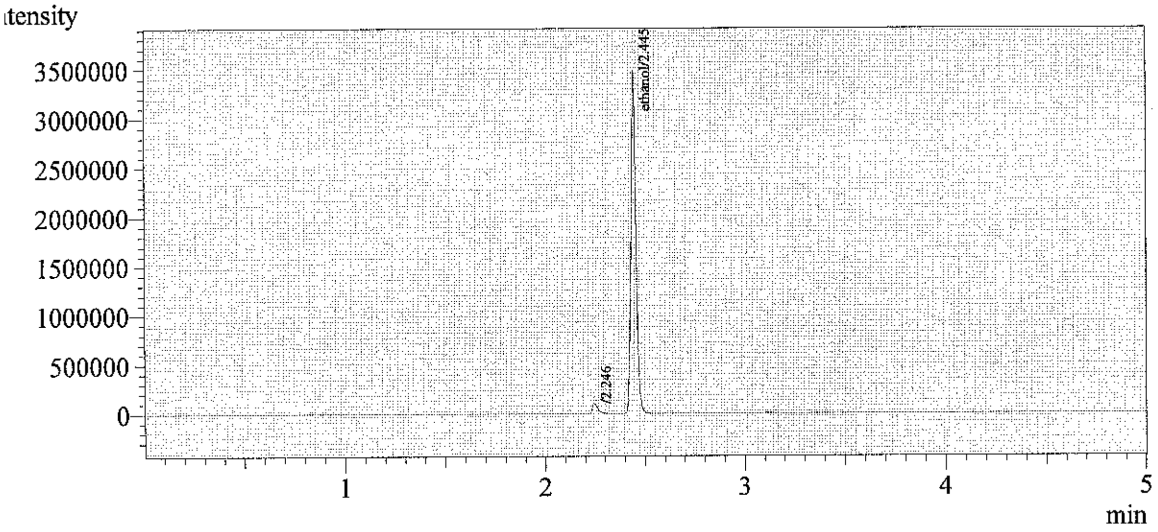
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 10wt%@100C C:\GCsolution\Data\FYPjan2005\fathullah\10wt%@100C.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.246	188869	99505	0.000		
2	2.445	6060311	3404687	26.687	%	ethanol
Total		6249180	3504192			

Sample Information

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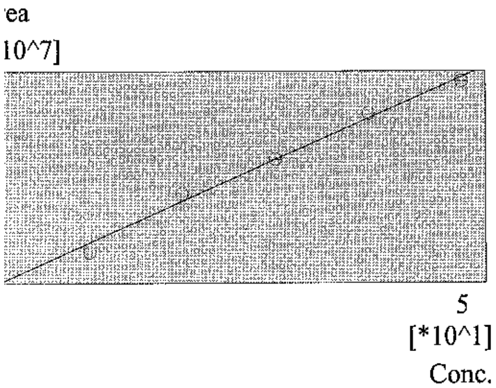
Calibration Curve - Analytical Line 1 - Channel 1

ıme:ethanol



No.	Conc.	Area
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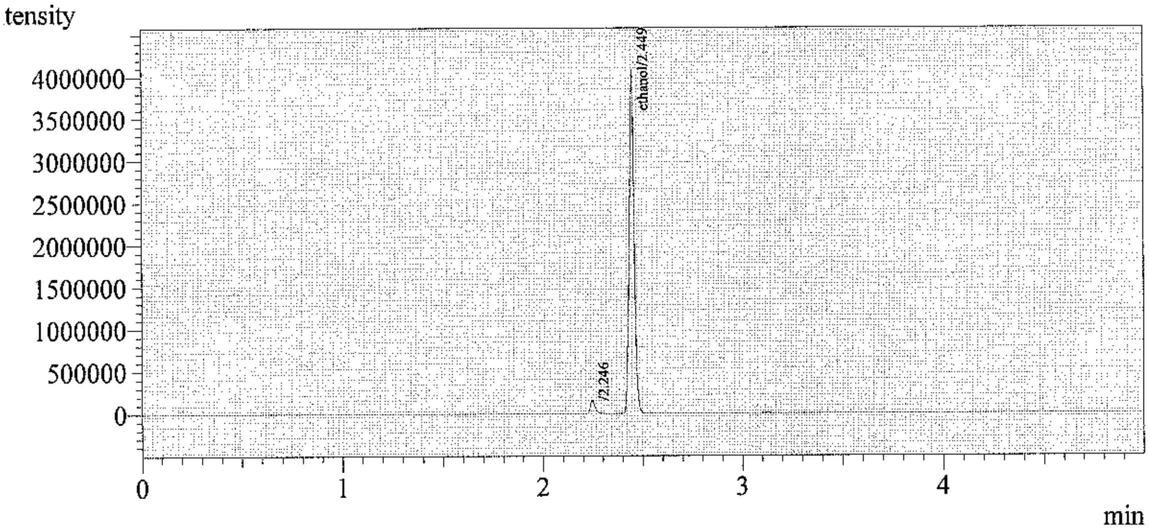
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 10wt%@100C C:\GCsolution\Data\FYPjan2005\fathullah\10wt%@100C-2.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.246	252033	147748	0.000		
2	2.449	7018131	3936785	30.905	%	ethanol
Total		7270164	4084533			

Sample Information

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Calibration Curve - Analytical Line 1 - Channel 1

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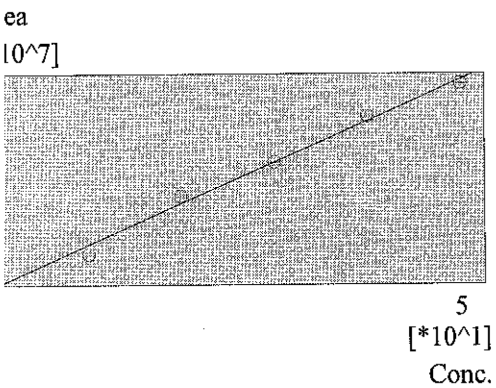

No.	Conc.	Area
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4	40.000	9321582
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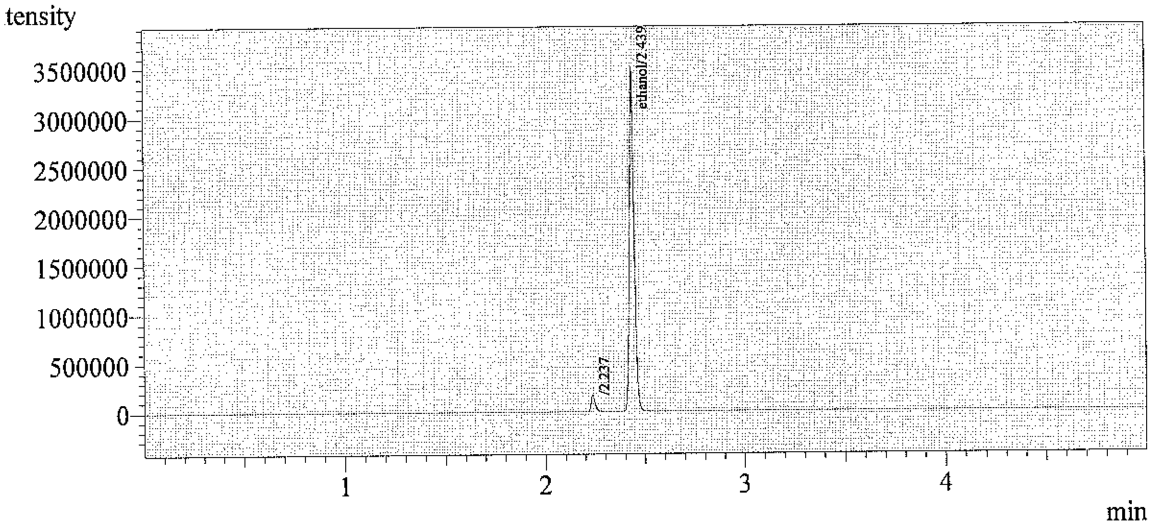
Chromatogram - Channel 1 10wt%@130C C:\GCsolution\Data\FYPjan2005\fathullah\10wt%@130C.gcd



Peak Table - Channel 1

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1	2.237	226509	163684	0.000		
2	2.439	5796945	3310944	25.527	%	ethanol
Total		6023454	3474628			

Sample Information

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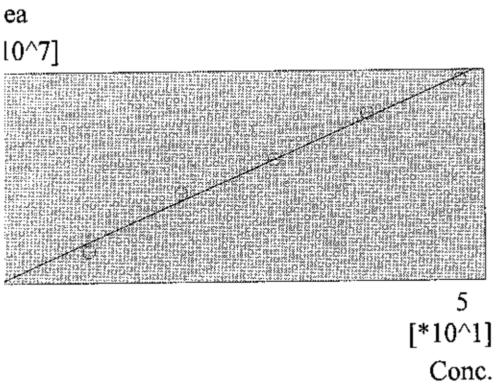
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
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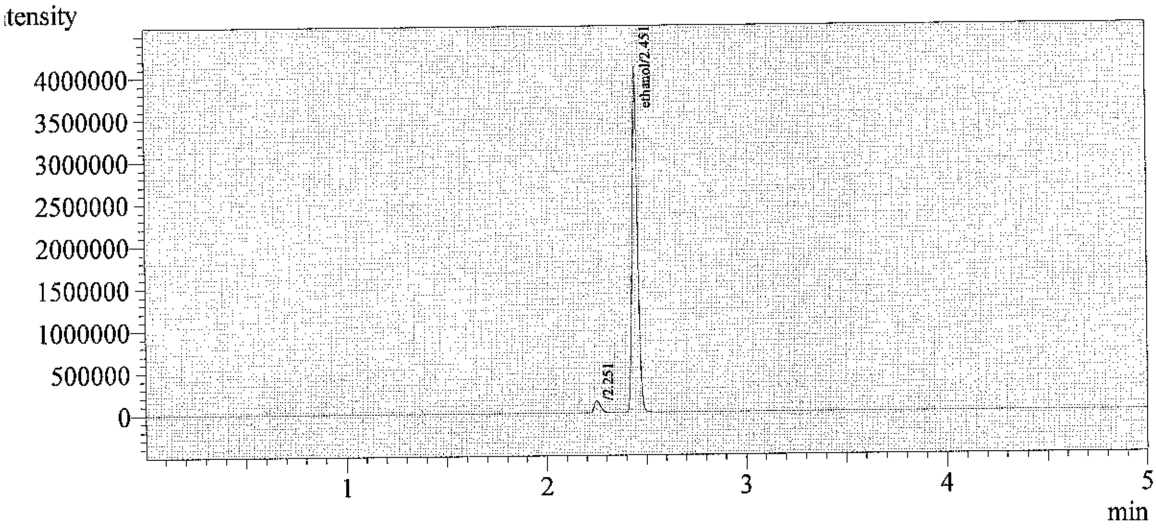

Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.251	299373	137794	0.000		
2	2.451	7810791	4041085	34.395	%	ethanol
Total		8110164	4178879			

Sample Information

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Calibration Curve - Analytical Line 1 - Channel 1

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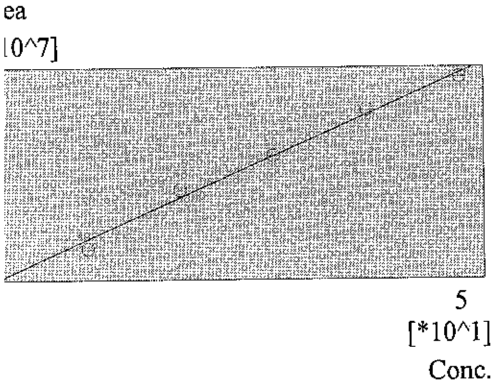

No.	Conc.	Area
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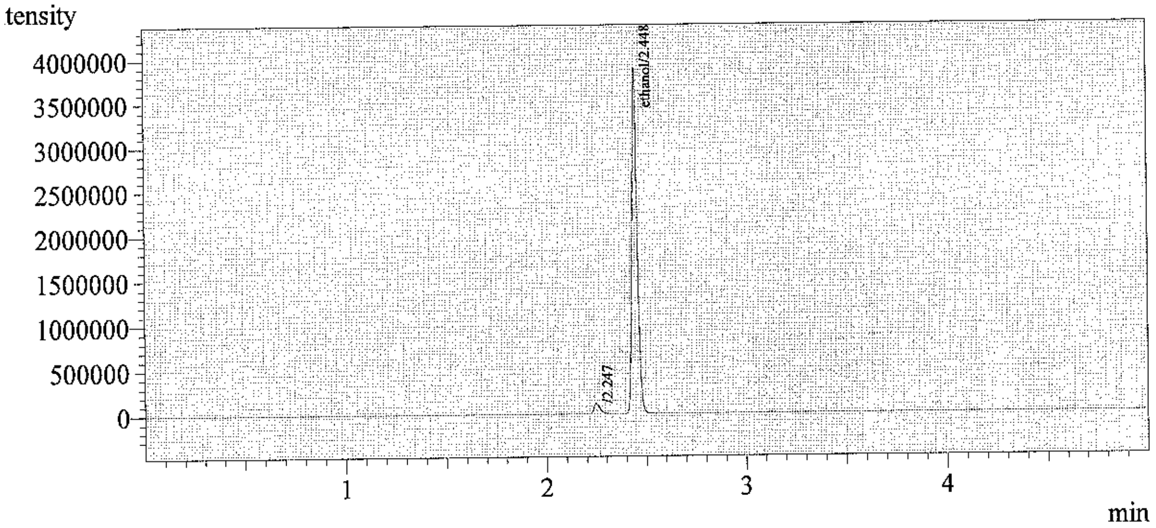
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 15wt%@40C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@40C.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.247	240423	111239	0.000		
2	2.448	7080902	3692928	31.181	%	ethanol
Total		7321326	3804167			

Sample Information

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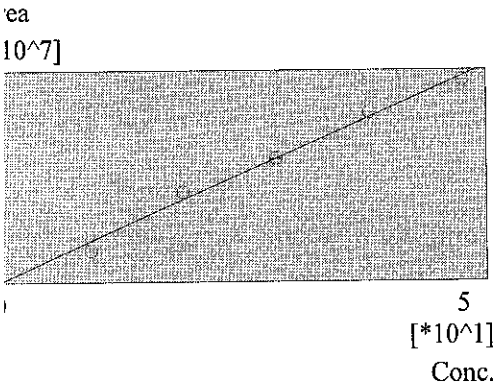
Calibration Curve - Analytical Line 1 - Channel 1

ıme:ethanol



No.	Conc.	Area
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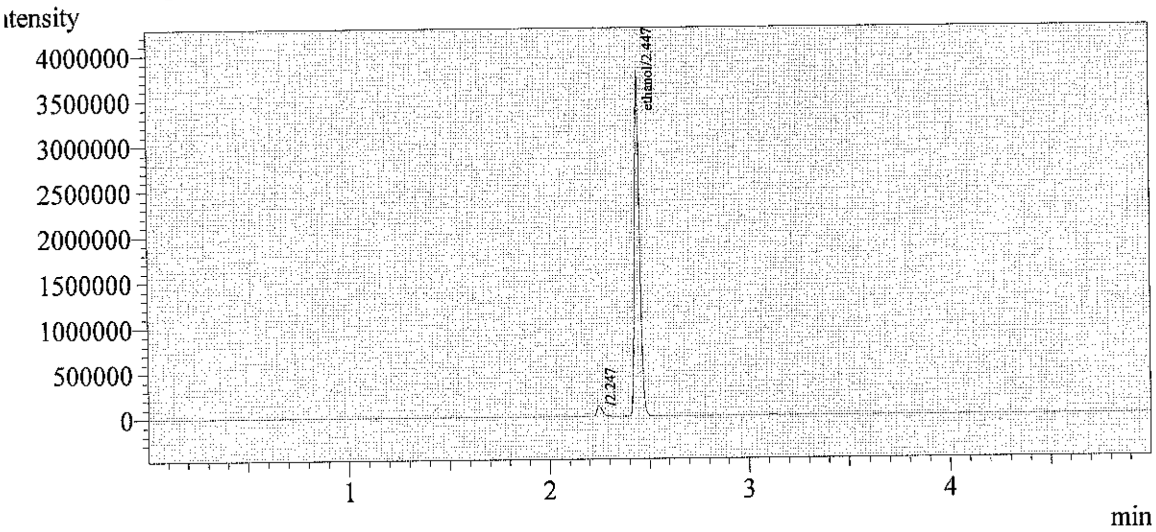
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 15wt%@40C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@40C-2.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.247	221915	112088	0.000		
2	2.447	6649300	3542353	29.281	%	ethanol
Total		6871215	3654441			

Sample Information

alysis Date & Time : 5/5/2005 12:03:20 PM
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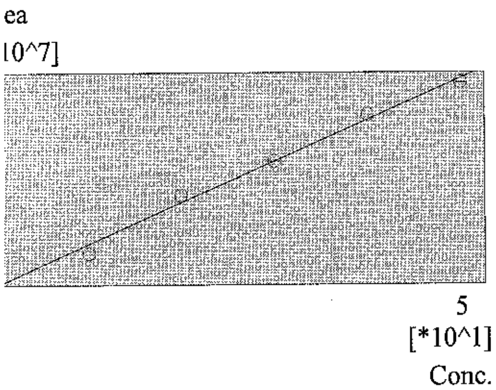
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
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3	30.000	6812707
4	40.000	9321582
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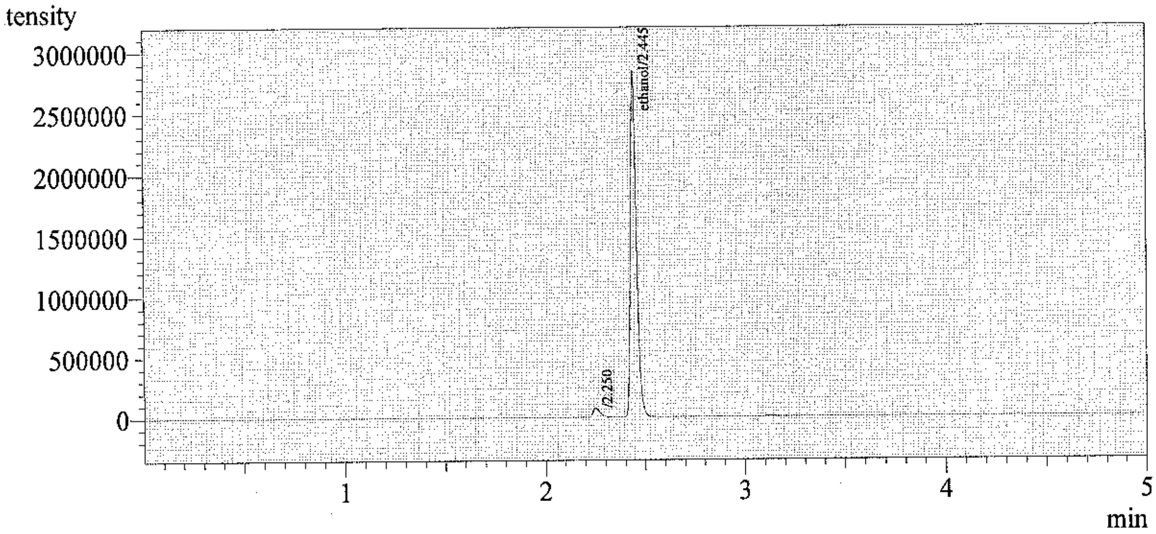
Chromatogram - Channel 1 15wt%@70C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@70C.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.250	180834	73996	0.000		
2	2.445	5952522	2800664	26.212	%	ethanol
Total		6133356	2874660			

