

**DEVELOPMENT AND CHARACTERIZATION OF BISPHENOL A FOR MAKING
POLYCARBONATE PLASTICS AND EPOXY RESINS**

By

USMAN OGIRIMAH AWWAL

(2006/24214EH)

**DEPARTMENT OF CHEMICAL ENGINEERING
FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA
NIGERIA**

November 2011

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**A PROJECT SUBMITTED TO THE
DEPARTMENT OF CHEMICAL ENGINEERING,
FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA
NIGERIA.**

**IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE AWARD OF
BACHELOR IN ENGINEERING (B.ENG) DEGREE IN CHEMICAL
ENGINEERING**

November, 2011

DECLARATION

I declare that the work in the project report entitled "Development and characterization of Bisphenol A for making polycarbonate plastics and epoxy resins" has been carried out by me under the supervision of Dr. M. O. Edoga. No part of this project report was presented for another degree or diploma elsewhere at any institution to the best of my knowledge.

Usman.Ogirimah Awwal

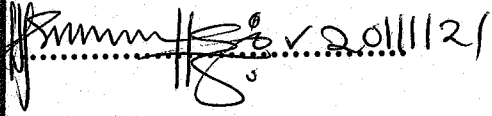
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CERTIFICATION

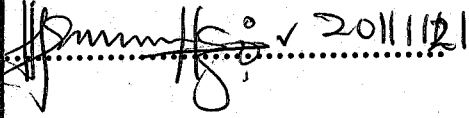
This is to certify that this project report entitled 'DEVELOPMENT AND CHARACTERIZATION OF BISPHENOL A FOR MAKING POLYCARBONATE PLASTICS AND EPOXY RESINS' by USMAN OGIRIMAH AWWAL meets the requirements for the partial fulfilment of the award of Bachelor of Engineering (B.Eng) degree in Chemical Engineering, Federal University Of Technology, Minna.

 20/11/21

21-11-11

Dr. M. O. Edoga
(Project Supervisor)

Date

 20/11/21

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Dr. M. O. Edoga
(Head of Department)

Date

.....

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External Supervisor

Date

DEDICATION

dedicate this work to Almighty Allah for making me start and complete this project in good health and to my family for their support.

ACKNOWLEDGEMENT

I give thanks to almighty Allah the most beneficent, the most merciful, for his mercy and favour upon me throughout this programme. My sincere gratitude to my supervisor in person Dr. M. O. Edoga for his commitments, devotion and encouragement towards the completion of this work.

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I would like to make my deepest appreciation to my parents, Mr and Mrs Usman, for their support, prayers and encouragement. They are the source of my financial strength. In short, they are the best. May Almighty Allah give you both long life to eat the fruit of your labour amin.

I cannot also forget at this time the supports and care of my siblings Usman Jemilah, Usman Ashraf and Usman .A. Eneye. Enizle, and my cousin, Sezu Salami for their encouragement and prayers. They never give up on me. They are the engine that burns the fuel zeal and strength to go on in my system.

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ABSTRACT

This research project is aimed at the production of Bisphenol A which is used in the production of epoxy and poly carbonates. Bisphenol A is produced by reacting one volume of acetone and three volume of phenol in the presence of an acid catalyst. The mixture is heated to a temperature of about 60 °C in a round bottom flask for an hour. The resulting product is a two layer liquid of Bisphenol A and unreacted phenol and the acid catalyst (HCL). The resulting Bisphenol A was characterized, the solubility in water, flash point boiling point relative density and percentage purity were carried out and the values obtained and compared with the standard values from literatures, it was found to be close to each other. The little disparity was as a result of the percentage purity of Bisphenol A produced which is 71 % as measured using an IR spectrophotometer. The objective of the research was achieved by the production and characterization of the Bisphenol A produced.

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Chapter One

1.0 INTRODUCTION

Most modern manufacturing industries in Nigeria and other developed countries employ the use of chemical compounds in their various production processes. Bisphenol A is an important industrial raw material which is widely used in companies engaged in the production of polycarbonate plastics and epoxy resins; which are further processed into different forms of plastics and coatings on the inside of almost all food and beverage cans. Hence, the importance of Bisphenol A cannot be over emphasized especially to developing countries like Nigeria.

Bisphenol A, abbreviated as BPA is synthesized through condensation of phenol and acetone in the presence of an acid catalyst such as HCl or sulphonated styrene DVB catalyst. BPA exists at room temperature as a white solid and has a mild "phenolic" or hospital odour. (Uglea *et al*, 1991)

