PRODUCTION OF GOLD AND SILVER NANOPARTICLES RECOVERED THROUGH SONICATION OF REFLECTIVE METAL LAYER IN CD-R WASTE

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LIST OF ABREVIATIONS

CD	Compact Disc
CD-R	Compact Disc
CD-ROM	Compact Disc Read Only Memory
CRT	Cathode Ray Tube
DNA	Deoxyribonucleic acid
EDX	Energy Dispersive X-Ray
	Spectroscopy
EELS	Electron Energy Loss Spectroscopy
EF-TEM	Energy Filter Transmission Electron
	Microscopy
EOL	End-of-Life
EPA	Environmental Protection Agency
e-waste	Waste electrical and electronic
	equipment
FE-SEM	Field Emission Scanning Electron
	Microscopy
METSS	Technology-based company that
	develops innovative solutions to
	technically challenging problems
MSW	Municipal solid waste
NP	Nano Particle
PC	Polycarbonate
PCCS	Photon Cross correlation

Spectroscopy

S1-S11	Step 1 to Step 11
US	United States

LIST OF SYMBOLS

%	Percent
Ψ	Span value
°C	Degree celcius
μm	Micron meter
μs	Micron second
Ag	Argentum
Al	Aluminium
atms	Standard atmosphere
Au	Aurum
c	Velocity
d ₁₀	Particle diameter at 10% cumulative
	size
d ₅₀	Particle diameter at 10% cumulative
	size
d ₉₀	Particle diameter at 90% cumulative
	size
dl/g	Deciliter per gram
Н∙	Hydrogen radicals
H_2O_2	Hygrogen peroxide
$HO_2 \bullet$	Perhydroxyl radicals
Hz	Hertz
Κ	Kelvin
kHz	Kilohertz

ks ⁻¹	Kilo per second
kV	Kilovolt
kW	Kilowatt
М	Mol
MHz	Megahertz
min	Minute
ml	Mililiter
mm	Milimeter
ms ⁻¹	Meter per second
nm	Nanometer
nm ²	Nanometer square
ОН∙	Hydroxyl radicals
ppm	Part per million
rpm	Rotation per minute

PENGHASILAN NANO-PARTIKEL EMAS DAN PERAK YANG DIPEROLEH MELALUI PROSES SONIKASI LAPISAN LOGAM TERPANTUL DALAM CD-R TERBUANG

ABSTRAK

Lapisan logam terpantul (emas dan perak) dalam CD-R terbuang telah dipisahkan melalui kaedah pemisahan fizikal dan kimia. Kedua-dua jenis lapisan logam terpantul (emas dan perak) kemudiannya disonikasi di dalam unit sonikasi, Sonoswiss SW 12 H. Kesan daripada tiga parameter yang beroperasi ketika proses sonikasi dikaji iaitu ciri sonikasi (menghilangkan gas, menyapu dan meningkatkan), masa sonikasi dan suhu operasi. Kemudian, pencirian produk dilakukan melalui analisis saiz zarah, Mikroskopi Imbasan Elektron Pemancaran Medan dan Spektroskopi Serakan Tenaga dan Mikroskopi Penghantaran Elektron Penapis Tenaga dan Spektroskopi Kehilangan Tenaga Elektron. Melalui kajian ini didapati bahawa proses kitar semula CD-R terbuang boleh dijalankan dengan mudah seperti pemisahan fizikal dan kimia menggunakan 0.5 M aseton untuk emas dan 0.5 M HCL untuk perak. Penghasilan nano-partikel emas halus telah berjaya dilakukan dengan menggunakan ciri sapu pada julat operasi suhu yang tinggi (55-65°C) pada 10 min menghasilkan d₅₀ minimum pada 1.01 nm dan nilai jangka di 0.048. Begitu juga, penghasilan nano-partikel perak halus telah berjaya dihasilkan dengan menggunakan ransangan ciri pada julat operasi suhu yang tinggi (55-65°C) pada 30 min menghasilkan d_{50} minimum pada 1.33 nm dan nilai jangka pada 0.281.

PRODUCTION OF GOLD AND SILVER NANOPARTICLES RECOVERED THROUGH SONICATION OF REFLECTIVE METAL LAYER IN CD-R WASTE

ABSTRACT

Reflective metal layer (gold and silver) in CD-R waste were separated by physical and chemical separation methods. Both types of reflective metal layer (gold and silver) were then sonicated in the ultrasonic units, Sonoswiss SW 12 H. The effect of three operating parameters during the sonication process were investigated, namely sonication features (degas, sweep and boost), sonication time and operating temperature. The product was then characterized by particle size analysis, Field Emission Scanning Electron Microscopy and Energy Dipersive Spectroscopy and Energy Filter Transmission Electron Microscopy and Electron Energy Loss Spectroscopy. It has been confirmed through the present study that the recycling process of CD-R waste can be carried out using simple methods such as physical and chemical separation using 0.5 M of acetone for gold and 0.5 M of HCL for silver. Production of fine gold nanoparticles was successfully done using sweep feature at high operating temperature range (55-65°C) at 10 min resulting in the minimum d_{50} at 1.01 nm and span value at 0.048. Similarly, production of fine silver nanoparticles was successfully done using boost feature at high operating temperature range (55- 65° C) at 30 min resulting in the minimum d₅₀ at 1.33 nm and span value at 0.281.