Sample Information

alysis Date & Time : 5/10/2005 12:32:21 PM
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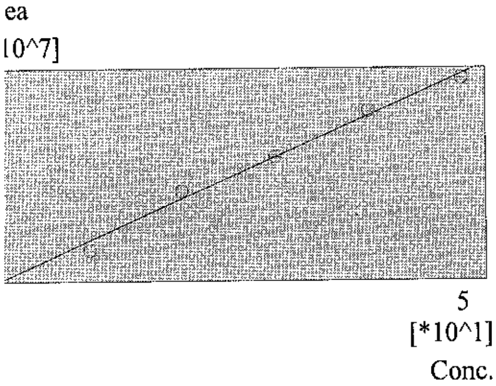
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
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2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
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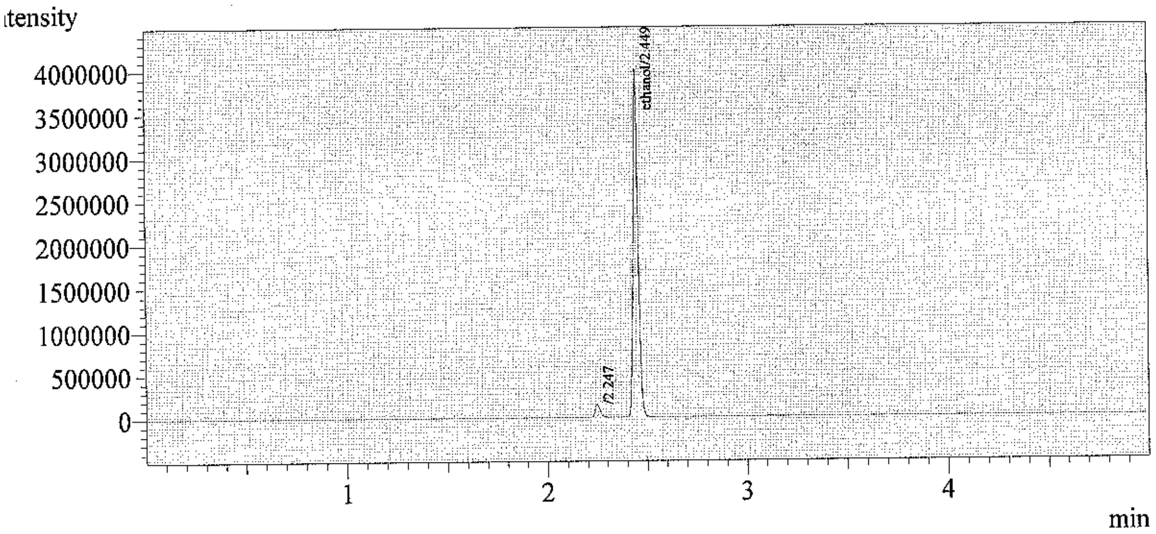
Chromatogram - Channel 1 15wt%@70C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@70C-2.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.247	248394	137683	0.000		
2	2.449	6830301	3829312	30.078	%	ethanol
Total		7078696	3966995			

Sample Information

alysis Date & Time : 4/29/2005 4:56:40 PM
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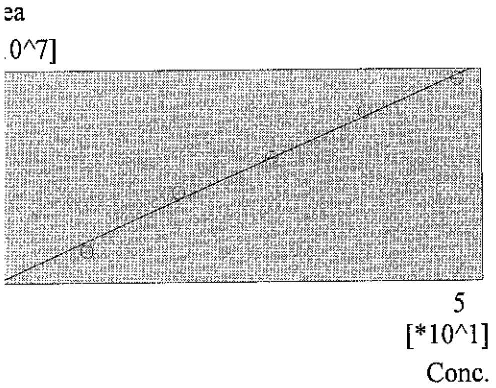
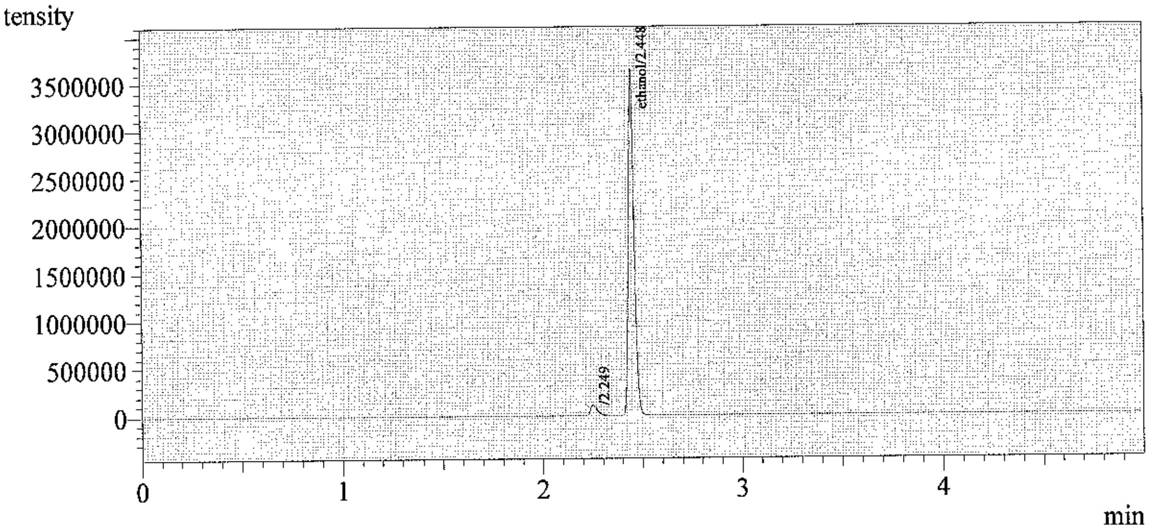
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 15wt%@100C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@100C.gcd



Peak Table - Channel 1

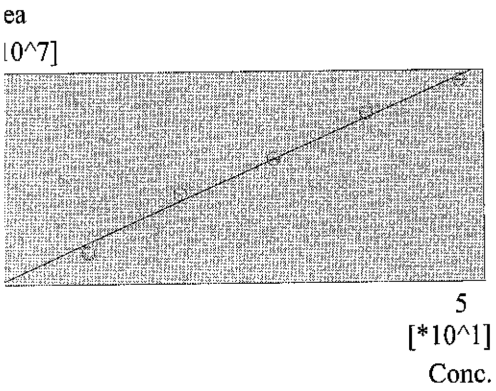
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.249	244804	103418	0.000		
2	2.448	7256650	3542456	31.955	%	ethanol
Total		7501453	3645874			

Sample Information

alysis Date & Time : 5/5/2005 12:22:04 PM
mple Name : 15wt%@100C
ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@100C-2.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

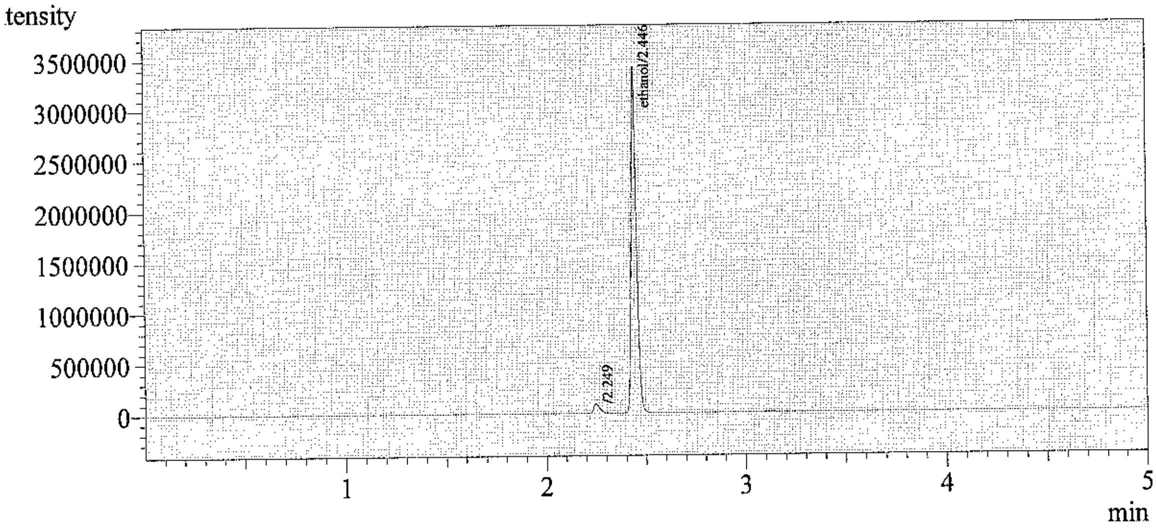
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 15wt%@100C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@100C-2.gcd



Peak Table - Channel 1

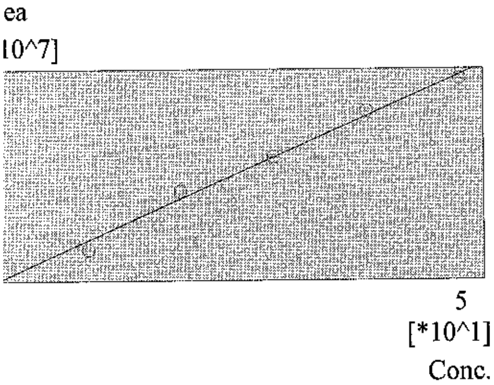
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.249	192187	90773	0.000		
2	2.446	6251421	3305561	27.529	%	ethanol
Total		6443607	3396334			