BPA, being a chief source of polycarbonate plastics and epoxy resins, companies producing plastics, canned or bottled foods and drinks, baby bottles, eye glasses, medical and dental devices, CD lenses, adhesives etc finds it very important in their daily production processes. Polycarbonate plastics have many applications including use in certain food and drink packaging, e.g., water and infant bottles, compact discs, impact-resistant safety equipment, and medical devices. Polycarbonate plastics are typically clear and hard. Polycarbonate plastic can also be blended with other materials to create. Epoxy resins are used as lacquers to coat metal products such as food cans, bottle tops, and water supply pipes. The applications for epoxy-based materials are extensive and include coatings, adhesives and composite materials such as those using carbon fiber and fiberglass reinforcements, although polyester, vinyl ester, and other thermosetting resins are also used for glass-reinforced plastic (Heinz-Weiner, 2011). The chemistry of epoxies and the range of commercially available variations allow cure polymers to be produced with a very broad range of properties. In general, epoxies are known for their excellent adhesion, chemical and heat resistance, good-to-excellent mechanical properties and very good electrical insulating properties. Many properties of epoxies can be modified, for example silver-filled epoxies with good electrical conductivity are available, although epoxies

typically electrically insulating, variations offering high thermal insulation or thermal conductivity combined with high electrical resistance for electronics applications are available (Weiner - Weiner, 2011)

In spite of all these applications, the global economic recession that began in late 2008 and continued through 2009 significantly affected Bisphenol A demand. Polycarbonate and epoxy resin demand, closely tied to the overall health of the economy, declined in 2008 and decreased significantly in 2009 in the developed regions. Therefore, the overall health of the world economy will continue to play a major role in the future demand for Bisphenol A, as its derivatives' major end-use markets include automotive, construction and electrical/electronic applications.

In light of the fact above, there will arise more demands on the products of Bisphenol A i.e. polycarbonate plastics and epoxy resins which will also lead to more demands of Bisphenol A. This work is to develop or put forward schemes that will lead to more production of BPA as a product of the condensation reaction of phenol and acetone in the presence of an acid. This scheme suggested here is expected to result in increased productivity of Bisphenol A through efficient condensation reaction of phenol and acetone.

1.1 Aim and Objective

The aim of this work is to present a detail description of a process leading to the formation of Bisphenol A from the condensation of phenol and acetone in the presence of an acid catalyst. It is also aimed at characterizing the properties of the produced Bisphenol A which make it suitable for the production of Polycarbonates and Epoxy resins.

1.2 Scope of Work

The approach is a laboratory production and characterization of Bisphenol A with a consideration of its suitability for the production of Polycarbonates and Epoxy resins.

1.3 Economic Justification of the Work

Bisphenol A is a major component in the production of polycarbonate plastics and epoxy resins. Thus, the production of Bisphenol A will make it readily available for usage. Phenol and acetone are the major raw materials in its production and can be sourced from the Petrochemical industry.

Chapter Two

2.0 LITERATURE REVIEW

2.1 Background

Bisphenol A was first synthesized by Dianin in 1891 but saw little use until the 1930s when it was used as a synthetic estrogen product. Use of Bisphenol A slowed with the discovery of DES, a more potent artificial estrogen, later found to cause reproductive cancer in the children of mothers who took it. In 1930, the properties of BPA were investigated and it made its return in the 1950s as polycarbonate and epoxy resin; most commonly found in plastic bottles and the inside lining of cans. In the 1940s and 1950s, scientists discovered that Bisphenol A, when combined with the gas phosgene, helped create a clear, hard plastic called polycarbonate. This material was used in eyeglasses, baby bottles, shatter-resistant lights and many other applications (Fiege *et al*, 2002).

Due to the increasing popularity surrounding BPA containing products, a carcinogenesis study was done. In the late 1970s, by the National Cancer Institute (NCI) and National Toxicology Program (NTP) tested the safety of BPA. In 1998, Fred vom Saal and his research team published two studies, which investigated the effects of low-dose (below 50 micrograms/kilogram) BPA on mice and found changes in male reproductive organs, as well as, increased prostate weights (Helmut, 2002).

The primary source of exposure to BPA for most people is through the diet. While air, dust, and water are other possible sources of exposure, BPA in food and beverages accounts for the majority of daily human exposure. Bisphenol A can leach into food from the protective internal epoxy resin coatings of canned foods and from consumer products such as polycarbonate tableware, food storage containers, water bottles, and baby bottles. The degree to which BPA leaches from polycarbonate bottles into liquid may depend more on the temperature of the liquid or bottle, than the age of the container. BPA can also be found in breast milk (Heinz-Werner, 2011).

2.2 Production of Bisphenol A

A crystalline adduct of BPA and phenol having an APHA color of not greater than 15 and showing a pH of 4.9 - 5.5 when dissolved in an aqueous methanol solution having a pH of 5 in a concentration of 4 g per ml of water is used as a starting material for the production of BPA (USEPA, 2010).

