CHARACTERISATION OF GLASS BASED ON PHYSICAL PROPERTIES AND SCANNING ELECTRON MICROSCOPE COUPLED WITH ENERGY DISPERSIVE X-RAY SPECTROSCOPY

by

RADZIAH BINTI NORDIN

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LIST OF ABBREVIATION

Lab	:	Laboratory
MC1	:	Measuring Cylinder 1
MC2	:	Measuring Cylinder 2
MC3	:	Measuring Cylinder 3
MC4	:	Measuring Cylinder 4
BK1	:	Beaker 1
BK2	:	Beaker 2
BK3	:	Beaker 3
EF1	:	Erlenmeyer Flask 1
EF2	:	Erlenmeyer Flask 2
BG	:	Bottle Glass
CG	:	Container Glass
DG	:	Drinking Glass
SEM/EDX	:	Scanning Electron Microscope coupled with Energy Dispersive X-ray Spectroscopy
GRIM 3	:	Glass Refractive Index Measurement 3
MT	:	Match Temperature
CT	:	Cooling Temperature
HT	:	Heating Temperature
e.g	:	Example
Si	:	Silica
Ca	:	Calcium
Mg	:	Magnesium
K	:	Potassium
Na	:	Sodium

C	:	Carbon
O	:	Oxide
B	:	Boron
Al	:	Aluminium

LIST OF SYMBOL

%	:	Percentage
°C	:	Degree Celsius
°F	:	Degree Fahrenheit
gcm^{-3}	:	Gram per cubic centimetre
mm	:	Millimetre
µm	:	Micrometre
mL	:	Milliliter
g	:	Gram

ABSTRACT

Glass is one of the important physical evidence in forensic science as it can link the suspect or assailant with the victims and crime science. It is frequently encountered at crime scenes, particularly those involving motor vehicle accidents or burglaries. This study emphasized on the characterization of two difference types of glass, laboratory and household glassware based on physical properties (thickness, density and refractive index) and elemental composition by SEM-EDX. Thickness analysis was divided into three parts, upper, middle and base. The thickness of upper part for laboratory glassware was found in the range of 1.41 mm - 2.02 mm while for household glassware was in the range from 3.06 mm - 4.09 mm. Next, the range for middle part of laboratory glassware was 2.88 mm - 3.48 mm while household between 1.41 mm - 2.02 mm. Then, the thickness for base part of laboratory glassware was in the range between 0.9 mm - 4.16 mm while for household glassware in the range 4.22 mm - 5.06 mm. The density of laboratory glassware also different with the household glassware as lab glass has density 2.2 gcm^{-3} while household was 2.4 gcm^{-3} . Other than that, the refractive index for the laboratory glassware showed the range between 1.472 - 1.506 while for the household glassware gave the refractive index from 1.515 - 1.522. Next, for the elemental composition detection by using SEM-EDX has identified all expected elements (Si, Ca, Mg, Na, C, O, Al, K) except for boron (B).

ABSTRAK

Kaca adalah salah satu bukti fizikal yang penting dalam sains forensik sebagaimana ianya boleh dikaitkan antara suspek, mangsa dan tempat kejadian. Kebiasaannya di tempat kejadian, bukti ini dikaitkan dengan kes-kes kemalangan dan rompakan. Kajian ini adalah berkenaan dengan pencirian dua kaca yang berbeza jenis iaitu kaca makmal dan kaca dari barangan rumah berdasarkan ciri-ciri fizikal (ketebalan, ketumpatan, dan refraktif indeks) dan juga komposisi elemen menggunakan SEM-EDX. Analisis ketebalan terbahagi kepada tiga bahagian iaitu atas, tengah dan tapak. Kajian menunjukkan, ketebalan pada bahagian atas untuk kaca makmal adalah dalam lingkungan 1.41 mm - 2.02 mm manakala untuk kaca barangan rumah pula mempunyai ketebalan dalam lingkungan 3.06 mm - 4.09 mm. Selain itu, ketebalan untuk bahagian tengah pada kaca makmal adalah 2.88 mm - 3.48 mm manakala untuk kaca barangan rumah ialah 1.41 mm -2.02 mm. Ketebalan tapak untuk kaca makmal pula dalam lingkungan 0.9 mm – 4.16 mm manakala untuk kaca barangan rumah pula adalah 4.22 mm - 5.06 mm. Ketumpatan bagi kaca makmal juga berbeza dengan kaca barangan rumah dengan 2.2 gcm^{-3} dan 2.4 gcm^{-3} bagi kedua-duanya. Selain daripada itu, refraktif index untuk kaca makmal menunjukan perbezaan dan mepunyai lingkungan 1.472 - 1.506 manakala untuk kaca barangan rumah mempunyai refraktif indeks 1.515 - 1.522. Selain itu, untuk komposisi elemen dikesan menggunakan SEM-EDX telah mengidentifikasi kesemua elemen yang dijangkakan (Si, Ca, Mg, Na, C, O, Al, K) kecuali utnuk boron (B).