Sample Information

alysis Date & Time : 5/10/2005 12:51:04 PM
mple Name : 15wt%@130C
ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@130C.gcd
ethod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

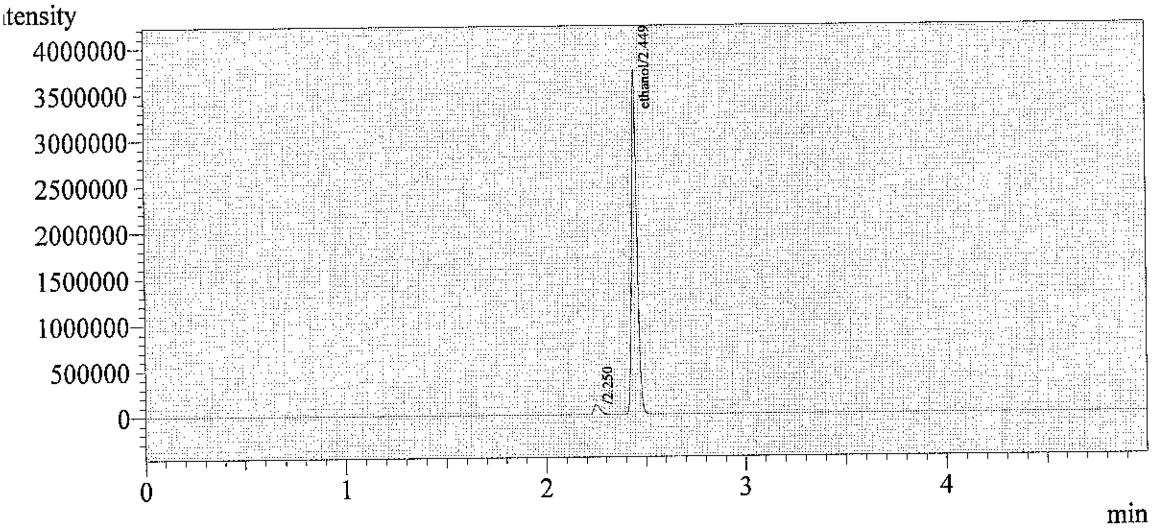
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 15wt%@130C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@130C.gcd



Peak Table - Channel 1

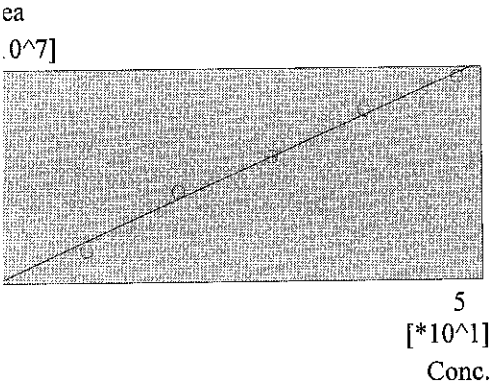
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.250	232512	105710	0.000		
2	2.449	6924840	3656709	30.494	%	ethanol
Total		7157353	3762419			

Sample Information

.alysis Date & Time : 5/10/2005 1:00:28 PM
mple Name : 15wt%@130C
.ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@130C-2.gcd
:thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

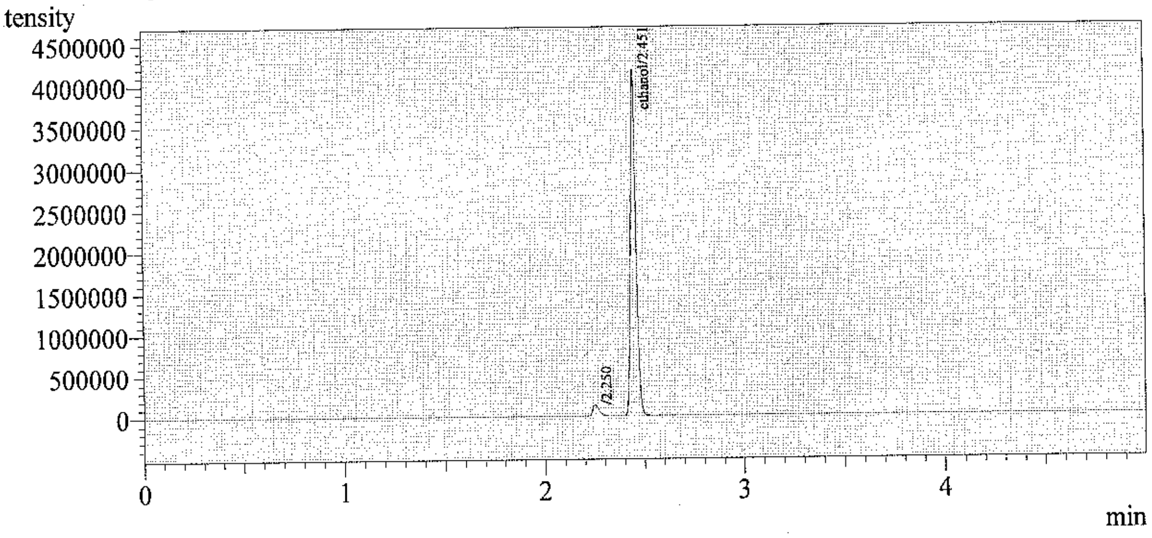
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 15wt%@130C C:\GCsolution\Data\FYPjan2005\fathullah\15wt%@130C-2.gcd



Peak Table - Channel 1

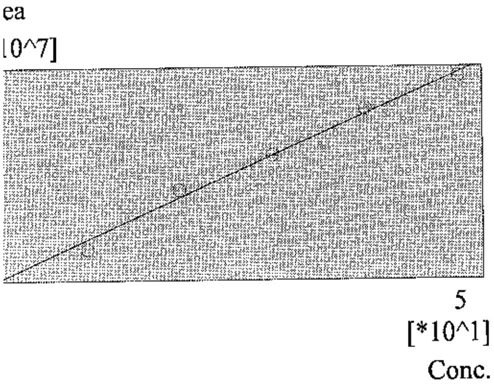
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.250	299108	133466	0.000		
2	2.451	7960903	4102620	35.056	%	ethanol
Total		8260011	4236087			

Sample Information

alysis Date & Time : 4/29/2005 4:38:08 PM
mple Name : 20wt%@40C
ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@40C.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

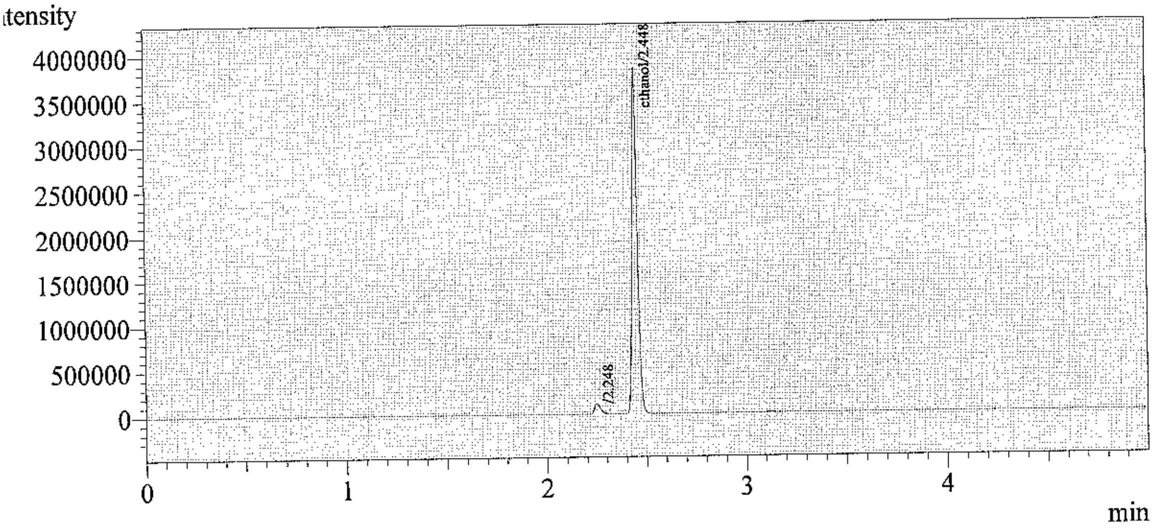
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@40C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@40C.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.248	243852	114657	0.000		
2	2.448	7077381	3689341	31.166	%	ethanol
Total		7321233	3803999			