CHAPTER 1

INTRODUCTION

1.1 Introduction

It is undeniable that optical discs are one of the main media which have been extensively used nowadays. Optical disc have been produced in large amount, particularly after the great success of the compact disc (CD), which was introduced in the market in 1982, and accompanied by related CD-ROM. One of the reasons is that the Compact Disc Recordable Once (CD-R) is mainly used as a test disc before starting mass production of CD/CD-ROMs, since CD-R is compatible with CD/CD-ROM. When CD-R is used for this purpose, it can be disposed of after being used. In this sense, CD-R generally has a shorter life-time compared to ordinary CD and CD-ROM. Therefore recycling of CD-R is an urgent issue (Tomita et al., 1998) and need attention from community.

The production of compact discs worldwide has reached 7000 million in 2006 and has decreased significantly in recent years. The production of compact disc (CD-R) worldwide in 2013 was 2500 billion pieces; Figure 1.1 gives some statistical information about Global Demand and Production of CD-R from 2006 to 2013. It is observed that the trend decreased due to the new demand and production of Blue-ray discs (Semiconportal, 2011). It has been estimated that approximately 10% of all CDs manufactured are rejected due to the complex manufacturing process (Zevenhoven and Saeed, cited in Pohimann 1989). In the US alone, more than two billion polycarbonate compact discs are manufactured each year, and around 150200, million rejected CDs are scrapped annually. CDs are also destroyed because of artist's right issues. These together with some post consumer CDs are currently being reclaimed to produce grades of recycled polycarbonate (PC) (Zevenhoven and Saeed, cited in Scheir 1998).

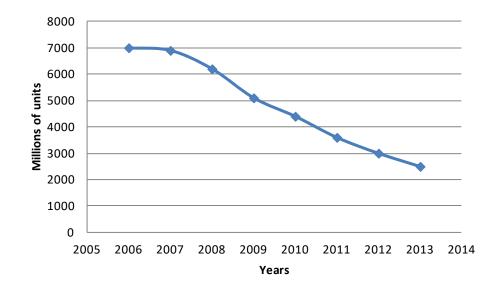


Figure 1.1: Global Demand and Production of CD-R from 2006 to 2013 (Semiconportal, 2011).

1.2 The structure of optical discs

The nature of the compact disc (CD) does not allow it to be recycled easily. Figure 1.2 shows that the disc is a multilayer product consists of different types of materials: (a) polycarbonate base (substrate); (b) recording layer (dye); (c) reflective layer (metal layer); (d) protective layer (laquer and printing layer) (Barletta et al., 2007; Angnes et al., 2000; Tomita et al., 1998). CD-R has valueable material such as polycarbonate substrate and reflective layer (Au, Ag and Al) (Tomita et al., 1998; Kimt & Min, 1997).



Figure 1.2: Schematic diagram showing the disc as a multilayer product of different types of materials: (a) polycarbonate base (substrate); (b) recording layer (dye); (c) reflective layer (metal layer); (d) protective layer (laquer and printing layer) (Barletta et al., 2007; Angnes et al., 2000).

1.3 Problem Statement

Recently, discarded electrical and electronic equipment are known as e-waste. CD-R waste also is one category of electrical waste and electronic equipment (ewaste). It can be potentially hazardous, if it is improperly handled. One example of the e-waste are printed circuit board and cathode ray tubes (CRTs) which contain heavy metals such as nickel, chromium, tin, lead, copper and lead oxide; respectively.

CD-R waste contains precious materials in the metal reflective layer which is gold and silver. If CD-R waste is disposed in landfills, the metals components will be lost for future use. In accumulation, these materials have also potential to leach out into landfills and will contaminate the surface and ground water. On the other hand, precious materials resources that could be recovered from CD-R waste will contribute towards sustainable development and environmental improvements. This will also support National Mineral Policy 2, 2009 towards of environmental stewardship (National Mineral Policy 2, 2009).

Research on application of silver and gold nanoparticles from metal reflective layer waste is still new and only limited study has been done on this work. Komine et al., (2000) proposed a method of recycling disc recording medium and apparatus for recovering metal reflective layer. However they do not identify the application of metal reflective layer in their report. There are varieties of methods for the optical disc recycling process especially for the removal of paint or painting from engineering plastics, ranging from the chemical to physic-mechanical procedures. Such techniques include chemical stripping or chemical recovery (high temperature alkaline treatment) melt filtration, mechanical abrasion, hydrolysis, liquid cyclone, compressed vibration, cryogenic grinding, dry crushing and roller pressing.