CHAPTER 1

INTRODUCTION

1.1 Background of study

Glass is one of the trace evidence which is significantly useful in forensic science to solve any cases involving crime. Trace evidence is a very small piece of evidence left at a crime scene that may be used to identify or link a suspect to a crime. Glass for an example is trace evidence that can be found in most places and has wide variety of forms and compositions, which differentiates the properties of their material. At the crime scene, the recovered glass might be very helpful in determination of the types of glass, pattern of the glass fracture, classification of glass according to the physical characteristic and also individualize the glass to a source.

There are a few studies which have covered and analysed the general type of glass, for an example window glass originated from house or vehicles. Glass from window is significant for the cases related to house break-in or accidents. The transfer of glass fragments from the original object to the other object may give a significant value as different type of glass would give different physical properties and element composition. Therefore it is very important to know the physical properties and element composition contains in the glass to help in complete the investigations.

Glass can be differentiated according to their raw material. Soda lime and borosilicate glass are the examples of the types of glass which are different in their raw material. Soda lime glass has been used in manufacturing of the household glass such as windows, glass containers, drinking glass and many others. Borosilicate glass is a type of glass which usually found in laboratory glassware, cooking equipment (pyrex) and other heat resistance glass. Many researchers have analysed soda lime glass instead of borosilicate glass. As it has more potential to be encountered in forensic investigation compared to borosilicate glass. However, borosilicate glass also has its own significant value that can be applied in forensic investigation.

One of the important properties of borosilicate glass is it can strongly withstand at a very high temperature. Therefore it is suitable to be used as laboratory glassware. Unfortunately, it is also utilize it for illegal drug manufacturing in clandestine laboratory. The discovery of borosilicate glass fragment at the suspected clandestine laboratory may give a sign of illegal drug manufacturing activities.

Basically, the clandestine drug laboratory or clan lab is a mini-chemical lab designed for one purpose, to make deadly, illegal drugs quickly and cheaply. According to Bureau of Narcotic Enforcement (BNE, 2014), the signs of illegally drug manufacturing in clan lab is strong or unusual chemical odours, fortifications on houses or outbuildings, such as heavily barred windows or doors, chemical cans or drums in the front or back yard (these containers often have the labels marked or painted over), automobile or foot traffic at all hours, people going outside the building only long enough to smoke, especially at motels or during bad weather, new high fences with no visible livestock or animals and laboratory equipment such glass tubes, beakers, bunsen burners, funnels, conical flask and measuring cylinder. Therefore, one should know the types of glass that usually found at clan lab as it will help authorities to get some clues about the illegal drug manufacturing activities.

1.2 Problem Statement

Glass differ according to the raw material which is element composition of the glass and physical properties of the glass itself. The problem arises when the suspicious glass found is broken into a tiny fragment and it is hard to determine the original sources of the glass. It is important especially when there is no control sample found at the crime scene for the comparison process (Zadora, 2001). Therefore knowledge on the type of glass may help forensic scientist in forensic glass investigation to know either the glass is originated from the household or laboratory equipment as it contribute to narrow down the information to seize the clan lab.

1.3 Objectives of the Study

1.3.1 General Objective

The general objective of this study is to characterize the glass based on physical properties and Scanning Electron Microscope coupled with Energy Dispersive X-ray Spectroscopy (SEM/EDX)

1.3.2 Specific Objectives

The specific objective of this study are:

i. To examine the similarities and different physical properties (thickness, density, refractive index) of the laboratory and household glassware
ii. To determine the element composition of the laboratory and household glassware using Scanning Electron Microscope/Energy Dispersive X-Ray Spectroscopy (SEM/EDX).