Sample Information

alysis Date & Time : 5/13/2005 11:51:34 AM
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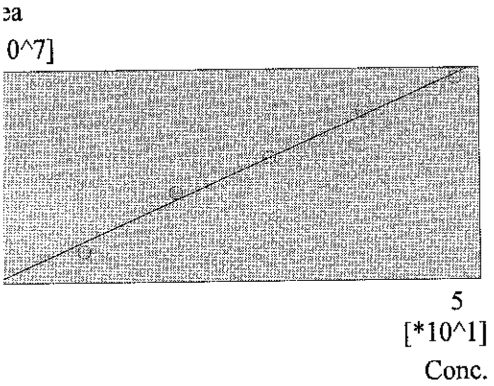
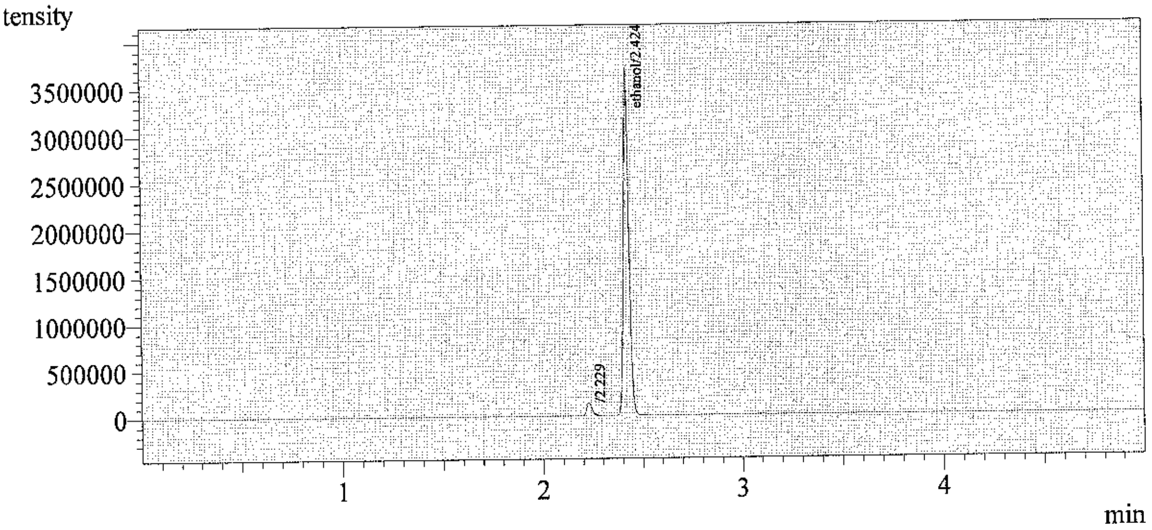
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@40C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@40C-3.gcd



Peak Table - Channel 1

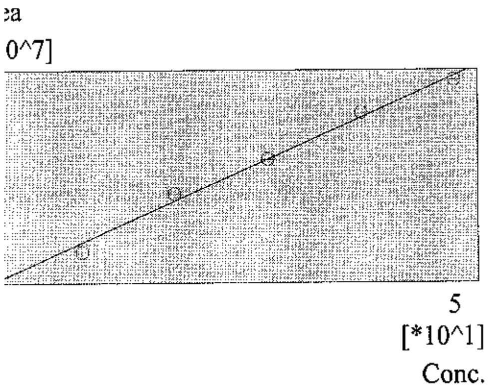
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.229	265630	138162	0.000		
2	2.424	6898459	3628669	30.378	%	ethanol
Total		7164089	3766830			

Sample Information

alysis Date & Time : 4/15/2005 4:35:26 PM
nple Name : 20wt%@70C
ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@70C.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

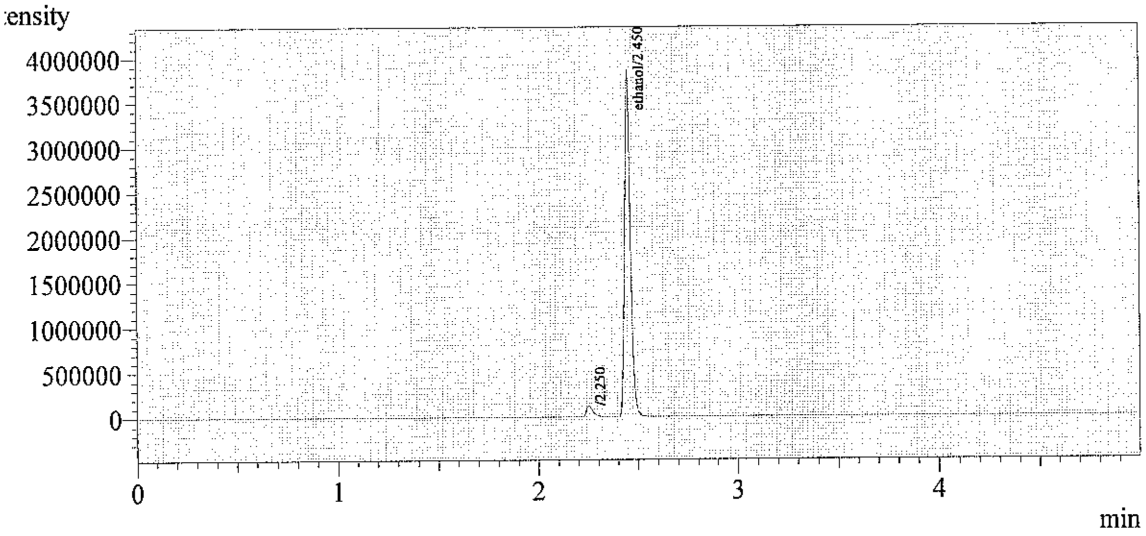
Calibration Curve - Analytical Line 1 - Channel 1

ne:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@70C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@70C.gcd



Peak Table - Channel 1

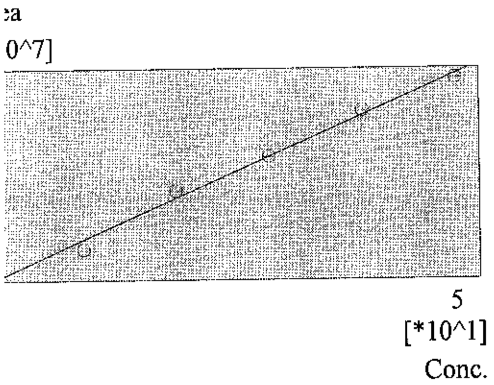
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.250	273816	121677	0.000		
2	2.450	7697971	3795925	33.899	%	ethanol
Total		7971787	3917601			

Sample Information

alysis Date & Time : 4/29/2005 4:47:25 PM
nple Name : 20wt%@70C
ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@70C-2.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

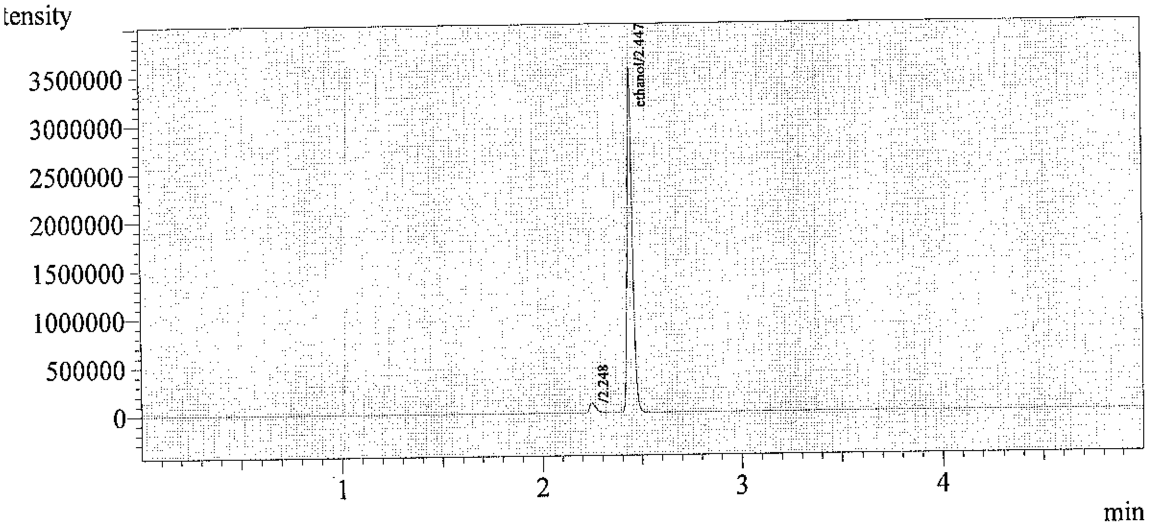
Calibration Curve - Analytical Line 1 - Channel 1

ne:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@70C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@70C-2.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.248	216566	99501	0.000		
2	2.447	6584305	3404354	28.994	%	ethanol
Total		6800872	3503855			

Sample Information

alysis Date & Time : 4/15/2005 4:44:39 PM
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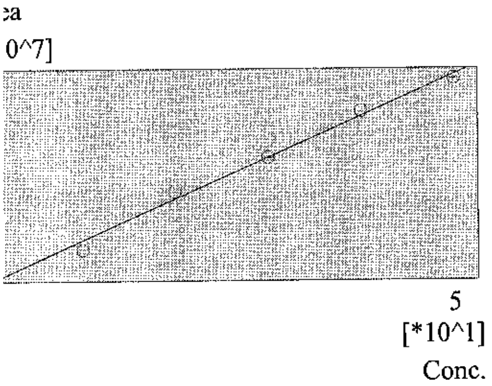
ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@100C.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