The aim of this study is to recover the metal reflective layers from CD-R waste using physical and chemical separation method followed by production of gold and silver nanoparticles through sonication method.

1.4 Objectives

The objectives of the present study are:

1. To recover gold and silver from the CD-R waste using separation methods; physical and chemical separation.

- 2. To optimize reduction size of gold and silver metal reflective layer in ultrasonic bath using different sonication features (degas, sweep and boost).
- 3. To determine the effect of sonication operating condition such as operating temperature and sonication time on the particle size and morphology of gold and silver obtained from metal reflective layer.

1.5 Scope of work

The scope of this work includes recovery of metal reflective layer (gold and silver) and the application of ultrasonic process to reduce size of gold and silver metal particles from reflective layer of CD-R waste. Ultrasonic bath with different operational parameters such as operating temperature, sonication features and sonication time were studied in the present study. Characterizations such as particle size analysis and particle size distribution, morphology using scanning electron microscopy and energy-dispersive X-ray Spectroscopy, and Energy Filter Transmission Electron Microscopy with Electron Energy Loss Spectroscopy were used to identify the particles size and elemental of the gold and silver metals recovered from the CD-R.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will be divided into nine sections. Explanation on characteristics of CD write once (CD-R), polycarbonate layer and metal reflective layer is made in the first section followed with legislations related to e-waste. This chapter also highlights various studies and published works on the recycling of compact disc. The explanation focuses on two categories which are mechanical and chemical recycling. Ultrasonic methods (degas, sweep and boost) are discussed in detail since this method is applied in the present work. General theory of ultrasonics, problems occurred during sonication process and three sonication features in ultrasonic units, Sonoswiss SW 12 H are explained in this chapter. Applications of nanosilver and nanogold in the three main fields such as environment, health and technology applications are discussed in brief at the end of this chapter.

2.2 Characteristics of CD write once (CD-R)

CD writes once (CD-R) is described as writable or recordable discs (Smith, 1997-2008). CD-R can be disposed of after being used for its purpose (Tomita et al., 1998). Various texts, pictures, sounds and moving pictures can be recorded in the disc. The disc can be used for data backup or for test distributions of newly developed software in small amounts (Kim et al., 1997). As mentioned in Chapter 1,

CD-R has high precious materials such as polycarbonate substrate and reflective layer (Au, Ag and Al) (Tomita et al., 1998; Kimt & Min, 1997). This chapter will extend the information about the characteristic of polycarbonate and metal reflective layers of CD-R.

2.3 Polycarbonate layer

Polycarbonate is the main body in CD-R. Properties of the polycarbonate fulfil requirements of CD-R manufactures. Its optical properties match the needs of CD-R production. Washing out the salt impurities and reducing the moisture content which produce high transmission at the wavelength reading of 780 nm become another excellent properties polycarbonate in CD-R manufacture (Barletta et al., 2007). All the properties above prove that polycarbonate is the best material compared to other thermoplastics (Barletta et al., 2007).

2.4 Metal Reflective layers

Reflective layer (Au, Ag, and Al) is required to be compatible with CD player (Angnes et al., 2000). This layer also known as nanometric metal layer, which contains very thin lines to be tracked during the recording process (Kim et al., 1997). Different metal used give different thickness, the thickness of this mirror like film is between 50 and 100 nm (a media value of 80 nm was found by Rutherford backscattering measurements), and the total area is ~ 100 nm². Gold used in CD-R is a real gold and it has very thin layer 80 nm, and the thickness of aluminium layer is only 55-70 nm thick (Zevenhoven and Saeed, 2003). Gold give larger and sharper

peaks when the light was bombared on the CD-R surface to save the data from other media CD-R (Kimt & Min, 1997) but at the low price of commercial recordable CDs. Aluminium also preferred as a reflective layer. Due to the weakness of aluminium such as 1) not fully exposed; 2) does not provide sufficient reflectively and 3) the dye would chemically attack aluminium. Silver used as the alternative to overcome the weakness by providing sufficient reflectively and not easy to expose to chemical attack.The metal reflective layer (gold and silver) is applied by sputtering process (Barletta et al., 2007).

2.5 Legislations

Recycling means the reprocessing in a production of waste materials for the original purpose or for other purposes. Recycling of electronic waste involves disassembly and/or destructions of the End-of-Life (EOL) equipment in order to recover materials (Cui & Zhang, 2008). When compact discs are placed in the trash, it is harmful to the environment. When discs are recycled properly, it will help to stop unnecessary pollution, conserve natural resources and slow global warming.