1.4 Significance of the Study

Different types of glass possess different qualities depending upon their raw material, chemical makeup and how they have been produced. Choosing the right type of glass for a particular application also means understanding the different physical properties each different type of glass possesses. Borosilicate glass is a type of glass which contributes a major constituent in making laboratory glass. The nature of the borosilicate glass such as heat and chemical resistant make it suitable for laboratory glassware. Moreover, laboratory glassware is usually more preferable for the illegal drug maker as it can withstand in high temperature to process the drugs. However, this type of glass may crack if it exposed to the extreme hot and sudden cold temperature in the same time and the glass fragment will be disposed. As mentioned before, one of the sign of clan lab is the discovery of the laboratory glass outside or inside the suspected area. The problem arises if the laboratory glass is not in the original form and has been fragmented into fragments. Thus, this study is embarked to assist the police or forensic investigators to characterize the fragmented laboratory glassware based on physical properties and elemental composition to relate the presence of the activities of illegal drug manufacturing.

CHAPTER 2

LITERATURE REVIEW

2.1 History of Glass

Obsidian, black volcanic glass was the very first glass known to stone age people which was used for making weapons and decortaive objects. The earliest known man made glass are dated back to around 3500 Before Century (BC), with finds in Egypt and Eastern Mesopotamia. Glass appears to have been produced as far back as 1500 BC by the Egyptians and perhaps the Phoenicians. Glass uses and manufacturing developments have gone through an interesting evolution throughout human history, influenced by many cultures including those in Africa, China and Europe (IFRI, 2008).

Basically, the manufacturing of the glass had far improved with the introduction of oven technology by the romans. After that, the Germans in the 19^{th} century manufactured optical glass and heat-resistant glass for use in thermometers and cooking glassware (Koons, 2002). Glass production ranges from simple glass containers to advanced micro-components which usually involved five steps to manufacture. It usually involving the preparation of raw materials, melting, forming, annealing and cooling also warehouse or secondary processing (IFRI, 2008).

2.2 Glass

Glass is a substance with innumerable uses as well as some qualities that are unique. In the context of forensic science, glass can be fragmented into a tiny form also can be easily transferred to other things such as hair, skin and also weapon. Therefore it can act as trace evidence which could lead in finding their original sources. Apparently solid, it is in fact a liquid, cooled well below its point of solidification and held between two highly stretched sheets, a fact that accounts for both its transparency and its tendency to shatter into tiny fragments (Walt, 2005). Glass is defined as an "inorganic production of fusion that has been cooled to a rigid condition without crystallization" (ASTM, 2000). In the other word, this material is composed of a mixture of inorganic materials made by melting sand, lime, or called calcium oxide (CaO) and sodium oxide (Na_2O) at a very high temperature, and then cooled to a rigid condition without crystallisation and will produce a glass (IFRI, 2008).

The main elements of the glass is silicon dioxide (SiO_2). Generally, silicon dioxide is a white or colourless crystalline compound found mainly as quartz, sand, flint, and many other minerals (Anon, 2015). Other than silicon dioxide, glass contains several element compositions such as sodium oxide, aluminium oxide, boron oxide or potassium oxide. Some elements are added in glass manufacturing is because to increase the toughness, resistant towards heat, and also give colour to the glass (Walt, 2005).

2.3 Types of Glass

Currently, there are many types of glasses based on the ones needs. Glass can be classified according to their intended use and main raw materials (IFRI, 2008). The different types of glass according to their intended use are flat glass, containers, glass fibres and specialty glass. The type of glass is based on the main raw materials are soda lime, leaded glass, borosilicate glass, and special glass.

2.3.1 Soda Lime Glass

Soda-lime glass, also called soda-lime-silica glass, is the most general type of glass used in daily life. The main raw materials utilized for the manufacture of soda lime glasses are approximately 70-72% of silica (SiO_2), 15% of soda (Na_2CO_3) and 9% of lime (CaO) (Anon., 2016). Basically addition of soda to silica has result a large decrease in the temperature required for melting (Shelby, 2005). Unfortunately, large amount of soda also lead to poor chemical durability of the glass (Shelby, 2005).