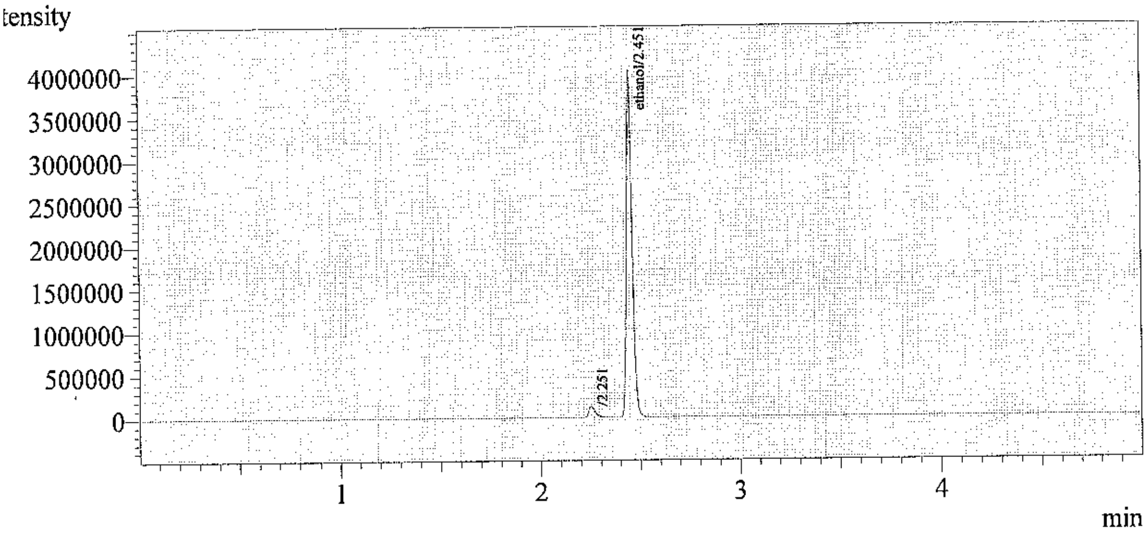
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@100C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@100C.gcd



Peak Table - Channel 1

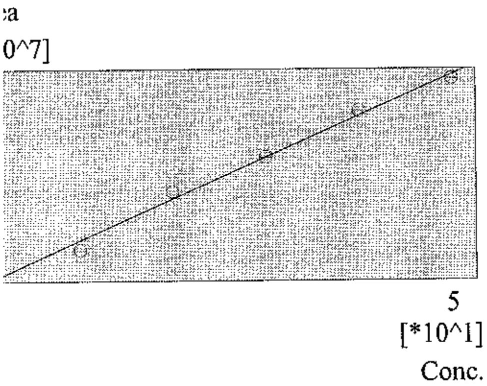
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.251	281153	125661	0.000		
2	2.451	7912877	3985917	34.845	%	ethanol
Total		8194030	4111578			

Sample Information

alysis Date & Time : 4/29/2005 5:05:58 PM
nple Name : 20wt%@100C
a Name : C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@100C-2.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

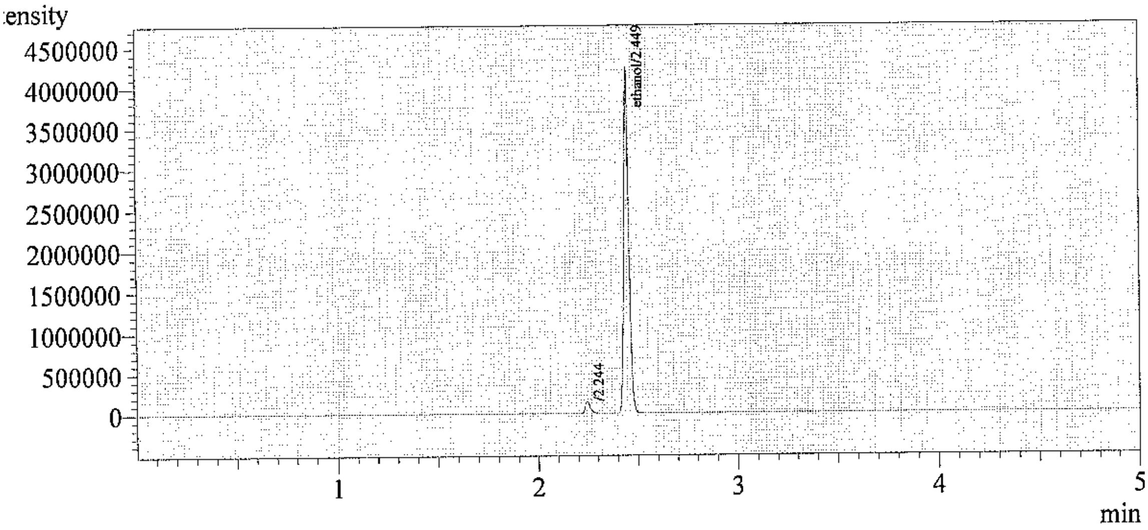
Calibration Curve - Analytical Line 1 - Channel 1

ne:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@100C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@100C-2.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.244	252497	145889	0.000		
2	2.449	7356330	4020315	32.394	%	ethanol
Total		7608827	4166204			

Sample Information

alysis Date & Time : 5/10/2005 1:09:52 PM
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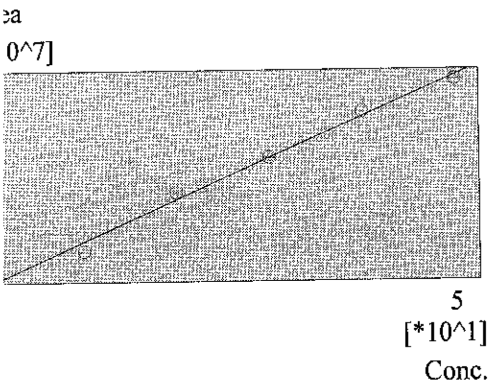
ta Name : C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@130C.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

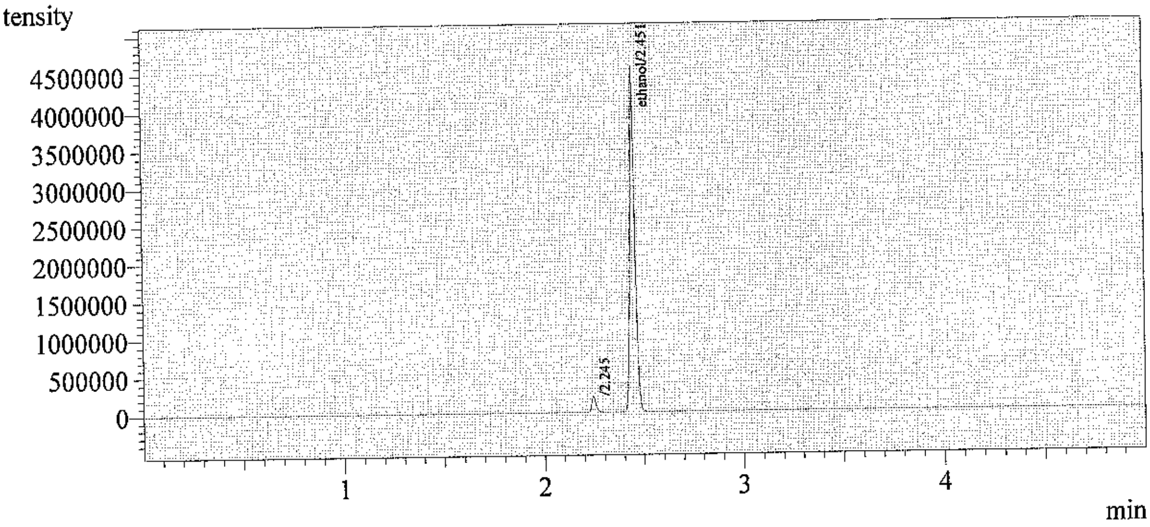
Calibration Curve - Analytical Line 1 - Channel 1

me:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@130C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@130C.gcd



Peak Table - Channel 1

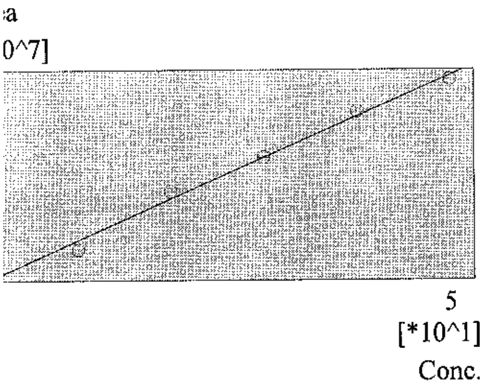
Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.245	309698	208461	0.000		
2	2.451	8086308	4473759	35.609	%	ethanol
Total		8396006	4682220			

Sample Information

lysis Date & Time : 5/11/2005 9:46:59 AM
nple Name : 20wt%@130C
a Name : C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@130C-2.gcd
thod Name : C:\GCsolution\Data\FYPjan2005\fathullah\etOH.gcm

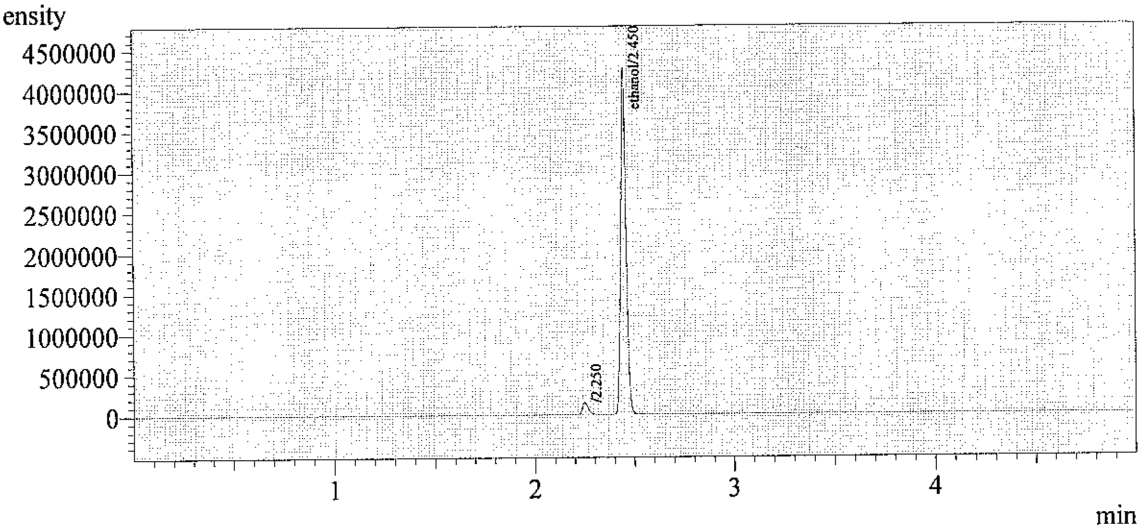
Calibration Curve - Analytical Line 1 - Channel 1

ne:ethanol



No.	Conc.	Area
1	10.000	1724955
2	20.000	4930706
3	30.000	6812707
4	40.000	9321582
5	50.000	11117602