Two important subjects which are highlighted in the recycling of electronic waste are waste treatment and recovery aspect of valuable materials. The US Environmental Protection Agency (EPA) has identified major benefits, such as saving in energy and reduction in pollutions when scrap metal are used instead of virgin materials. Using recycled materials in place of virgin materials result in significant energy saving (Cui & Zhang, 2008).

The increased demand and production of electronic products in the last decades have created problems with regards to the where about of rising number of waste electrical and electronic equipment (e-waste). Although a significant proportion of obsolete electronic waste eventually winds up in landfills and municipal waste incinerators, there is and growing number that ends up in recycling facilities in order to recover reusable and recyclable materials and trace amount of precious metals (Wang et al., 2009).

2.6 Recycling process

Recycling process in polymer can be classified into four categories e.g. primary, secondary, tertiary and quaternary recycling. Primary recycling is a preconsumer industrial scrap, primary recycling of industrial scrap produce during manufacture of food–contact articles is not expected to pose a hazard to the consumer (Sinha et al., 2008). It is the clean recycling process, uncontaminated single-type waste which remains the most popular, as it ensures simplicity and low cost, especially when done "in plant" and feeding with scrap of controlled history (Sinha et al., cited in Warren 1998). The recycled scrap or waste is either mixed with virgin material to ensure product quality or used second grade material (Sinha et al., cited in Naele 1983).

Mechanical recycling used for recycling CD-R is secondary recycling. The basic polymer is not altered during the process. Mechanical recycling includes the sorting and separation of the wastes, size reduction: melt filteration and reforming of the plastic material. In mechanical recycling, the polymer is separated from its associated contaminants and it can be readily reprocessed into granules by conventional melt extrusion (Azapagic et al., 2003).

Tertiary recycling used for recycling CD-R is chemical recycling; it is on the other hand uses chemical process to convert waste into useful products, such as monomers for new plastics, fuels or basics chemicals for general chemical production (Barletta et al., 2007). The chemical recycling is carried out either by solvolysis or by pyrolysis, the former through degradation by heat in absence of oxygen or air, or vacuum. Chemical recycling yields monomers, petroleum liquids and gases. Monomers are purified by distillation and drying, and used for manufactured of polymers (Sinha et al., 2008).

Lastly, is quaternary recycling or energy recovery. Energy recovery generates heat or electricity (or both) either by direct incineration of polymer waste for the example in municipal solid waste (MSW) incinerators, or by replacing other fuels. e.g. in blast furnaces, cement kilns or power station (Barletta et al., 2007). In this thesis, discussion is focused on mechanical recycling (secondary recycling) and chemical recycling (tertiary recycling).

2.6.1 Mechanical Recycling

Physical and mechanicals means uses in mechanical recycling, such as grinding, heating and extruding to process waste plastics into new products. Clean and homogeneous waste is required for this process, which means that plastics have to be sorted by type and separated before they can be incorporated in virgin polymers of the same type or used on their own. The availability of homogeneous waste streams of known characteristics is thus a key criterion for successful recycling (Azapagic et al., 2003).

Separation of different polymers is particularly important for mechanical recycling because processing mixed materials would otherwise produce recyclate of low quality, which could only be used in a limited number of applications. Hence, mechanical recycling is really best suited to clean plastic waste, such as packaging material (Azapagic et al., 2003). The mechanical separation process, also called the mechanical abrasion process, appears to be best way for the recovery of polycarbonate from a compact disc since considered being safe, effective, and simple process. Coatings can be removed from engineering plastics by mechanical abrasion processing (Sapienza, 2002).

Method for defacing compact discs was built by Sargent, et al., (1996). The schematic of this method is shown in Figure 2.1. This system was built up for defacing compact discs so as destroy information stored thereon includes a defacer unit and feed unit for feeding discs one at a time, with vertical orientation, to the defacer unit. The feeder unit includes a conveyor which drops the disc into a vertically-oriented tube in which they are staked. One disc at a time is ejected from the bottom of the stack, through a narrow slot in the tube, and then drops with vertical orientation into the defacing unit. The defacing unit receives the vertically-oriented disc between two sets of driven rubber belts which engage opposite sides of the disc and pulls it between opposed knurling wheels which mark opposite sides of the disc with embedded grooves having an identifiable design. The knurling wheels

are mounted on a suspension system which controls their penetration to a depth to render the disc unplayable yet preventing cracking of the disc substrate (Sargent et al., 1996).