The present of soda in these glasses result in major change in physical properties of the glass. It reduces the electrical resistivity of the glass (Shelby, 2005). The other property of soda lime glass is refractive index. Usually, refractive index for soda lime glass approximately about (1.51-1.52) while the density of this type of glass is about $2.5gcm^{-3}$ (Shelby, 2005). As soda lime glass contains the main raw elements in glass-making, it can be further specialised for windowpanes (flat glass), glass bottles (container glass) and beverage jars, food, and some commodity items.

2.3.1.1 Flat Glass

Today, flat glass is comes in many highly specialised forms intended for different products and applications Flat glass is the basic material that goes into end products that we see every day. It is utilised to make windscreens and windows for automobiles and transport, windows and facades for houses and buildings, as well as solar energy equipment like solar thermal panels and photovoltaic modules (Anon., 2010). It is also encountered, in much smaller quantities, for many other applications like interior fittings and decoration, furniture, "street furniture" (bus stops), appliances and electronics (Anon., 2010).

Flat glass is produced by way of the float process is often further processed to give it certain qualities or specificities (Anon., 2010). The float process, originally able to make only 6mm thick glass, now makes it as thin as 0.4 mm and as thick as 25 mm. Molten glass, at approximately 1000°C, is poured continuously from a furnace onto a shallow bath of molten tin. It floats on the tin, spreads out and forms a level surface. Thickness is controlled by the speed at which solidifying glass ribbon is drawn off from the bath. After annealing (controlled cooling) the glass emerges as a 'fire' polished product with virtually parallel surfaces.

2.3.1.2 Container Glass

Container glass is a type of glass for the production of glass containers such as bottles, jars and bowls. It has been manufactured by automatic process which includes two methods which are The Blow and Blow method also The Press and Blow method (Anon.. 2013). The Blow and Blow method starts when molten 'gobs' of glass are delivered into a mould known as a 'blank' or parison mould. A puff of compressed air blows the glass down into the base of the mould to form the neck or 'finish' part of the bottle or jar.

A second blast of compressed air is then applied through the already formed neck of the container to form the 'parison' of pre-form for the bottle against the walls of the parison mould cavity. The thick walled parison is then transferred to the final mould during which time the surface of the glass 'reheats' and softens again enough to allow the final container shape to be fully formed against the walls of the final mould cavity by the application of either compressed air or vacuum. The container is then removed and transferred to an annealing oven (lehr) where it is reheated to remove the stresses produced during forming and then cooled under carefully controlled conditions. A second method is The Press and Blow Method. It happens as molten 'gobs' of glass are delivered into the parison mould and a plunge is used to press the glass into the parison shape (Anon., 2013). The final mould stage of the process is the same as that described for the Blow and Blow Process.

2.3.2 Glass Fibres

A glass fiber can be defined as a material consisting of extremely fine filaments of glass that are combined in yarn and woven into fabrics, used in masses as a thermal and acoustical insulator, or embedded in various resins to make boat hulls, fishing rods, and the like (Imtiaz, 2014). Glass fibers are made from silica dioxide (SiO_2), which melts at 1720°C/3128°F (Gardiner, 2009). SiO_2 is also the basic element in quartz, a naturally occurring rock. Quartz, however, is crystalline (rigid, highly ordered atomic structure) and is 99% or more SiO_2. If SiO_2 is heated above 1200°C/2192°F then cooled ambiently, it crystallizes and becomes quartz.

There are two basic types of glass fiber products, textile and wool and both are manufactured by similar processes (Schorr, 1995). The fibre manufacturing process has effectively two variants. One involves the preparation of marbles, which are remelted in the fiberisation stage. Second, use the direct melting route in which a furnace is continuously charged with raw materials which are melted and refined as that glass reaches the forehearth above a set of platinum–rhodium bushings from which the fibres are drawn. Both variants involved five steps which are batching, melting, fiberization, coating and drying or packaging and afterward they are ready to be palletized and shipped or further processed into chopped fiber, roving or yarn (Imtiaz, 2014).