Chromatogram - Channel 1 20wt%@130C C:\GCsolution\Data\FYPjan2005\fathullah\20wt%@130C-2.gcd

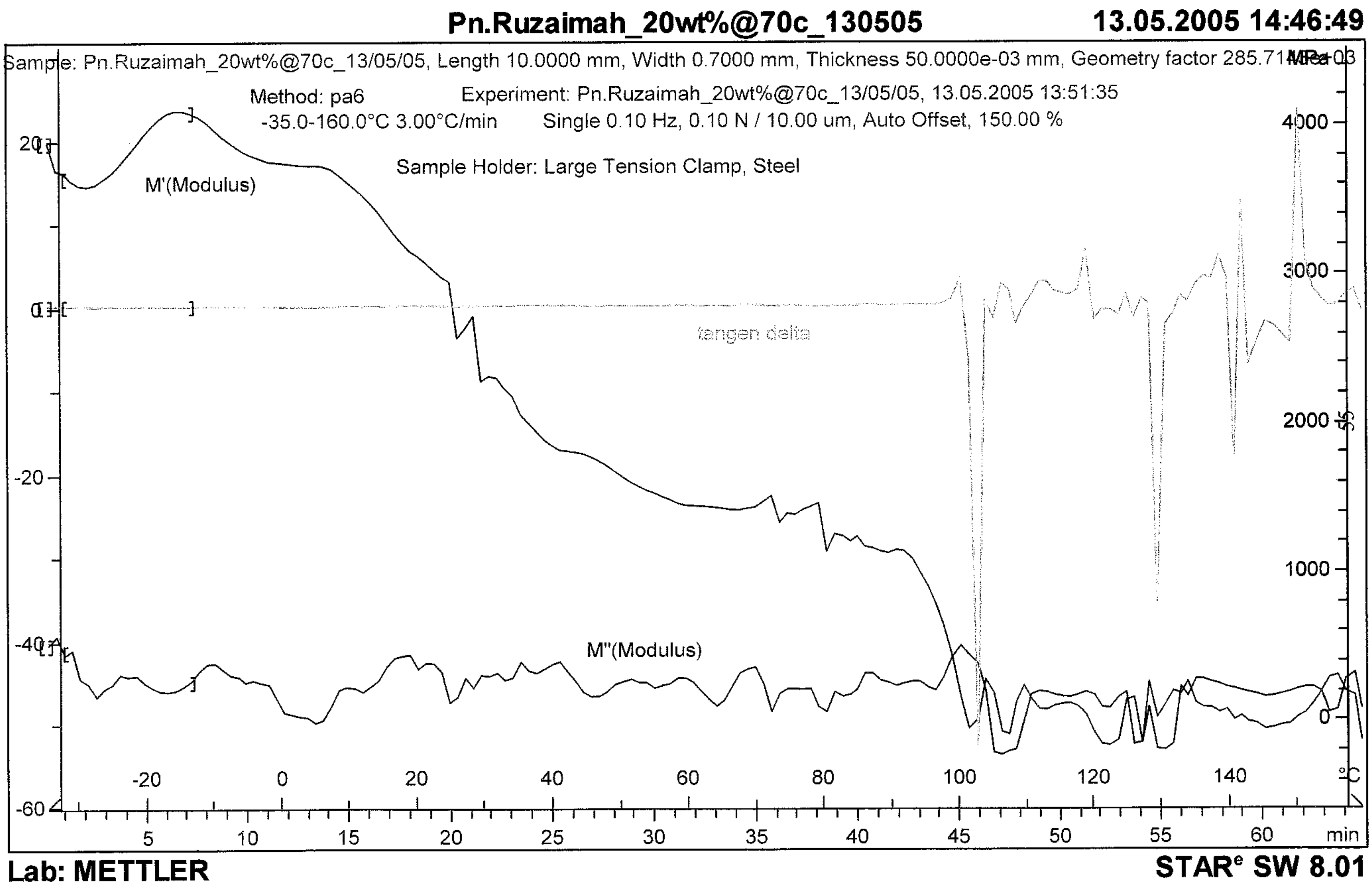


Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Cmpd Name
1	2.250	298539	142680	0.000		
2	2.450	7868030	4190785	34.647	%	ethanol
Total		8166569	4333466			

APPENDIX C

Data from characterization of membrane
by using DMA.



Pn.Ruzaimah_20wt%@70c_130505 **13.05.2005 14:46:49**

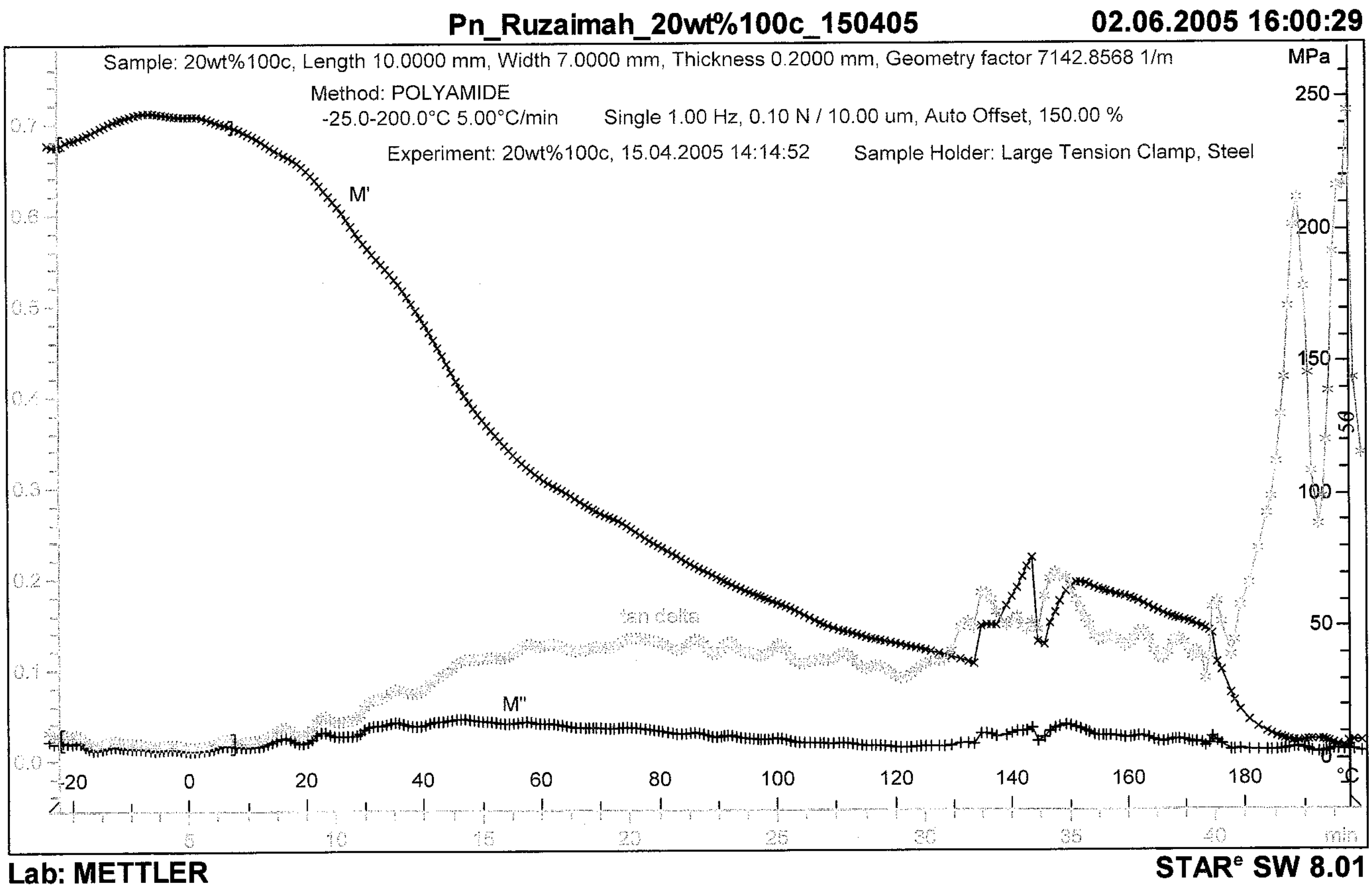
Sample: Pn.Ruzaimah_20wt%@70c_13/05/05, Length 10.0000 mm, Width 0.7000 mm, Thickness 50.0000e-03 mm, Geometry factor 285.71[illegible]e+03

Method: pa6 Experiment: Pn.Ruzaimah_20wt%@70c_13/05/05, 13.05.2005 13:51:35

-35.0-160.0°C 3.00°C/min Single 0.10 Hz, 0.10 N / 10.00 um, Auto Offset, 150.00 %

Sample Holder: Large Tension Clamp, Steel

Lab: METTLER **STAR^e SW 8.01**



Pn_Ruzaimah_20wt%100c_150405 **02.06.2005 16:00:29**

Sample: 20wt%100c, Length 10.0000 mm, Width 7.0000 mm, Thickness 0.2000 mm, Geometry factor 7142.8568 1/m

Method: POLYAMIDE
-25.0-200.0°C 5.00°C/min Single 1.00 Hz, 0.10 N / 10.00 um, Auto Offset, 150.00 %

Experiment: 20wt%100c, 15.04.2005 14:14:52 Sample Holder: Large Tension Clamp, Steel

Lab: METTLER **STAR^e SW 8.01**

APPENDIX D

Data from characterization of membrane
by using SEM.