The reaction of acetone with phenol for the production of BPA is generally performed in the presence of a strongly acidic substance, such as hydrochloric acid, sulfuric acid or a sulfonic acid type ion exchange resin, as a catalyst. These acidic substances, however, serve to facilitate the production of colored substances and impurities at an elevated temperature and thereby reduce the purity of the BPA product. Thus, after the completion of the BPA producing reaction, the acid catalyst is generally neutralized or removed from the reaction mixture so as to avoid the presence thereof in the succeeding phenol removing stage. For example, in a process in which hydrochloric acid is used as catalyst, the reaction mixture after the completion of the BPA producing reaction is heated to remove hydrogen chloride and is then neutralized with an alkali to a pH of 5-6. In a process in which a strong-acid type ion exchange resin is used as catalyst, the reaction mixture effluent is contacted with a weak base ion exchange resin to neutralize organic sulfonic acids produced by decomposition of the strong-acid type ion exchange resin during the BPA forming reaction. In the above cases, since an alkali adversely affects the purity and color characteristics of BPA and phenol, it is important that the amount of an alkali to be added to the BPA-containing reaction product should not exceed beyond the neutralization point. Thus, in the production of BPA by reaction of phenol with acetone in the presence of a strongly acidic substance, it is necessary, after the completion of the reaction, to remove the acidic substance or to neutralize the reaction with an alkali. In this case, when an excess alkali is added, it is also necessary to remove the alkali remaining in the reaction product. The removal of the acidic substance or the residual alkali from the reaction product can be performed in a subsequent crystallization step in which BPA produced is crystallised as an adduct with phenol (Toshikazu, 1999).

World production of this compound was 1 million tonnes in the 1980s, and more than 2.2 million tonnes in 2009/ in 2003, U.S. consumption was 856,000 tonnes 72 % of which was used to make polycarbonate plastic and 21 % going into epoxy resin.

2.2.1 Properties of Bisphenol A

The Table below shows the properties of Bisphenol A

Table 4.1: Properties of Bisphenol A

	Properties
Molecular formula	$C_{15}H_{16}O_2$
Molar mass	$228.29 \text{ g mol}^{-1}$
Appearance	White to light brown flakes
Melting point	158 to 159 $^{\circ}\text{C}$
Boiling point	220 $^{\circ}\text{C}$
Solubility in water	120-300 ppm (21.5 $^{\circ}\text{C}$)

2.2.2 Uses of Bisphenol A

1. Bisphenol A is used primarily to make plastics, and products containing bisphenol A-based plastics.
2. It is a key monomer in production of epoxy resins and in the most common form of polycarbonate plastic. Polycarbonate plastic, which is clear and nearly shatter-proof, is used to make a variety of common products including baby and water bottles, sports equipment, medical and dental devices, dental fillings and sealants, eyeglass lenses, CDs and DVDs, and household electronics.
3. BPA is also used in the synthesis of polysulfones and polyether ketones, as an antioxidant in some plasticizers, and as a polymerization inhibitor in PVC.

4. Epoxy resins containing bisphenol A are used as coatings on the inside of almost all food and beverage cans.
5. Bisphenol A is also a precursor to the flame retardant tetrabromobisphenol A, and was formerly used as a fungicide.
6. Bisphenol A is a preferred color developer in carbonless copy paper and thermal paper, with the most common public exposure coming from some thermal point of sale receipt paper.
7. BPA-based products are also used in foundry castings.

2.2.3 Environmental Impact of Bisphenol A

In general, studies have shown that BPA can affect growth, reproduction and development in aquatic organisms. Among freshwater organisms, fish appear to be the most sensitive species. Evidence of endocrine-related effects in fish, aquatic invertebrates, amphibians and reptiles has been reported at environmentally relevant exposure levels lower than those required for acute toxicity. There is a widespread variation in reported values for endocrine-related effects, but many fall in the range of 1 µg/L to 1 mg/L (Andrea, 2004).

BPA can contaminate the environment either directly or through degradation of products containing BPA, such as ocean-borne plastic trash.

As an environmental contaminant this compound interferes with nitrogen fixation at the roots of leguminous plants associated with the bacterial symbiont *Sinorhizobium meliloti*. Despite a half-life in the soil of only 1–10 days, its ubiquity makes it an important pollutant. According to Environment Canada, "initial assessment shows that at low levels, bisphenol A can harm fish and organisms over time. Studies also indicate that it can currently be found in municipal wastewater (USAid, 2009).

A 2009 review of the biological impacts of plasticizers on wildlife published by the Royal Society with a focus on annelids (both aquatic and terrestrial), molluscs, crustaceans, insects, fish and amphibians concluded that BPA have been shown to affect reproduction in all studied

animal groups, to impair development in crustaceans and amphibians and to induce genetic aberrations.

2.2.4 Health Effects

Bisphenol A is an endocrine disruptor, which can mimic the body's own hormones and may lead to negative health effects. Early development appears to be the period of greatest sensitivity to its effects. Regulatory bodies have determined safety levels for humans, but those safety levels are currently being questioned or under review as a result of new scientific studies (Heindel, 2009).

In 2007, a consensus statement by 38 experts on bisphenol A concluded that average levels in people are above those that cause harm to many animals in laboratory experiments. However, they noted that while BPA is not persistent in the environment or in humans, biomonitoring surveys indicate that exposure is continuous, which is problematic because acute animal exposure studies are used to estimate daily human exposure to BPA, and no studies that had examined BPA pharmacokinetics in animal models had followed continuous low level exposures. They added that measurement of BPA levels in serum and other body fluids suggests that either BPA intake is much higher than accounted for, or that BPA can bioaccumulate in some conditions such as pregnancy, or both. A 2011 study, the first to examine BPA in a continuous low level exposure throughout the day, did find an increased absorption and accumulation of BPA in the blood of mice (Parmigiani, 2011).