2.3.3 Borosilicate Glass

Borosilicate glass is the material of choice for a wide range of applications, from cookware to laboratory. Typically referred to by one of its brand names, such as Pyrex or Kimax, borosilicate glass consists of a silica, boric oxide, calcium oxide, sodium oxide and potassium oxide mixture, which varies slightly depending on the particular brand in question (Anon., 2016). Pyrex is the most common type of borosilicate glass and is produced by Corning Glass Works. The main distinction of borosilicate glass from traditional glass is the substitution of boron oxide for soda and lime in the manufacturing process. Borosilicate glass must contain at least 5% boron oxide, which helps bind the silicate and aluminium oxide and sodium oxide (Gardiner, 2009).

Borosilicate glass is perfect for scientific and medical laboratory use, since it offers excellent chemical resistance in addition to its other useful qualities. Everything from tubes, rods and beakers to test tubes, graduated cylinders, pipettes and stopper attachments are produced from borosilicate. Although borosilicate glass offers exceptional acid resistance, it is less resistant to a range of alkalis, and occasionally other materials should be considered. Borosilicate glass is also utilised in certain optics, such as mirrors, because it retains shape well throughout changes in temperature. Other uses include the strengthening of various plastic compounds and in various gages and protective glass surfaces (Anon., 2016).

Creating borosilicate glass requires higher temperatures than those necessary for the production of regular glass, although this also accounts for its higher heat resistance. It also faces far less material stress than regular glass due to its lower thermal expansion coefficient, which also adds to its exceptional performance at high temperatures. Additionally, borosilicate glass is far more durable than traditional glass, and can withstand accidents that would break other glassware. Even when it does crack, it typically performs better, as it rarely shatters (Anon., 2016).

There are three classes of materials are preferred in making borosilicate glass including formers, fluxes, and stabilizers (IFRI, 2008). Formers are the main ingredients in all glassmaking. These are crystalline materials that, when heated high enough, can be melted and cooled to create glass. The primary formers used for making borosilicate glass include silica sand and boric acid (IFRI, 2008). Fluxes are compounds that help lower the temperature required to get the formers to melt. Stabilizers are materials that help keep glass from crumbling, breaking, or falling apart. They are needed because fluxes typically destabilize glass compositions (IFRI, 2008).

2.4 Physical Properties of Glass

Glass has variety of physical properties which gives information about the type classification of glass and also the physical fit of the glass fracture. The measurement of a physical property may change the arrangement of matter in a sample, but not the structure of its molecules. In other words, a physical property may involve a physical change, but not a chemical change. The physical properties of glass can be divided into two which are optical property and non-optical property. The example of optical property is colour and refractive index while the non-optical property includes thickness, surface features, curvature, hardness and density.

2.4.1 Thickness

ASTM International (ASTM), formerly known as the American Society for Testing Materials, Standard C1036-06 lists the tolerances for standard thicknesses of flat glass. The tolerances for flat glass in the thickness ranges typically encountered in forensic casework are on the order of ± 0.25 mm (ASTM C1036-06), but the range observed in casework for a pane of flat glass is usually much lower (Koons *et al.*, 2002). Thickness is usually tightly controlled by the manufacturer, on the order of thousandths of an inch in a single sheet. Variations in thickness can produce unsightly ripples in the glass sheet, lessening the market value of the glass. Thickness is a function of the viscosity of the glass. Viscosity can be changed only by changing the glass composition or the furnace temperature (Tooley 1984; Varshneya 1994). Changing the glass composition can produce other, even less desirable, changes in the end product, and increasing the furnace temperature is expensive because of increased fuel costs (Tooley 1984). Thickness in the forensic laboratory can be measured using a micrometer or caliper but requires that fragments possess both original surfaces. Because thickness produces a quantitative measure, the precision and accuracy of the micrometer must be considered when evaluating the results of thickness measurements. When the thickness of a piece of flat glass is measurably different from the range expressed in the known glass standard, those glasses can be determined to have come from different sources.

2.4.2 Surface features

Certain surface features are imparted during glass manufacturing and fabricating processes or during use. These features can include mold and polish marks, mirrored backings, scratches, and decorative finishes such as texturing, etching or frosting, and coatings. The majority of these features can be compared visually using a stereomicroscope, but coatings are not usually apparent to the naked eye and may require sophisticated instrumentation for detection and comparison. Transmission electron microscopy (Bravman and Sinclair 1984), X-ray scattering (Misture, 1999), atomic force microscopy (Arribart and Abriou 1999), and Fourier transform infrared spectroscopy (DeRosa and Condrate 1999) have all been employed for the analysis of glass coatings. These features also can be utilised to discriminate between glass objects.