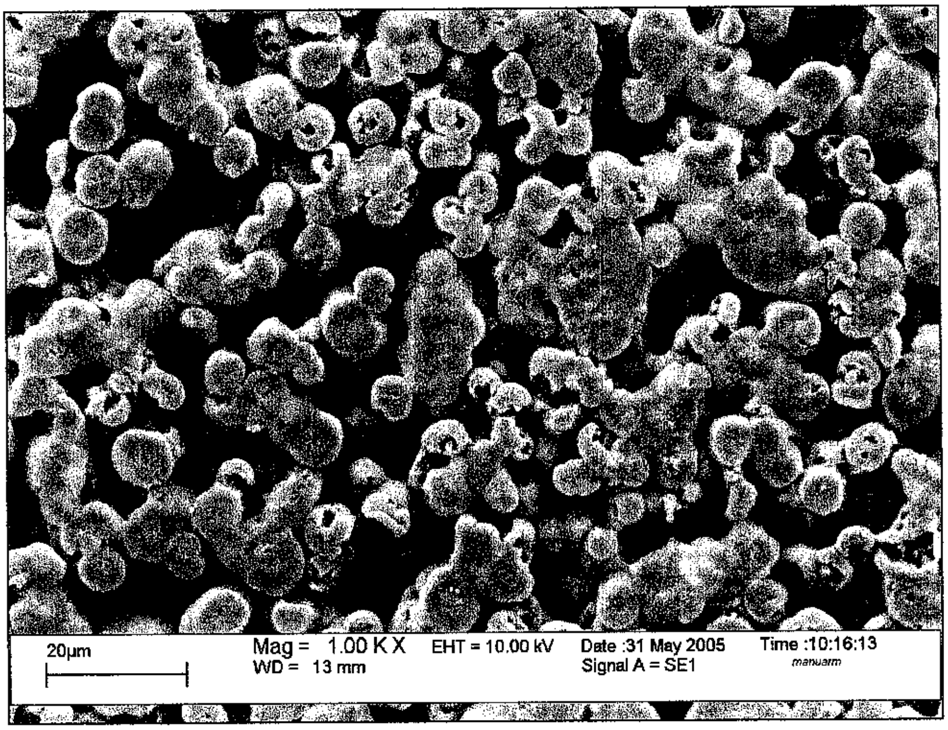


Figure 9 : 15wt% PA-6 content dried at 70°C.

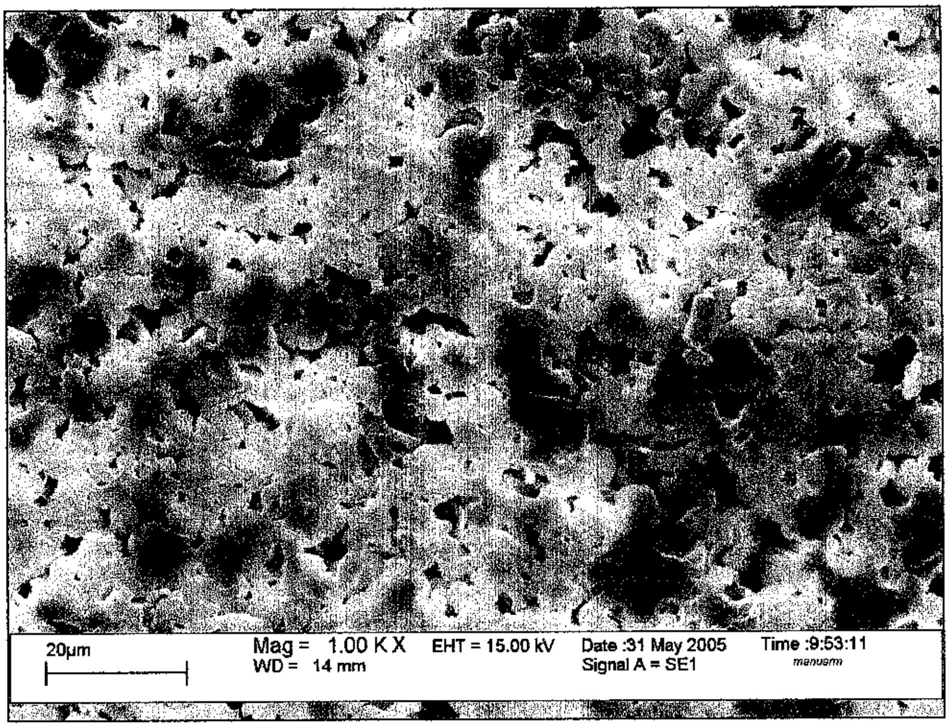


Figure 10 : 15wt% PA-6 content dried at 100°C.

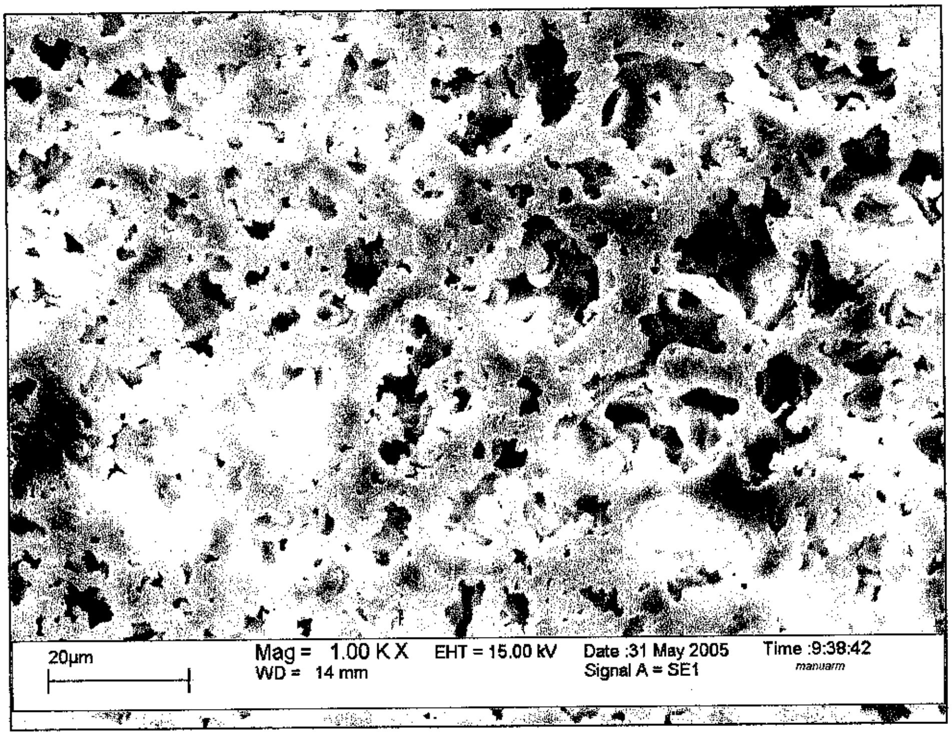


Figure 11 : 10wt% PA-6 content dried at 100°C.

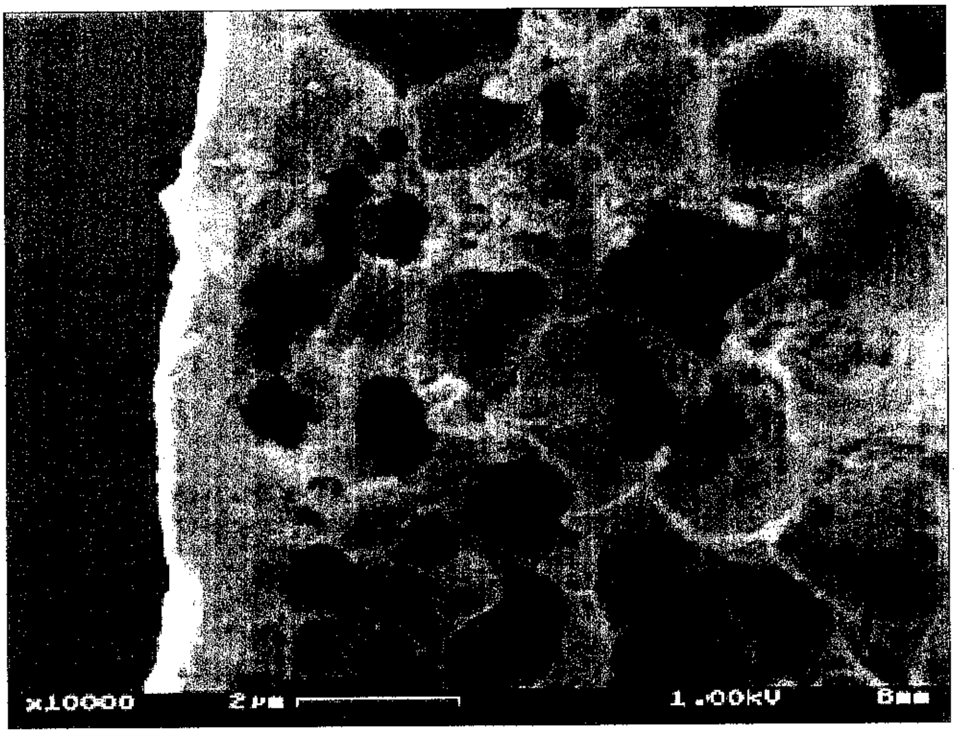


Figure 12 : Cross-section view of membrane.