A 2007 review has concluded that BPA, like other xenoestrogens, should be considered as a player within the nervous system that can regulate or alter its functions through multiple pathways. A 2007 review has concluded that low doses of BPA during development have persistent effects on brain structure, function and behavior in rats and mice. A 2008 review concluded that low-dose BPA maternal exposure causes long-term consequences at the level of neurobehavioral development in mice. A 2008 review has concluded that neonatal exposure to Bisphenol-A (BPA) can affect sexually dimorphic brain morphology and neuronal adult phenotypes in mice. A 2008 review has concluded that BPA altered long-term potentiation in the hippocampus and even nanomolar dosage could induce significant effects on memory processes.

A 2009 review raised concerns about BPA effect on anteroventral periventricular nucleus (Polston, 2008).

Table 2.1: Many studies confirm BPA's low-dose toxicity across a diverse range of toxic effects on animal.

Dose ($\mu\text{g}/\text{kg}/\text{day}$)	Effects (measured in studies of mice or rats, descriptions (in quotes) are from Environmental Working Group)	Study Year
0.025	"Permanent changes to genital tract"	2005
0.025	"Changes in breast tissue that predispose cells to hormones and carcinogens"	2005
1	long-term adverse reproductive and carcinogenic effects	2009
2	"increased prostate weight 30%"	1997
2	"lower bodyweight, increase of anogenital distance in both genders, signs of early puberty and longer estrus."	2002
2.4	"Decline in testicular testosterone"	2004
2.5	"Breast cells predisposed to cancer"	2007
10	"Prostate cells more sensitive to hormones and cancer"	2006
10	"Decreased maternal behaviors"	2002
30	"Reversed the normal sex differences in brain structure and behavior"	2003
50	Adverse neurological effects occur in non-human primates	2008
50	Disrupts ovarian development	2009

Source: Wikipedia, 2011.

2.2.5 Studies on Human

The first large study of health effects on humans associated with bisphenol A exposure was published in September 2008 by Iain Lang and colleagues in the *Journal of the American Medical Association*. The cross-sectional study of almost 1,500 people assessed exposure to bisphenol A by looking at levels of the chemical in urine. The authors found that higher bisphenol A levels were significantly associated with heart disease, diabetes, and abnormally high levels of certain liver enzymes (Mukai, 2007).

A later similar study performed by the same group of scientists, published in January 2010, confirmed, despite of lower concentrations of BPA in the second study sample, an associated increased risk for heart disease but not for diabetes or liver enzymes. Patients with the highest levels of BPA in their urine carried a 33% increased risk of coronary heart disease.

Studies have associated recurrent miscarriage with BPA serum concentrations, oxidative stress and inflammation in postmenopausal women with urinary concentrations, externalizing behaviors in two-year old children, especially among female children, with mother's urinary concentrations, altered hormone levels in men and declining male sexual function with urinary concentrations. The Canadian Health Measures Survey, 2007 to 2009 published in 2010 found that teenagers carry 30 percent more 1 bisphenol A (BPA) in their bodies than older adults. The reason for this is not known. A 2010 study that analyzed BPA urinary concentrations has concluded that for people under 18 years of age BPA may negatively impact human immune function. A study done in 2010 reported the daily excretion levels of BPA among European adults in a large-scale and high-quality population-based sample, and it was shown that higher BPA daily excretion was associated with an increase in serum total testosterone concentration in men. A 2011 study found higher BPA levels in women with polycystic ovary syndrome compared to controls. Furthermore, researchers found a statistically significant positive association between male sex hormones and BPA in these women suggesting a potential role of BPA in ovarian dysfunction. A 2010 study found that people over age 18 with higher levels of BPA exposure had higher CMV antibody levels, which suggests their cell-mediated immune system may not be functioning properly (Cheng, 2010).

Sexual difficulties

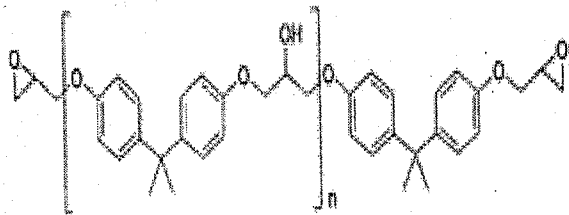
A 2009 study on Chinese workers in BPA factories found that workers were four times more likely to report erectile dysfunction, reduced sexual desire and overall dissatisfaction with their sex life than workers with no heightened BPA exposure. BPA workers were also seven times more likely to have ejaculation difficulties. They were also more likely to report reduced sexual function within one year of beginning employment at the factory, and the higher the exposure, the more likely they were to have sexual difficulties (Lee, 2009).