2.4.3 Curvature

Whether or not a fragment of glass is flat or curved often can be determined visually with the aid of low-power magnification. For small particles, interferometry can be used (Locke 1984). The curvature of glass can be used as a point of comparison and as a method of determining a broad product type.

2.4.4 Refractive Index

Refractive index is a unitless measure of the speed of light in a transparent medium and is defined by Snell's law as the ratio of the velocity of light in a vacuum to the velocity of the wave in the transparent medium (Stoiber and Morse 1981). Refractive index (RI) is a function of chemical composition and atomic arrangement (Stoiber and Morse 1981). In glass, these are controlled by the composition of the batch (chemical composition) and the cooling history of the glass (atomic arrangement) (Varshneya 1994). Tempering changes the cooling rate of the surface of the glass relative to the interior and imparts a continuous change in the RI from the surface to the center of the glass (Varshneya 1994).

Refractive index is the most commonly measured property in the forensic examination of glass fragments because precise refractive indices can be measured rapidly on the small fragments typically found in casework. Other than that, it can aid in the characterization of glass and provides good discrimination potential. (Koons *et al.* 2002). RI varies with wavelength of light and temperature (Bloss, 1961). Dispersion is the change in RI with a change in wavelength of illumination. For glass, relative dispersion, or dispersive power, is used to quantify dispersion (Koons *et al.* 2002). The precision refractometer measures indices only at the surface of the glass (Skoog and West, 1980). A V-block refractometer can measure only the average refractive index through a block of glass. Although both methods when applied appropriately are accurate to six decimal places, they require the use of large polished glass samples. Samples adequately large for these methods are rarely encountered in forensic casework (Koons *et al.*, 2002).

2.4.4.1 Immersion Method

Immersion methods also known as Becke Line method is selected to measure RI in some laboratories. These methods take advantage of the fact that when using monochromatic light, a particle immersed in a liquid of identical refractive index will become invisible (Bloss, 1961). In the Becke line method, a bright halo which represent of Becke line is observed around the particle. Movement of the Becke line with respect to the particle on changing the microscope focus indicates refractive index of the particle relative to the immersion oil.

Basically, the method depends upon the fact that, under the conditions to be described, if the immersion liquid has a refractive index considerably higher than that of the glass, the glass will appear amber, golden, or "beer bottle brown" (Davis, 1956). If the liquid has an RI considerably lower than the glass, the specimen will appear outlined with a "cold" blue or blackish blue line (Davis, 1956). The colour will appear as stated if the refractive index of the liquid and glass are within 0.1 to 0.05 of each other, depending upon dispersion ratios (Davis, 1956). The amount of contrast between the particle and the immersion liquid indicates the magnitude of the difference in refractive index. The fragment is then removed from the liquid, washed, and placed in another liquid with a refractive index closer to the match point. This process is repeated until the refractive index of the match point has either been reached or bracketed by two oils. When the match point is approached, the results can be plotted on Hartmann dispersion nets, which allows for the extrapolation of the results between liquids (SWGMAT, 2005).

2.4.4.2 Automated Glass Refractive Index Measurement (GRIM)

An automatic instrument for Glass Refractive Index Measurement (GRIM), developed by Foster and Freeman Ltd., has been evaluated and adapted to meet the requirements of the UK Forensic Science Service. Basically, GRIM consists of a microscope, a TV camera, a hot stage and a computer serving to direct and register the measured signal. Measurement automation allows determination of the RI value of the glass sample with accuracy up to10^{-4} (ZADORA, 2001). **Table 2.1** shows data of index of refraction ranges for several types of glass measured by using GRIM 3 (Saferstein, 2007).