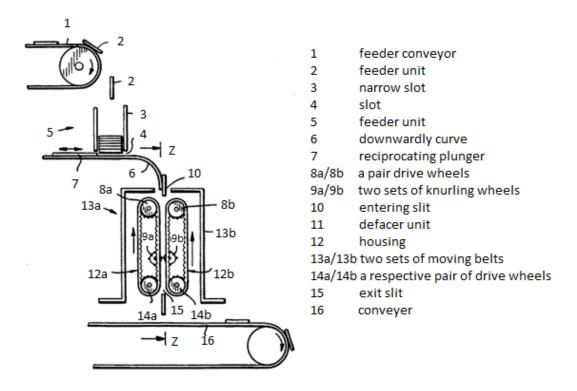


Figure 2.1: Schematic diagram of defacing compact disc (Sargent et al., 1996).

Figure 2.2 shows adhesive principles have in optical recording disc recycling method and ultrasonic separation method to separate substrate from thin-film. Optical recording disc recycling method has been introduced by Uryu et al., (1999). Recycling of optical disc including at least a light transmitting substrate and at least one metal layer containing a metal such as aluminium and gold provided on one side of the substrate. This recycling method includes an adhesion step for adhering an adhesive member to the substrate side having a metal layer, and separation step for peeling out the adhesive member so as to remove the metal layer from the substrate

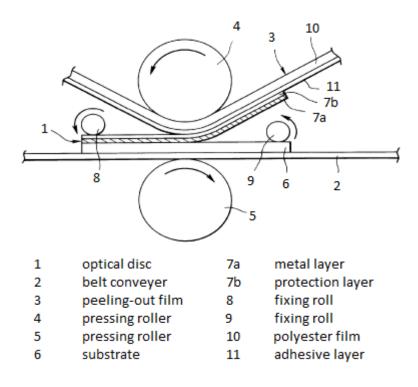


Figure 2.2: Schematic diagram of peeling off process of peeling-off adhesive film (Uryu et al., 1999).

Method of recovering resin suggested by Shinomiya et al., (2000). They reported on a process for recovering the resin contained in optical information recording media having either a thin metal film layer or a printed coating film layer, coated on at least one side of a resin substrate. The following steps were comprised clearly about this method; (1) rolling the recording media, (2) feeding the rolled recording media into contact with hot water; (3) separation process the coating film peeled from the recording media; (4) grinding the obtained recording media; (5) feeding the obtained small pieces of the recording media into contact with hot water. This is to detach the thin metal film. Component feeding roll led the thin metal film and coating layer to peeled off together with hot water from polycarbonate. In this method coating layer was effectively can remove from substrate, and the substrate can be selectively recovered advantageously in industry.

As suggested by DeFazio (1993), Figure 2.3 shows a schematic diagram of milling apparatus for shaving down the face of a plastic compact disc. This patent is useful in the recycling process of manufacturing compact discs that have metal layer and a resin-coated surface, the improvement is in the stripping off the metal layer and resin coated surface from rejected discs after the surface has been stripped off for remelting. In this method, plastics having coatings are reused by first removing the coatings by shaving or treatment with solvents or jets of hot liquid or vapour that melt the coatings and blow them away. Thin plastic (compact discs) are passed through surface milling apparatus which shaves off the coatings. After shaving, the discs are brushed while rotating around their centers.

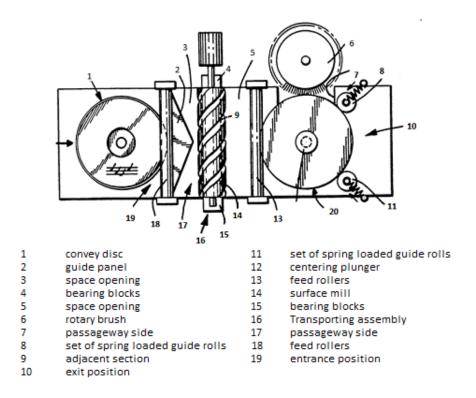


Figure 2.3: Schematic diagram of milling apparatus to shave compact disc (DeFazio, 1993).

Figure 2.4 shows schematic diagram of recovery silver and polycarbonate from optical discs. Giovanella et al. (2008) focused on recovered compact disk and a

method and an apparatus for recover thereof using the method comprises the steps of holding the compact discs. The method involves a rotating brush to remove coating and a conveyer belt to continuously move the discs. Then, the particles are carried to special filters as to recovery the aluminium after the brushes remove the coatings. While the disc was being stripped, a vacuum was applied to each of the holders to provide stability, for cooling and to prevent melting of polycarbonate being stripped, compressed air, inert gas, or a water mist can be applied at the interface. The discs, once stripped, can be saved as a whole or can be granulated.

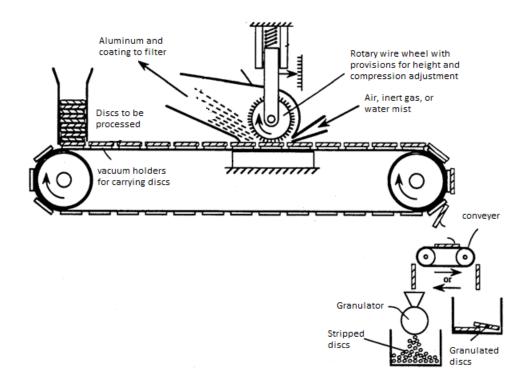


Figure 2.4: Schematic diagram of recovery silver and polycarbonate from optical discs (Giovanella et al, cited in Boudreau 1992).

Witt (1997) introduced a process and device for mechanically removing a layer from the substrate material of a disc-shaped information carrier. Figure 2.5 shows a vertical section through an arrangement comprising a tool top part and tool bottom part, exhibiting an inserted disc-shaped in-formation carrier. The hold-down clamps prevent the compact disc from lifting out of the receiving fixture. A centric centering bolt was served the centering, which catering bolts juts partially into a center-hole in the compact disc from lifting out of the receiving fixture, the hold-down clamps was used .

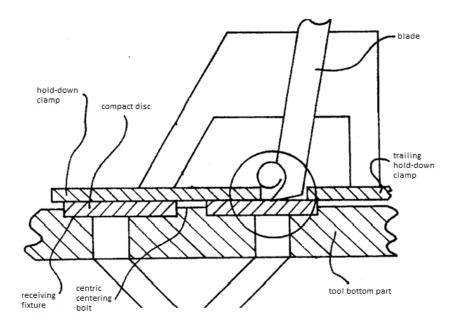


Figure 2.5: Schematic diagram of a disc-shaped information carrier (Witt, 1997).

Method for separating substrate and thin-film layer of optical recording memory media using ultrasonic had been designed by Wang et al. (2009). The processing methods included steps of placing an optical recording memory media in an ultrasonic environment and using ultrasonic to damage weaker interconnected interfaces to absorb energies for attenuating or losing adhesive forces thereof layers. Classification process where diverse optical recording memory media are classified according recycling materials was carried out as an initial step. For example, material such as gold, silver and aluminium contained in the reflective layers are classified differently. For, the optical recording memory media may be classified according to disparities in substrate materials thereof. Then, followed by separation using ultrasonic. In this step, at least one optical recording memory media is placed in an ultrasonic environment, conditions including a frequency of 10-22 KHz, a power of 2 KW and a time period of 1 minute was used. Such that a reflective layer and a dye layer of the optical recording memory media is also separated, and a substrate layer of the optical recording memory media is also separated from a thin-film layer thereof. Lastly, is the recycling process where the separated substrate material and the thin-film material are individually recycled for further utilization.

Method for recycling optical disc, resin composition for forming film, and resin molded article was performed by Inagaki (2006). The method for recycling an optical disc includes crushing a recovered optical disc without further treatment into chips that used as a raw material of a resin composition for forming a film. The resin composition can be formed by injection molding to form a resin molded article. A film is formed on the surface of the resin molded article by plating or coating. Table 2.1 shows the use of various recovered optical discs instead of the new polycarbonate as a material provided molded articles having superior adhesiveness of plated and coated films. In this case, the resin composition produced by the method for recycling of invention has superior adhesiveness to the plated film or the coated film. Therefore, the resin composition and the resin molded article of the present invention are very useful for example, casing of electronic devices. Furthermore, according to the present invention, since the optical disc is recycled by crushing without further treatment, chemical used for, for example removing the films are not required. Consequently, waste such as waste liquid and waste water is not generated. This method contributes to global environmental protection in terms of effective utilization of resources and reducing waste. According to Inagaki (2006), by using this method, a used optical disc, i.e., a waste material can be effectively utilized as a raw material for a resin composition or a resin composition or a resin molded article for forming a plated film or a coated film.

	Ι	Base resin				Film	
Component A Component B			Adl	nesiveness			
No.	Resin	% by	Resin	% by	Film formation	Peel-	Peeling
Test		weight		weight		strength	(kgf)
1	PC	100	-	-	Copper electroplating	Unsuccessful plating	-
2	Recovered CD	100	-	-	Copper electroplating	0.9	-
3	Recovered DVD	100	-	-	Copper electroplating	1	-
4	PC	100	-	-	Chemical nickel plating	Unsuccessful plating	-
5	PC	100	-	-	Two-compenent acrylic-urethane coating	-	92/100
6	PC	70	ABS	30	Chemical nickel plating	0.8	-
7	Recovered CD	70	ABS	30	Chemical nickel plating	1.1	-
8	PC	70	ABS	30	Two-compenent acrylic-urethane coating	-	97/100
9	Recovered DVD	70	ABS	30	Two-compenent acrylic-urethane coating	-	100/100
10	PC	80	HIPS	20	Chemical nickel plating	0.7	-
11	Recovered DVD	80	HIPS	20	Chemical nickel plating	1.2	-
12	PC	80	HIPS	20	Two-compenent acrylic-urethane coating	-	90/100
13	Recovered CD	80	HIPS	20	Two-compenent acrylic-urethane coating	-	100/100

Table 2.1: Recycling optical disc, resin composition for forming film, and resin molded (Inagaki, 2006).

2.6.2 Chemical Recycling

Chemical recycling means the use of chemical processes to convert waste into useful products (Azapagic et al., 2003). Several processes can be included under the heading of chemical recycling, including gasification, hydrogenation, pyrolysis (sometimes referred to as thermolysis), and the use of waste polymer as a reducing agent in a blast furnace (Azapagic et al., 2003). Besides used chemical processes to convert its for others products, chemical stripping can also be used to remove coatings from polymer by this case compact disc. Chemical stripping involves the use of solvents or aggressive chemicals to remove coatings from polymer. Proprietary solutions based on alkaline cleaners and inorganic salt are often used on coatings stripped from engineering plastics which is polycarbonate layer, and its generally performed at elevated temperatures. Paint removal is initiated by submitting the parts to preliminary granulation; the paint film (metal layer) can then be chemically broken down in an alkaline solution at high temperatures. However, this technique requires the use of potentially harmful solvents and expensive chemical recovery equipment. Furthermore, there is the possibility of interactions between the solvent and the polymer (Giovanella et al, cited in Sheirs 1998).

U.S. Patent No. 7,105,632 B2 represents a process of recycle method for polycarbonate resin waste. The invention disclosed is a method for recycling polycarbonate using melt polycondensation equipment. By combining a polycarbonate oligomer and a polycarbonate waste component to form a polycarbonate polycondensation component, wherein the polycarbonate oligomer is the reaction product of a dihydroxy compound and a carbonate diester. The

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polycarbonate waste as subjecting component to one or both of a transesterification reaction and a polycondensation reaction, by this the polycarbonate waste component has an OH group concentration and comprises polycarbonate resin waste and by changing an OH group concentration of the polycarbonate waste component to suppress the formation of branched compound, wherein the OH group concentration is changed before subjecting the polycarbonate waste component to either of the transesterification reaction or the polycondensation reaction (Ikeda et al., 2006).

Figure 2.6 shows illustration of the recycle method for polycarbonate resin comprises adjusting the OH concentration of polycarbonate polycondensed products obtained in transesterification reaction during the progress of the polycondensation reaction when polycarbonate waste resin or/and polycarbonate oligomer is added to the transesterification equipment. In the transesterification reaction, the OH concentration of polycarbonate resin waste and polycarbonate oligomer, before polymerization is preferably about 200 to about 25000 part per million (ppm). If OH concentration is in above-mentioned range, the reaction will progress smoothly, and polycarbonate with outstanding hue, transparency, and less branched compound established.

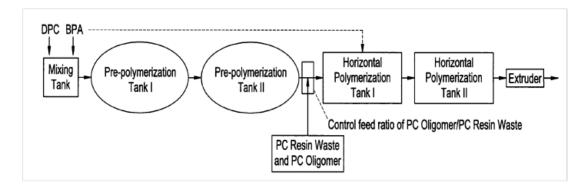


Figure 2.6: Illustrasion of the cycle method for polycarbonate resin comprises adjusting the OH concentration of polycarbonate (Ikeda et al., 2006).

The intrinsic viscosity of polycarbonate finally obtained by the disclosed method about 0.1 to about 1.0 deciliter per gram (dl/g). All intrinsic viscosity discussed herein is measured in methylene chloride of 0.5 dl/g concentration at 20 °C by using Ubbelohde viscometer. The above mentioned that polycondention method can be operated continuously or batch wise. The equipment for the above mentioned reaction can be tank type, tube type, or tower type.

Method of recycling disk recording medium and apparatus for recovering medium and apparatus for recovering metal reflective film had been performed by Komine et al. (2000), which has a layered structure including a substrate, a dye layer, a reflective film, and a protective layer, includes the steps of radiating ultrasonic waves onto the disc recording medium in a liquid medium such that the substrate and the reflective film are separated from each other, and bringing a solution which dissolves the layer into contact with the substrate such that a dye component is separated in order to recover the substrate.

Figure 2.7 shows the method of recycling disc recording media reported by Komine et al. (2000). Firstly, S1: Preferably notch at least the protective layer in the disk, S2: Applying ultrasonic vibration to the disc in a liquid medium, S3: The gold reflective layer (metal film or metal layer) separated from the substrate, S4: The gold reflective layer and the protective layer (protective film) separated from the substrate are obtained, then in S5: Incinerate the gold reflective layer and the protective film by heating at temperature that is nearly the melting point of the metal reflective layer, for example, at approximately 1200 °C, in order to remove the protective film from the gold reflective layer, and thus gold alone is obtained.

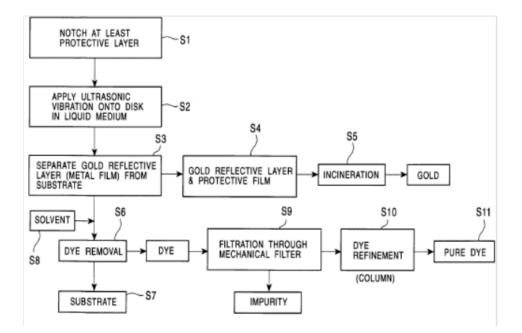


Figure 2.7: Schematic diagram of the method recycling disc for recovery metal reflective layer (Komine et al. 2000).

Proceed to steps S6, S7 and S8: Solvent was bring into contact with the substrate from which gold refelective layer has been separated, in order to separate the dye, and recover the resinous substrate by itself. In step S9: Filter the dye obtained in step S6 with, for example, a mechanical filter, to completely separate impurities from the dye. Then in Step 10: Refine the filtered dye in column, and the pure dye is obtained in step 11.

The solvents shown in Table 2.2 exhibits the solvent used to dissolve dyes and which do not affect substrates such as acetone and acetic acid 2-methoxyethyl ester. While, n-hexane and water shown in Table 2.3 do not dissolve dyes. Besides that Table 2.4 shows solutions having the composition dissolve dyes, excluding water/ethanol (10%). The statement above described that in step S7 the polycarbonate substrate alone can be separated, in step S5 pure gold alone can be recovered and the pure dye can be recovered in step 11. By recovering the resinous

substrate to be recycled, it can be recycled for example, as a material for substrates for disk recording media or like. Polycarbonate resin pallets was produced from the recovered substrate by palletized process.

Solvent	Dye	Effect on Substrates
	Dissolution	
Methanol	Observed	None
Ethanol	Observed	None
Isopropyl alcohol	Observed	None
N-buthyl alcohol	Observed	None
Ethylene-1,2-diol	Observed	None
Glycerin	Observed	None
2-methoxyethanol	Observed	None
2-ethoxyethanol	Observed	None
2-hydroxy-2-methyl-3-butanone	Observed	None
4-hydroxy-2-butanone	Observed	None
4-hydroxy-4-methyl-2-pentanone	Observed	None
Acetone	Observed	Observed
Acetic acid 2-methoxyethyl ester	Observed	Observed
Ethanol/water	Observed	None

Table 2.2: Solvents dissolve dyes and affect substrates excluding acetone and acetic acid2-methoxyethyl ester (Komine et al., 2000).

Table 2.3: Solutions composition dissolve dyes (Komine et al., 2000).

Solvent	Dye Dissolution	Effect on Substrates
N-hexane	None	None
Water	None	None

Table 2.4: Solvents dissolve dyes and effect n-hexane and water (Komine et al., 2000).

Solution Composition	Dye Dissolution	
Water/Ethanol	Partially observed	
Water/Ethanol	Observed	
Water/Ethanol	Observed	
Water/Ethanol	Observed	
Water/Ethanol	Observed	
Water/Methanol	Partially observed	
Water/Methanol	Observed	
Water/Methanol	Observed	
Water/Methanol	Observed	
Water/Methanol	Observed	
Water/Ethanol	None	

Sapienza et al. (2002) introduced a method for optical media demetallization process. This method was developed and patented by METSS Corporation. This method involves a process for removing the coating and any metals from polymeric substrates such as compact discs (CDs) by applying high shear conditions to fluidized particulates of the polymeric substrate optionally in the presence of an accelerant. There also provided a process for cleaning a polymeric substrate having a coating thereon, the process comprising the step of; (a) mechanically flaking or granulating the polymeric substrate into relatively small particles; (b) delivering the particles of polymeric substrate to a mechanical shearing apparatus; (c) fluidizing the polymeric substrate particles; (d) shearing the fluidized particles at a relatively high rate of shear, optionally in the presence of an accelerant selected from the group consisting of carboxylic acids, carboxylic acid esters and mixture thereof, to cleaned polymeric particles, wherein the coating on the polymeric particles includes a metallic substance and the process of the present invention substantially completely demetallizes the polymeric particles, i.e., down to 20 ppm of metal or less.

Nee (1994) developed a method for removing the lacquer and aluminium coatings from the compact discs. The polycarbonate (compact discs) immersed in an alkaline solution, the alkaline solution is heated to a temperature in the range from about 55°C to 104°C and mechanically agitating, the barrel is rotated about its horizontal axis at a speed of 60-100 rpm. The best result of the process of stripping the lacquer coating are achieved in large amount by applying ultrasonic energy to the alkaline solution at a frequency of 20-40 kHz at the sufficient energy density and for a sufficient time to dissolve the lacquer and the aluminium into the solution. The solution containing the dissolved lacquer and aluminium is decanted from the