2.3 Epoxy Resins

Epoxy is a copolymer; that is, it is formed from two different chemicals. These are referred to as the "resin" and the "hardener". The resin consists of monomers or short chain polymers with an

epoxide group at either end. Most common epoxy resins are produced from a reaction between epichlorohydrin and bisphenol-A, though the latter may be replaced by similar chemicals. The hardener consists of polyamine monomers, for example Triethylenetetramine (TETA). When these compounds are mixed together, the amine groups react with the epoxide groups to form a covalent bond. Each NH group can react with an epoxide group, so that the resulting polymer is heavily cross linked, and is thus rigid and strong (Smith, 2009).

The process of polymerization is called "curing", and can be controlled through temperature, choice of resin and hardener compounds, and the ratio of said compounds; the process can take minutes to hours. Some formulations benefit from heating during the cure period,



Source: Wikipedia, 2011.

Figure 2.1: Structure of Unmodified Epoxy Resin

Structure of unmodified epoxy prepolymer resin. n denotes the number of polymerized subunits and is in the range from 0 to about 25

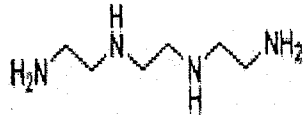


Figure 2.2: Structure of TETA

Source: Wikipedia, 2011.

Structure of TETA, a typical hardener. The amine (NH) groups react with the epoxide groups of the resin during polymerization.

Applications

The applications for epoxy-based materials are extensive and include coatings, adhesives and composite materials such as those using carbon fiber and fiberglass reinforcements (although polyester, vinyl ester, and other thermosetting resins are also used for glass-reinforced plastic).

The chemistry of epoxies and the range of commercially available variations allows cure polymers to be produced with a very broad range of properties. In general, epoxies are known for their excellent adhesion, chemical and heat resistance, good-to-excellent mechanical properties and very good electrical insulating properties. Many properties of epoxies can be modified (for example silver-filled epoxies with good electrical conductivity are available, although epoxies are typically electrically insulating). Variations offering high thermal insulation, or thermal conductivity combined with high electrical resistance for electronics applications, are available (Forman, 2003).

Paints and coatings

Two part epoxy coatings were developed for heavy duty service on metal substrates and use less energy than heat-cured powder coatings. These systems use a 4:1 by volume mixing ratio, and dry quickly providing a tough, protective coating with excellent hardness. Their low volatility and water cleanup makes them useful for factory cast iron, cast steel, cast aluminium applications and reduces exposure and flammability issues associated with solvent-borne coatings. They are usually used in industrial and automotive applications since they are more heat resistant than latex-based and alkyd-based paints. Epoxy paints tend to deteriorate, known as chalk out, due to UV exposure (Lindsey, 2001).

Polyester epoxies are used as powder coatings for washers, driers and other "white goods". Fusion Bonded Epoxy Powder Coatings (FBE) are extensively used for corrosion protection of steel pipes and fittings used in the oil and gas industry, potable water transmission pipelines (steel), concrete reinforcing rebar, etc. Epoxy coatings are also widely used as primers to improve the adhesion of automotive and marine paints especially on metal surfaces where corrosion (rusting) resistance is important. Metal cans and containers are often coated with epoxy to prevent rusting, especially for foods like tomatoes that are acidic. Epoxy resins are also used for high performance and decorative flooring applications especially terrazzo flooring, chip flooring and coloured aggregate flooring (Hanberg, 2005).

2.4 Adhesives

Special epoxy is strong enough to withstand the forces between a surfboard fin and the fin mount. This epoxy is waterproof and capable of curing underwater. The blue-coloured epoxy on the left is still undergoing curing.

Epoxy adhesives are a major part of the class of adhesives called "structural adhesives" or "engineering adhesives" (that includes polyurethane, acrylic, cyanoacrylate, and other chemistries.) These high-performance adhesives are used in the construction of aircraft, automobiles, bicycles, boats, golf clubs, skis, snowboards, and other applications where high strength bonds are required. Epoxy adhesives can be developed to suit almost any application. They can be used as adhesives for wood, metal, glass, stone, and some plastics. They can be made flexible or rigid, transparent or opaque/colored, fast setting or slow setting. Epoxy adhesives are better in heat and chemical resistance than other common adhesives. In general, epoxy adhesives cured with heat will be more heat- and chemical-resistant than those cured at room temperature. The strength of epoxy adhesives is degraded at temperatures above 350 °F (177 °C).

Some epoxies are cured by exposure to ultraviolet light. Such epoxies are commonly used in optics, fiber optics, optoelectronics, and dentistry.

Industrial tooling and composites

Epoxy systems are used in industrial tooling applications to produce molds, master models, laminates, castings, fixtures, and other industrial production aids. This "plastic tooling" replaces metal, wood and other traditional materials, and generally improves the efficiency and either lowers the overall cost or shortens the lead-time for many industrial processes. Epoxies are also used in producing fiber-reinforced or composite parts. They are more expensive than polyester resins and vinyl ester resins, but usually produce stronger and more temperature-resistant composite parts (Magher, 2003).

Health risks

The primary risk associated with epoxy use is sensitization to the hardener, which, over time, can induce an allergic reaction. It is a main source of occupational asthma among users of plastics. Bisphenol A, which is used in epoxy resin, is a known endocrine disruptor.

2.4 Polycarbonate Plastics

Polycarbonate is a tough, dimensionally stable, transparent thermoplastic that has many applications which demand high performance properties. This versatile thermoplastic maintains its properties over a wide range of temperatures, from -40 °F to 280 °F. It is available in three types: machine grade; window and glass-filled. It is the highest impact of any Thermoplastic, transparent up to 2" in special grades, outstanding dimensional and thermal stability, exceptional machinability, stain resistant and non-toxic with low water absorption.

Machine Grade is relatively stress free to permit the most demanding machining. It is also available in glass-filled. This polycarbonate is perfect for high performance uses in tough applications over a broad temperature range.

Window Grade is optically clear, providing total luminous transmittance and very low haze factor. The high impact strength makes it resistant to repeated blows, shattering and spalling.

Glass-Filled

Glass-reinforced polycarbonate is finding principal applications in designs where metals, particularly die-cast aluminum and zinc, are commonly used. The coefficient of thermal expansion is reduced by nearly 75 %, thus equaling that of some metals. While glass-reinforced has less impact strength than standard grades, it is still tougher and more impact resistant than most other plastics and die cast aluminum.

2.4.1 Advantages to Polycarbonate

2.4.1.1 *Impact strength*

Unnotched polycarbonate is virtually unbreakable, making it extremely safe in areas where parts

may be exposed to impact. When exposed to repeated heavy blow, the material tends to cold form rather than shatter.

2.4.1.2 Electrical

Polycarbonate is excellent for electrical applications, because of its high dielectric strength and high volume resistivity which decreases only slightly as temperature or humidity is increased.

2.4.1.3 Machinability

Parts can be easily machined from standard metal working tools. No special tools are needed, and finished parts can be polished to a high gloss. Water or water-soluble cutting oils should be used when machining polycarbonate, since some standard cutting oils will attack the material. Polycarbonate can be machined on standard metalworking or woodworking equipment. Its unique properties permit it to be machined without chipping, splitting, or breaking.

2.4.1.4 Annealing

Polycarbonate slab (Zelux) has been stressed relieved using Liquo-Temp annealing process. In some instances where extensive machining is required, a secondary annealing of semi-finished parts is highly recommended. Secondary annealing can be accomplished by heating parts at 250°F in a desiccated air circulating oven for one hour per one inch of thickness. After heating, the oven should be turned off and allowed to cool to room temperature spontaneously.

2.4.1.5 Bonding

Polycarbonate can be mechanically bonded by standard methods. It can also be cemented by using a solvent such as methylene chloride or adhesives such as epoxy, urethane and silicone. Polycarbonate can also be ultrasonically or vibrationally welded.

2.4.1.6 Removing Paint or other Materials

Fresh paint may be removed by rubbing lightly with a cleaning material such as isopropyl alcohol, or VM-P grade naphtha. Then the sheet should be washed immediately with a mild soap or detergent in warm water, and rinsed thoroughly with clean water. Grease and glazing compound may similarly be removed from the surface with the above mentioned cleaning

materials. Weathered paint may be lifted off the sheet with masking tape. Razor blades or other sharp scraping tools should never be used.

2.4.1.7 More Advantages of Polycarbonate

Formability

Standard polycarbonate sheet is not heat formable; however, formable sheet is available on a custom basis. Standard Lexan polycarbonate sheet can be heat formed with proper pre-drying. Lexan sheets up to 114" may also be cold formed under special conditions.

Cleaning

Kleenmaster Brillianize may be used. (Also see Novus) Products such as abrasive or highly alkaline cleaners, acetone, carbon tetrachloride, benzene or leaded gasoline should not be used, and the sheet should not be cleaned in hot sun or at high temperatures.

Scratch Removal

Craftics 20/20 Plasti-Polish Scratch Remover is often all that is required to subdue hairline scratches and minor abrasions. Any polish, however, should be tested first on a sample area of the polycarbonate sheet. San Diego Plastics stocks Craftics 20/20 in 8 ounce and 1 gallon sizes.

Decorating

Polycarbonate products will accept painting, printing, or vacuum metalizing as decorating methods.

UV Stabilization

Natural and Black Machine grade and Window grade are UV stabilized. Polycarbonate rod, machine grade, is not UV stabilized, but is available on custom quotation.

2.4.2 Applications of Polycarbonate

- Lenses
- High temperature and pressure windows

- Face shields
- Industrial equipment and housing components
- Medical equipment components
- Instrument components
- Electrical insulators and connectors
- Aircraft & Missile components
- Portholes in pressure chambers
- Jet pump impellers and diffusers
- Automotive parts
- Card guides
- Assembly line cogs

Chapter Three

3.0 METHODOLOGY

This chapter covers the gathering of materials and equipment for the experimental procedure and analysis of the samples.

The Table below shows the list of materials for the experiment and their sources.

Table 3.1: List of Materials for the Experiment

S/N	Name	Research code name	Source	Comment
1	Carbolic acid	Phenol	Chemical enr. Laboratory	Highly inflammable
2	Acetone	Acetone	Chemical enr. Laboratory	Highly inflammable
3	Hydrochloric acid	HCL	Chemical enr. Laboratory	Concentrated HCL

The Table below shows the list of equipment used for the experiments and their sources.

Table 3.2: List of Equipment for the Experiment.

S/N	Name	Source	Comment
1	Three necked round bottom flask	Science equipment development institute	Mixing flask &
2	Magnetic stirrer & heater	Chemical Laboratory	enr. Heater and stirrer
3	Liquid phase chemical reactor	Armfield England	Water flow in the condenser
4	Condenser	Chemical Laboratory	enr.
5	Retort stand	Chemical Laboratory	enr. Holding equipment
6	Beaker	Chemical Laboratory	enr.
7	Separating funnel	Chemical Laboratory	enr. Separating equipment

3.1. Experimental Procedure

Procedure for the production of Bisphenol A (BPA);

The setup for the production of Bisphenol A is as shown in the figure below.

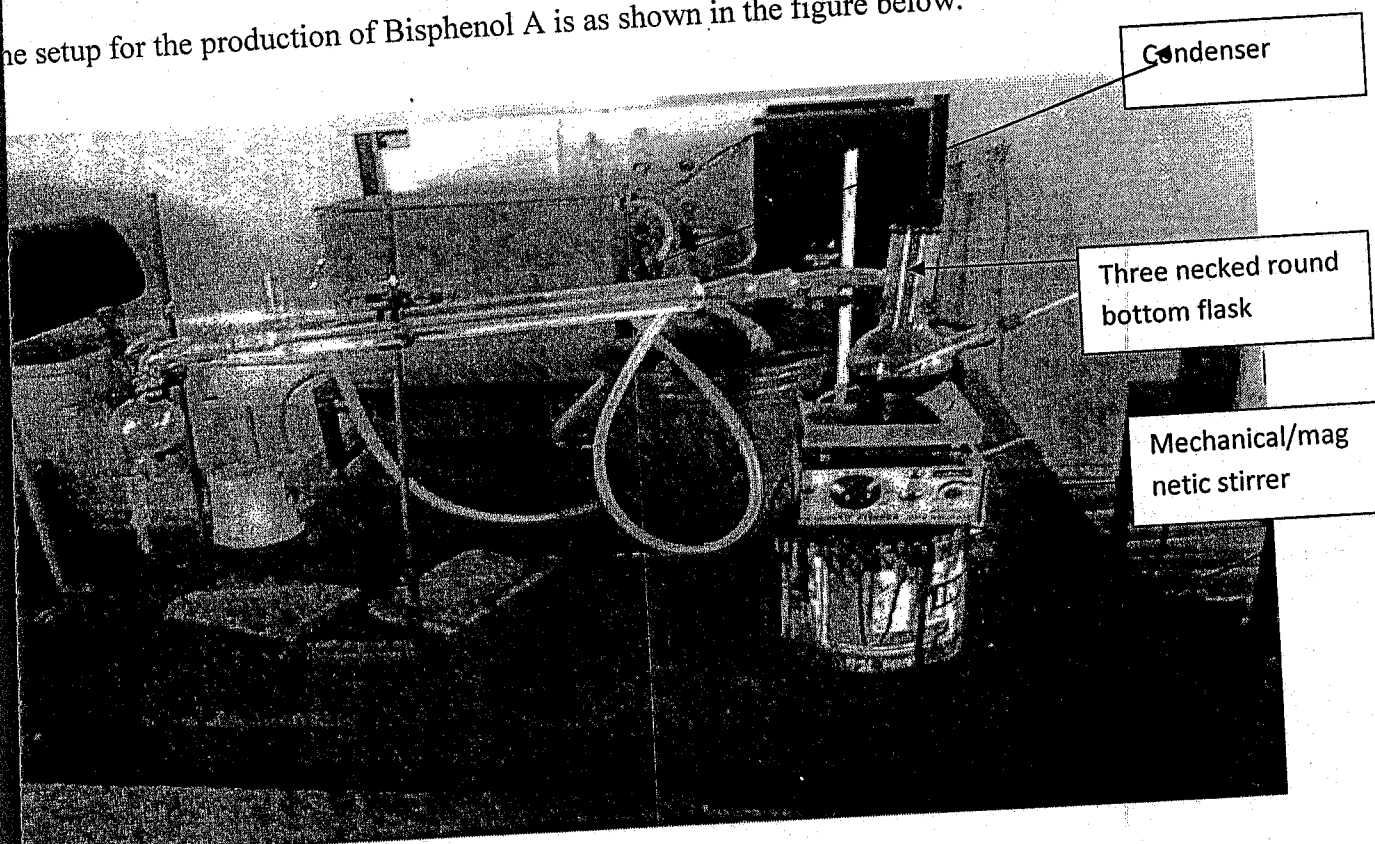


Plate 3.1: Experimental set up

167 g of phenol was weighed and poured into a beaker and then transferred poured into the three necked round bottom flask, followed by 86.65 g of acetone then 110.5 g of HCL. This mixture was allowed to settle for five minutes before heating commenced. The mixture was heated in the three necked round bottom flask using a magnetic stirrer/heater for one hour at a temperature of 60°C . After a definite period of time (30 minutes), the colour of the reaction mixture changes to a light brown which settles at the bottom of the flask, whilst the upper part of the mixture remained dark red (phenol), the light brown colour obtained is the Bisphenol A. A separating funnel was used in separating the mixture.

3.2 Characterization

After the production of bisphenol A, the properties were characterized as follows:

2.1 Determination of Solubility in water

A few drops of Bisphenol A was dropped into 20 ml of water and was stirred to ensure that the mixture was well mixed.

2.2 Determination Flash point (ASTM D93)

Procedure

The testing cup made of non-rusting metal Brass was inserted into the Pensky – Martens closed cup tester after Bisphenol A has been poured into it. A thermometer was stuck into the apparatus and allowed to touch the sample in order to check the temperature at which the flash occurred. The test flame was applied across an open slit above the sample in a test cup at right angle to it, and not more than 3 mm above it, the flame was removed intermittently in order to allow for stirring. The temperature at which the instantaneous flash occurred on the application of the flame was taken and recorded as the flash point of the sample.

3.2.3. Determination of Boiling point

The boiling point of a liquid is the temperature at which the vapour pressure of the liquid equals the atmospheric pressure surrounding the liquid.

Procedure

20 ml of the Bisphenol A was placed in a beaker on the hot plate. As the BPA liquid boils, a thermometer was immediately inserted and the boiling point of the reaction was recorded.

3.2.4 Determination of Relative density

Procedure

The relative density was measured using hydrometer. The hydrometer was inserted into the sample. The reading on the hydrometer corresponding to the liquid level was noted.

2.5 Determination of purity

phenol A was injected into a Philips PU 9712 infra red spectrophotometer to test for the purity.

CHAPTER FOUR

4.0. RESULT AND DISCUSSION

4.1 Result

The following results were obtained after the production and characterisation Bisphenol A.

Table 4.1 Characterization of Bisphenol A.

Properties	Values obtained	Standard Value
Solubility in water	Insoluble in water	Insoluble (300mg/l)
Flash point $^{\circ}\text{C}$	192	207
Appearance	Light brown	White – Light brown
Boiling point ($^{\circ}\text{C}$)	235	220
Relative density (g/cm^3)	0.93	1.20 solid
Purity %	71	Not stated

4.2 Discussion of Result

The Bisphenol A (BPA) produced was characterized and the values were compared with the standard values from literatures. Table 4.1 shows the results of the characterization of the Bisphenol A produced, the purity was measured using a Philips UO971 infra red spectrophotometer, and it was found to be 71 % pure. This could be as a result of the temperature reaction, phenol/acetone molar ratio, volume of acid catalyst, concentration of the promoter. Rahimi and Farhangzadeh (2001) studied the kinetics of Bisphenol A by condensation reaction, they found that certain factors are responsible for the yield and purity of the Bisphenol A produced. The purity of the Bisphenol A produced is 71 %, this could be as result of the reaction temperature, phenol/ acetone molar ratio, volume of acid catalyst, concentration of promoter.

The Bisphenol A produced is insoluble in water. This is in view of the fact that Bisphenol A is a non-polar organic compound. In addition, the product has a higher boiling point contrary to the value obtained from standard literature. The discrepancy could be as a result of the presence of impurities, the Bisphenol A produced is 71 % pure. This implies that the increment in the boiling point is as a result of the 30 % impurity which may be unreacted phenol and acetone, the catalyst used in the reaction.

The relative density of the Bisphenol A is 0.92 g/cm^3 liquid, it was kept as a liquid for storage purpose, but an almost pure Bisphenol A exists as solid at room temperature so its relative density in solid state is 1.20 (solid). It was also observed that the Bisphenol A produced is light brown in colour, this conform well to the standard colour.

Chapter Five

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The Bisphenol A was developed by reacting 3 volumes of phenol with 1 volume of acetone at a temperature of 60 °C for a residence time of 1 hour. The product was characterized by testing for its purity, relative density, flash point, solubility in water and boiling point. The values of the Bisphenol A obtained were compared to the standard ones. The results of the comparison showed that the experimental values obtained are in agreement with the standard ones reported in the literature.

The purity of the Bisphenol A can be improved by quenching the product in water and passing the product to further purification stages.

5.2 Recommendation

The following recommendation will be helpful for future research on this work.

1. A steady supply of electricity is very necessary for the production of the Bisphenol A, so power alternatives should be put in place.
2. Research should be carried on the optimum conditions for the production of Bisphenol A.
3. A more standard laboratory should be used for the experiment. This will ease the stress of production of the product.

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