Generally, a glass particle (either whole or crushed) is mounted in a silicone oil (A,B, or C type) and is observed microscopically as the temperature of the hot-stage is varied. The temperature at which the glass becomes invisible is the temperature at which the glass and oil have the same RI and is usually referred to as the 'match temperature' (MT) (ZADORA, 2001). Accurate determination of MT is carried out by determination of the so-called heating temperature (HT) and cooling temperature (CT) (ZADORA, 2001). The heating temperature, HT, is the temperature at which the edge of a micro trace immersed in immersion oil vanishes during heating of the sample (ZADORA, 2001). After its determination the sample is heated further until the moment of re-appearance of the observed edge. Then the analysed sample is cooled down until it reaches a temperature at which the micro trace edge vanishes again. This is the cooling temperature, CT. The matching temperature, MT, is calculated as the mean value of HT and CT.

Table 2.1 Index of Refraction ranges for several types of glasses

Glass	Index of Refraction
Headlight glass	1.47-1.49
Television glass	1.49-1.51
Window glass	1.51-1.52
Bottles	1.51-1.52
Ophthalmic lenses	1.52-.153

2.4.5 Density

Density is mass per unit volume represented by the units of g/cm^3. Like refractive index, density is a function of chemical composition and atomic arrangement, which are controlled by the composition of the batch and the cooling history of the glass, respectively (Varshneya, 1994). The typical density variation measured in a bottle is 0.002 g/cm^3, and the typical density variation measured across a sheet of glass is 0.001 g/cm^3 (Koons, 2002). Density in glass can be assessed either quantitatively by direct measurement or qualitatively by simultaneous comparison of two or more specimens. Basically there are few standard test methods in practices in order to obtain the density of glass. There are Density gradient column method and sink/float method.

2.4.5.1 Density Gradient Column Method

The method involves placing, in a vertical glass tube, a liquid containing a gradient of density (Kirk, 1951). The gradient is such that the density at any level is less than that at any level lower in the tube and greater than that of any level higher in the tube (Kirk, 1951). When glass fragments are introduced to the column, each will become suspended in the liquid at the level that is the same density as that glass fragment (Kirk, 1951). Fragments of different density will settle at different levels in the column.

2.4.5.2 Sink/Float Method

The method involves suspending glass fragments in a density solution within a constant temperature bath (Koons *et a.,* 2002). If two or more fragments are suspended, their densities are the same. The numeric density value is not determined in this method but can be determined using the absolute density determination methods. After glass fragments are suspended in a density solution using the sink/float method, a numeric determination can be made using the Analytical balance and plummet method.

The method involves measuring the suspending density solution using an analytical balance and weighed plummet (Koons *et al.,* 2002). An analytical balance capable of accurately determining weights within 0.0001 g with a tall weighing chamber and a plummet are required. The plummet must sink in a liquid with a density above 2.6 g/cm^3 (Beverage and Semen 1979). Analyse a reference glass sample of known density prior to each use to ensure that the system is operating within acceptable parameters. Determine the volume of the plummet by weighing it in air and then in water at a known temperature. Place the glass fragment in the density column and suspend it using the density liquids. Weigh the plummet immersed in the density column. Calculate the density of the liquid and the glass (Koons *et al.*, 2002).

Other numerical method to obtain the density of glass is by density meter method (Beveridge and Semen 1979). The method involves measuring the density of a suspending liquid mixture using a density meter. A density meter with its sample chamber at the same constant temperature as the density column is required (to +/- 0.1 degrees Celsius). Analyse a reference glass sample of known density prior to each use to ensure that the instrument is operating within acceptable parameters. A portion of the suspending density liquid mixture is transferred into a calibrated density meter and the density is recorded.

2.5 Elemental Composition by SEM/EDX

According to Grzegorz Zadora (2001), refractive index value alone for an analysed glass sample constitutes insufficient information to classify it into a definite glass-use group. Since RI can furnish only a limited amount of information concerning the type or class of glass of an unknown sample, various methods of chemical analysis have been employed for the forensic examination of glass samples. At present, the elemental content of glass is determined most often using such analytical methods as Scanning Electron Microscope with Energy Dispersive X-ray Spectrometry (SEM-EDX) or Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) (Zadora, 2001).

According to of Zadora and Brożek-Mucha (2003), there are two important aims in forensic investigation. The first aim is comparison (discrimination) of the evidence with the reference material and the second aim is when there is no comparative material available. When there is no comparative material, the classification of the evidence sample into a group of objects taking into account its specific chemical and physical properties (Zadora and Brożek-Mucha, 2003). In the other word, classification and discrimination are different as the determination of different kinds of glass is usually based on the major elements, whereas discrimination is mostly based on minor or trace elements (Kurzweil, 2008). For glass composition, SEM-EDX has provided information about major and minor elements, such as Oxide (O), Sodium (Na), Aluminium (Al), Magnesium (Mg), Silica (Si), Potassium (K), Calcium (Ca), Ferum (Fe), from any glass fragment. (Zadora, 2009).

Generally, Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM–EDX) is a powerful tool for forensic scientists to classify and discriminate evidence material because it can simultaneously examine the morphology and the elemental composition of objects. SEM is a "surface" rather than a "bulk" technique that allow the chemical characterisation of minor and mayor elements (IFRI, 2008). The size of the interaction volume varies with sample and accelerating voltage (IFRI, 2008). Typical penetration depths are between 2- 5 μm or < 1 μm3 (IFRI, 2008). It can produce extremely high magnification images (up to 200000x) at high resolution up to 2 nm combined with the ability to generate localised chemical information EDX (IFRI, 2008). This means the SEM/EDX instrument is a powerful and flexible tool for solving a wide range of product and processing problems for a diverse range of metals and materials.

CHAPTER 3

METHODOLOGY

3.1 Reagents

The reagents preferred are listed in **Table 3.1**.

Table 3.1 List of reagents

No.	Chemical	Brands
1	Bromoform	MERCK
2	Bromobenzene	MERCK
3	Silicon Oil type C	-
4	Ethyl Alcohol	-

3.2 Apparatus

List of apparatus that have been used are tabulated in **Table 3.2**

Table 3.2 List of apparatus employed

No	Apparatus	Brand
1	Measuring cylinder (25mL ± 0.4mL)	Ihmbg
2	Test tube (20mL)	-
3	Reagent bottle (250ml)	-
4	Microscope glass slide and Cover slip	Leica
5	Pestle	-

3.3 Material

Table 3.3 shows the disposable material that is frequently used in this work

Table 3.3 List of disposable materials

No	Material	Brands
1	Tissue Papers	Scott
2	Gloves	
3	Goggle	-
4	Label sticker	-
5	Plastic packaging with seal	-

3.4 Equipment

Table 3.4 has displayed a few of equipment which has been used in this work. The photo in **Figure 3.1** shows a Glass Refractive Index Measurement 3 (GRIM 3) while the photo in **Figure 3.2** is the SEM-EDX.

Table 3.4 List of equipments

No	Name of Equipment	Brand
1	Glass Refractive Index Measurement System 3	Foster + Freeman
2	Leica Phase Contrast Microscope	Leica
3	Mettler Toledo FT82HT Hot Stage	Foster + Freeman
4	Video camera system	Foster + Freeman
5	Processing unit for match-point detection	Foster + Freeman
6	Electronic Digital Caliper	-
7	SEM-EDX	ZEISS

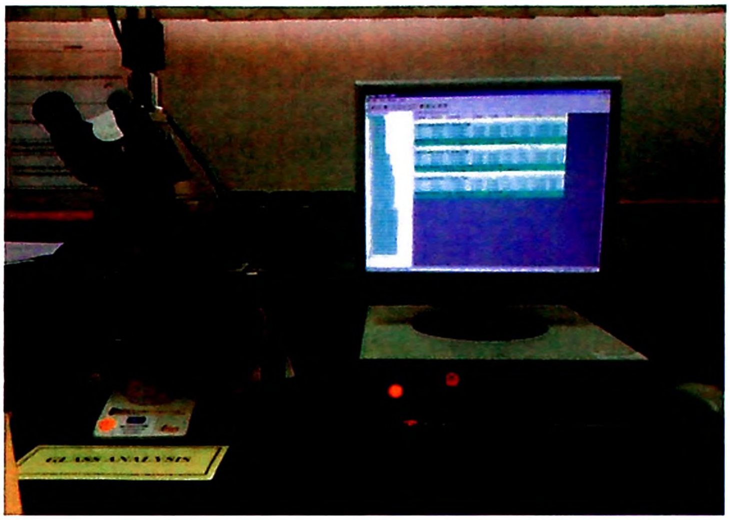

Figure 3.1 Picture of.Glass Refractive Index Measurement 3

Figure 3.2 Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy