Faculty of Resource Science and Technology

SYNTHESIS AND CHARACTERISATION OF SELECTED IONIC LIQUID AND ITS APPLICATION FOR CELLULOSE DISSOLUTION

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Bachelor of Science with Honours
(Resource Chemistry)
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UNIVERSITI MALAYSIA SARAWAK

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Synthesis and Characterisation of Selected Ionic Liquid and Its Application for Cellulose Dissolution

Mohammad Asrul Nizam Bin Matnin (31096)

A final project report submitted to fulfill the requirement for the degree of Bachelor of Science with Honours (Resource Chemistry)

Supervisor: Assoc. Prof. Dr. Pang Suh Cem

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2014

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Mohd Asrul Nizam Bin Matnin

Resource Chemistry Programme

Faculty of Resource Science and Technology

Universiti Malaysia Sarawak

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ABSTRACT

1-ally-3-methylimidazolium chloride (AMIMCl) is currently being used as a solvent for cellulose dissolution. AMIMCl is a green solvent and has excellent dissolution ability, wide liquid range and ease of recycling. Development of green technology nowadays promotes the use of cellulosic materials in the production of daily used products. Cellulose was chosen due to its properties as biodegradable, low cost and easily obtained. 33wt% of cellulose was successfully extracted from sugarcane bagasse through the alkali and nitric acid hydrolyzed treatments. Cellulose characterization was carried out by using Scanning Electron Microscope and Fourier Infrared Transform spectroscopy. Cellulose could be easily dissolved in the AMIMCl and could be regenerated by the water coagulation process. The slight different in absorption peaks of FTIR spectrum in original cellulose and the regenerated cellulose had proven that AMIMCl was a direct solvent. The AMIMCl was easily recycled due to its thermostable and nonvolatile nature.

Keywords: 1-ally-3-methylimidazolium chloride(AMIMCl), green solvent, regenerated cellulose, and recycled AMIMCl

ABSTRAK

1-ally-3-methilimidazolium chloride (AMIMCl) sekarang digunakan sebagai pelarut perungkai selulosa . Ianya merupakan pelarut yang baru, berfungsi dengan baik dan *AMIMCl* juga terbukti pelarut yg mesra alam kerana mempunyai ciri ciri seperti pelarut yang berkesan, julat cecair yang luas dan pelarut lansung. Pembangunan teknologi mesra alam sekitar telah mengalakkan pengunaan bahan selulosa sebagai bahan untuk produk yang digunakan di dalam kehidupan seharian. Selulosa dipilih kerana mempunyai ciri-ciri seperti mudah dilupuskan, kos yang rendah dan mudah untuk didapati Sebanyak 33% selulosa telah berjaya dipisahkan dari tebu berdasarkan reaksi alkali diikuti dengan natrium hidroksida proses. Pencirian selulosa dijalankan menerusi mikroskop imbasan electron dan *Fourier Transform Infrared spectroscopy (FTIR)*. Selulosa juga mudah untuk dilarutkan di dalam *AMIMCl* dan juga mudah untuk dihasilkan semula melalui kaedah proses pengumpulan air. Kurang perbezaan pada penyerapan di setiap puncak di dalam *FTIR* spektrum pada selulosa tulen dan selulosa yang dihasilkan semula menunjukkan bahawa *AMIMCl* adalah pelarut lansung. *AMIMCl* mudah untuk dikitar semula kerana ianya mempunyai ciri-ciri tahan panas dan tidak mudah meruap.

Kata Kunci: *1-ally-3-methylimidazolium chloride (AMIMCl)*, pelarut mesra alam, selulosa yang dihasilkan semula dan kitar semula *AMIMCl*.

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LIST OF ABBREVIATIONS

$AlCl_3$	Aluminium Chloride
AMIMCI	1-allyl-3-methylidazolium chloride
BMIMCI	1-butyl- 3-methylidazolium chloride
CS_2	Carbon disulphide
CNC	Cellulose nanocrystals
DMAc LiCl/N,N	Dimethylacetamide
DMSO	Paraformaldehyde
FTIR	Fouried Transform Infrared
H_2SO_3	Sulfurous acid
MS	Mass spectroscopy
NCC	Nanocrystalline Cellulose
NMMO	N-methylmorphine-N-oxide
NMR	Nuclear magnetic resonance
ILs	Ionic Liquids
SCB	Sugarcane Bagasse
SEM	Scanning electron microscope

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1.0 INTRODUCTION

The challenge of research on using the green chemistry principle on chemical industry are increasing significantly. The chemical industry has given a lot of contribution to society recently. However, with fully attention on just production sector leads to environment pollution. The impact from the chemical industry has not been limited to the environment, but also on human health. Intense energy consumption, CO_2 emissions, and resource depletion are just some of the challenges facing the chemical industry that threaten our environment. Due to this problem, the green chemistry principle that has been introduced by the Paul Anastas, is used by chemical companies (Sanderson,2011).

This study is conducted based from green chemistry principles such as use a renewable feedstock, less hazardous synthesis, prevent waste and atom economy (Anastas and Kirchhoff,2002). This study was focused on value-added utilization of agricultural waste. By reducing the agricultural waste could help in provided clean environment. Sugarcane bagasse is one of agricultural waste that contained the cellulose. Cellulose can be dissolved in several ionic liquids and then regenerated by addition of water (Laus et al.,2005). In this study, the focus was on synthesising and characterization of an ionic liquid, which is the best solvent to dissolve the cellulose derived from the sugarcane bagasse. Cellulose has been chosen, due to its biodegradability, cheap and abundance on earth.

Ionic liquid has been recently recognised as the most efficient solvent for dissolving cellulose. The existence of problems such as low efficiency and environmental pollution in chemical industry leads to ionic liquid synthesis. The objectives of this study are to synthesis a selected ionic liquid, characterized it and evaluate the dissolution of cellulose in the ionic liquid. The selected ionic liquid is 1-allyl-3-methylimidazolium chloride (AMIMCl). Cellulose sample can be dissolved without derivation in this type of ionic liquid (Zhu et al.,2006). AMIMCl also could be reused after separating the regenerated cellulose. Due to its reusability, less viscosity, and less toxicity AMIMCl is considered as a green solvent (Zhang *et al.*,2005). Ionic Liquids may be easily modified through changing the structure of cations or anions, which can broaden their application fields (Nathany, n.d.). The synthesized ionic liquid is characterized using NMR and FTIR.

For the sample preparation, the chosen sample was sugarcane bagasse. One of objectives in this study was to dissolve the cellulose in the AMIMCl. The cellulose was retrieved first from the sugarcane bagasse. The cellulose samples without any pre-treatment also can be readily dissolved in AMIMCl (Nanthany,n.d.). Raw sugarcane bagasse powder and cellulose were characterised by the SEM and FTIR. The characterization of prepared sample can be run on the SEM and FTIR.

The focus of this study is to determine the effectiveness of ionic liquid for cellulose dissolution. Temperature is the vital factor in determining the effectiveness of the ionic liquid. For industrial, less temperature used could help in reducing the cost of production. Hence, continuous development of ionic liquid in its dissolution capabilities is needed to promote better solvent which produce less waste and cost for industrial application purposes.

Objectives of this study are:

- To synthesis and characterize 1-allyl-3-methylimidazolium chloride ionic liquid
- To isolate and characterize the cellulose from sugarcane bagasse
- To evaluate the dissolution of cellulose in the ionic liquid and regeneration of cellulose

2.0 LITERATURE REVIEW

2.1 Sugarcane Bagasse

Sugarcane is largely grown in tropical and subtropical places where the wet and dry season is alternate such as Brazil, Australia, India, South Africa, Cuba, Peru and Mexico (Ripoli *et al.*, 2000). Malaysia is also one of the countries that produce sugarcane due to its hot climate and humid suitable for the sugarcane production. The sugarcane basically consists of stem and straw. Stem is the part normally associated with sugarcane. Sugarcane straw is composed by fresh leaves, dry leaves and tops available before harvesting (Canilha *et al.*,2013). Bagasses are a fibrous residue obtained from sugarcane stalks after it is crushed to obtained juice used for sugar and ethanol production (Mothe and Miranda, 2009). Sugarcane bagasse structure is made up from lignocellulose materials. Lignocellulosic material is consisting of several components such as cellulose, hemicellulose and lignin.

2.1.1. Lignin

Lignin is a highly complex, three-dimensional polymers of three different phenyl-propane units, which is p-coumaryl, sinapyl and coniferyl alcohols (Amen-Chen *et al.*, 2001). They are bound together by aryl-aryl, alkyl-aryl and alkyl-alkyl ether bonds. Lignin is embedded in the cellulose and hemicellulose structure which helps to strengthen the cell walls rigidity. Moreover, lignin helps as protection against bacterial and fungi degradation. Lignin contributes around 20% to 30% of sugarcane bagasse mass. After cellulose, it is the most abundant renewable carbon source on Earth. Between 40 and 50 million tons per annum are produced worldwide as a mostly non commercialized waste product. Lignin made up from three monomer that is p-hydroxyphenyl(H), guaiacyl (s) and Syringyl which is shown in Figure 2.1 (Hon and Shiraiski, 1991).

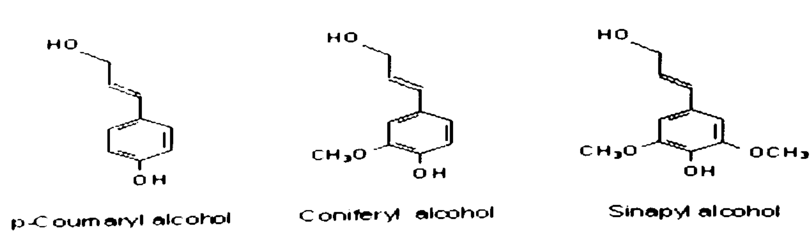

Figure 2.1 : Monomers of lignin(Salameh, 2009).

Those monomers are product from the phenylalanine biosynthesis. The lignin is the result from the polymerization of those monomers and the structure of lignin is shown in Figure 2.2. The chemical structure of lignin is irregular in the sense that the structural elements are not linked to each other in any systematic order (Salameh, 2009).Chemical bonds have been reported between lignin and hemicellulose and even cellulose. Lignin are extremely resistant to chemical and enzymatic degradation(Palmquist and Hahn, 2000). This is due to its properties as highly hydrophobic and poses the heterogeneous linkage.

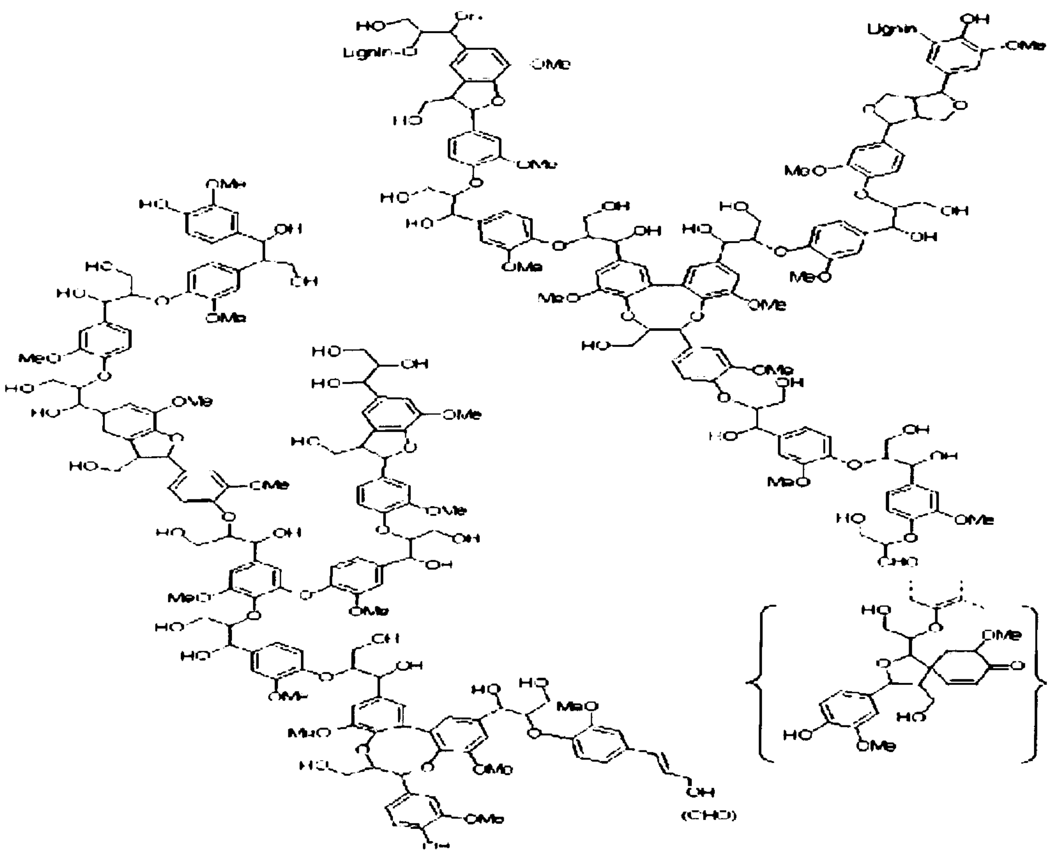

Figure 2.2 : Structure of Lignin (Salameh, 2009).

2.1.2. Hemicellulose

Hemicellulose is second main component that exist in the lignocellulose material. Mosteur (2005) stated that, Hemicellulose is heterogeneous group if polysaccharides and consist of 20 – 25% of biomass on dry basis. The hemicelluloses rank next to cellulose as the most abundant natural carbohydrate polymer in biosphere. The structure of this compound could be grouped into xyloglucan, xylans,mannans and glucomannans. These are usually classified according to the sugar residue present. Some of polysaccharides could be grouped into hemicellulose (Mosteur *et al.*, 2005). Furthermore, hemicellulose structures consist of hexoses and pentoses with addition to uronic adids and acetyl groups with the exact composition depends on the type of hemicellulose. The hemicellulose structure is different between pant types and tissue types (Scheller and Ulvskov, 2010). The glucuronoarabinoxylan structure is most likely to be found in the sugarcane bagasse (Hettinga *et al.*,2009). The structure of glucuronoarabinoxylan is shown in Figure 2.3. structure of hemicellulose is shown in figure 2.4.

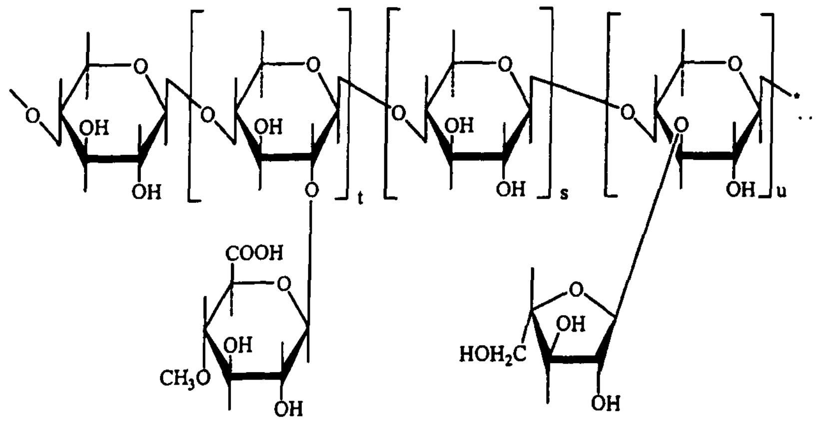

Figure 2.3 : The Glucuronoarabinoxylan structure(Rehders &Sheehan,2004)

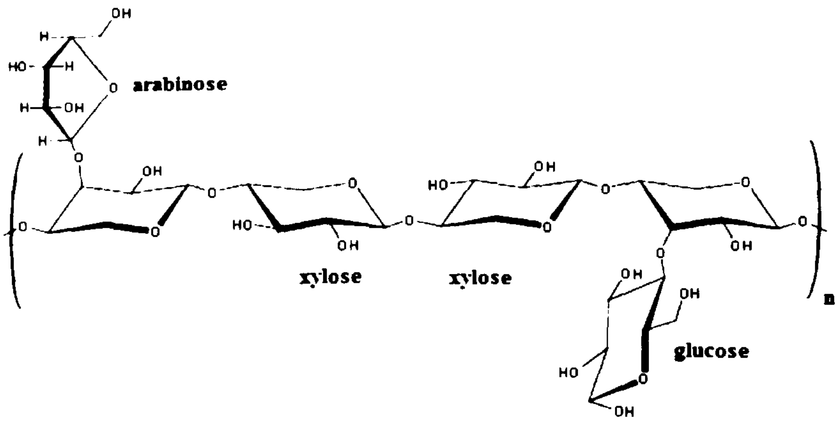

Figure 2.4: Hemicellulose Structure(Hong,2013)

The applications of material from hemicelluloses that have been identified include packaging films, food coatings, cationic biopolymers, hydrogels and biomedical uses. Films consisting of glucomannan have demonstrated good gas barrier properties, and therefore have potential for use as components in barrier coating for cardboard used in food packaging (Danielsson, 2012). The cross linking structure of glucomannan helps in better water absorbing properties.

2.2. Cellulose

Cellulose is the most bio renewable material, low cost and abundantly amount on earth. Derivative products have many important applications in the fiber, paper, membrane, polymer, and paints industries (Swatloski *et al.*,2002). Cellulose basically extracted from cell wall of plant and wood. Cellulose, hemi-cellulose and lignin are the main components of plants such as wood and leaves which build the spongy three-dimension structure(Vogli *et al.*,2002). Sugarcane bagasse is one sample that builds up from cellulose structure. Sugarcane bagasse contain 42% of cellulose, 27% of hemicellulose and 30% of lignin(Karatzos, 2011).Abundant exploitation of cellulose as a bio renewable feedstock has been delayed by the lack of a suitable solvent that can be used in the chemical processing of cellulose. The materials based on cellulose and its derivatives have been used for more than 150 years in a wide variety of applications, such as food, paper production, biomaterials and pharmaceuticals (Coffey *et al.*, 1995).The growing willingness to develop new cellulosic materials results from the fact that cellulose is a renewable resource, although many of the technologies currently used in cellulose processing are decidedly non-friendly environment (Johnson,1985).

Synthetic polymer industry has brought glory to modern life. However, due to non-biodegradable properties, plastics have become major threat for our environment. Hard to recycle and lack of waste management facilities lead to various effect on environment such as river pollution and marine life degradation. The new regulations based on green technology industry have been set up by government to prevent or minimised the pollution from occurred. The consequence is making the cellulose as a renewable feedstock material. Due to limitation on volatility, toxicity and cost make these solvents are not ideal enough for the dissolution of cellulose. Ionic liquid is considered as the solution of this problem. Cellulose, whether it is refined or natural, can be dissolved, without derivation, in some hydrophilic ionic liquids such as BMIMCl and AMIMCl (Swatloski *et al.*,2002).

Sugarcane bagasse is the large amount of waste that produced from the sugar industries. Around 1 ton of sugarcane bagasse generates 280kg of bagasse as the by-product after the sugar extraction from sugarcane (Suhardy *et al.*,n.d.). Large amount of waste bagasse generated is totally causing a hard waste management for the sugar industries. This problem could create as a solution for huge amount of world paper consumption every year. Due to shortage raw materials for pulping, the sugarcane bagasse waste can be recycled to reuse as pulp production (Suhardy *et al.*,n.d.). The bagasse also can be used as a raw material for cultivation of microorganisms for the production of value-added products such as xylitol and ethanol. Due to these the bagasse is not only a sub-product of sugar industry, but it is a co-product with high added-value (Pandey *et al.*,2000).

2.2.1. Cellulose extraction process

Dissolving pulp is a high-grade cellulose pulp, with low contents of hemicellulose, lignin and resin. There are two major types of chemical pulping known in the art Kraft pulping and Sulfite pulping(Salameh, 2009). In Kraft pulping process wood is treated with a solution of sodium hydroxide and sodium sulfide at elevated temperature and pressure. The output products are separated pulp and solution that contains degraded lignin and hemicellulsose. However, Kraft pulping process bring negative feedback as reduce yield and environmental harm due to side production of malodorous gases (Salameh,2009). For Sulfite process, this process relies on acid solution of sulfurous acid (H_2SO_3) and bisulfate ion to break the bond between cellulose and lignin. The pulping process separates the cellulose from the lignin and hemicellulose leaving it in a fibrous form that is purified, dried, and shipped in large rolls.13 Cellulose produces from pulping usually contain small amount of lignin which cause it to have some brown discoloration. Cellulose then undergoes bleacing and alkali extraction process for purification. Another purpose of bleacing process are to make pulp resistant to aging and improve cleanliness of pulp (Salameh, 2009). Christoffersson (2005) explained that, the clean pulp is suitable as a raw material for different kinds of cellulose products, such as staple fibres, films and derivatives. Making cellulose fiber by the dissolving pulp method currently requires the use of big volumes of various chemical additives.

2.2.2. Recent application of cellulose and future potential

Nanoscience and nanotechnology has been rapidly being discussed by scientist or government. In this case the exploitation of Nanocrystalline Cellulose will become a connector between nanoscience and natural resource products (Peng, Dhar, Liu and Tam, 2011). The main application of NCC is for making the polymeric matrix stronger in nanocomposite materials. In addition, cellulose nanocrystals (CNC) that derived from wood pulp extract can be used to create the strongest materials. It even defeats the tough materials such as Kevlar and carbon fiber, the material is lightweight, cheap, and abundant. Found in on products such as sawdust and wood chips, CNC could be used to create materials with applications in military defense, engineering, medicine, and consumer products (Matus, 2012).Favier *et al.* (1995) was the first to report that, the use of NCC as reinforcing fillers in poly(styreneco-butyl acrylate) (poly(S-co-BuA))-based nanocomposites. Chemical functionalization of NCC improves its dispersability in organic solvents lead to futher future applications. Due to less toxicity properties, NCC capable of being used as carriers in the targeted delivery of therapeutic or the simplest known as drug delivery (Roman *et al.*,2010). The very large surface area and negative charge of NCC suggests that large amounts of drugs might be bound to the surface of this material with the potential for high payloads and optimal control of dosing. The cellulose material has excellent compaction properties when blended with other pharmaceutical excipients so that drug-loaded tablets form a dense matrices suitable for the oral administration of drugs. Shi *et al.* (2003) used microcrystalline cellulose as host beads, where drug nanoparticles dispersion was spray coated onto them, and they examined the morphology using scanning electron and AFM. NCC particles were incorporated into hydrogels based on cyclodextrin/polymer inclusion (Zhang *et al.*, 2010), and found that the new nanocomposite hydrogels can be used as a controlled delivery vehicle. A novel nanocomposite consisting of NCC and gold nanopaticle was recently investigated as a matrix for enzyme/protein immobilisation (Mahmoud *et al.*, 2009). The NCC has already been used as a template in the synthesis of mesoporous materials due to its attractive properties. Porous titania with anatase structure was prepared using NCC as a template. Titania material have high specific surface area, and being used as catalysis or catalyst support and photovoltaics. Some metal nanoparticles synthesised on NCC surface via a reduction method, such as Ni nanoparticles (Shin *et al.*,2007) and Au–Ag alloy nanoparticles (Shin *et al.*, 2008). For this processes NCC serves as a matrix and as a stabilising template. Crystallinity of NCC was maintained to produce stable dispersion nanoparticles on NCC surface (Shin *et al.*, 2008). These reducing processes could be recognised as eco-friendly processes because use of NCC and then applied to the preparation of transition metal nanoparticles in which have high oxidising property without additional reducing agents (Peng *et al.*,2011). NCC have future potential as may be used in security paper (Revol *et al.*, 1997, 1998), due to its solidified liquid crystals property. NCC also may be used in lithium battery products as a mechanical reinforcing agent for low-thickness polymer electrolytes. NCC just been explored on nanocomposites field for current application.

2.3. Solvents used for dissolving cellulose

Due to their stiff molecules and close chain packaging via numerous intermolecular and intramolecular hydrogen bonds, it's difficult to dissolve cellulose in water and organic solvent(Zhang *et al.*, 2005).Since green regulation implemented, scientists have tried to for search proper solvents to dissolve the cellulose. According to Zhang *et al.*(2005), since 1970s, novel solvents for celllose have been sought to replace the viscous system. However, only few types of solvents and methods can dissolve the cellulose before the introduction of ionic liquid. The solvents are LiCl/N,N-dimethylacetamide(DMAc), DMSO/paraformaldehyde, N-methylmorphine-N-oxide(NMMO) and cupriethylenediamine. Methods used are Visco Method, Lyocell and alkali mixture.

2.3.1. Visco method

It does involve the combination of sodium hydroxide (NaOH) and Carbon disulphide (CS_2). The process was invented in 1982 and has been used for period of time(Heinze and Koschella, 2005). This method could be considered as non-derivatizing solvent, which dissolve cellulose by disruption of intermolecular hydrogen bond, without chemically altering the monomeric unit or the polymeric linkages (Keskar,2011). Heinze and Koschella (2005) also mentioned that, pre-treatment cellulose with NaOH, Xanthion with carbon disulphide, leads to derivative cellulose soluble in solvent.

2.3.2. Lyocell

Lyocell method involved N-methymorphine-N-oxide (NMMO) as a direct solvent. It's has ability of dissolving high concentration of cellulose without altering the chemical properties of the cellulose chain as could be recycled back. NMMO solvent system has clear advantages in its non-derivatizing since many process steps may be left out(Olsson and Westman, 2013). Highly polar N-O group of NMMO can from hydrogen bond with cellulose molecules,thus disrupt intra and inter molecular hydrogen bond of cellulose itself and leads to the dissolution(Olsson and Westman,2013).

2.3.3. DMAc/LiCl

Dimethyl acetamide/lithium chloride mixture was the most frequent used solvents systems for cellulose and other polysaccharides when it comes to homogenous modification. Striegel (1997) described that, the DMAc/LiCl has the ability to dissolve the cellulose with higher molecular weight such as cotton. Moreover, the solvent system was colourless and compatible with GPC column(McCormick, 1985). The mechanism of the solvent system worked is shown in Figure 2.5.

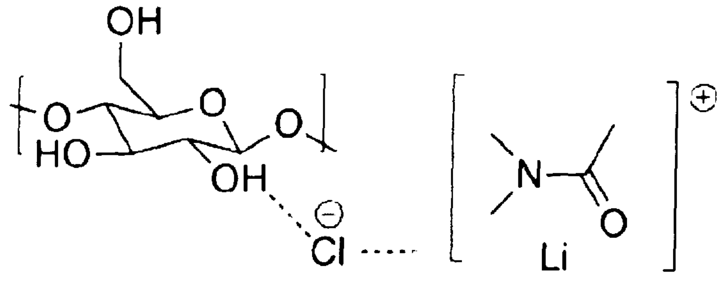

Figure 2.5 : Dissolution mechanism of DMAc/ LiCl (Olsson and Westman, 2013)

Striegel(1997) mentioned that, the hydroxyl groups of cellulose interact with a lithium-DMAc macrocation via hydrogen bonding bridged with chloride anion. As the lithium cation interacts with carbonyl oxygen, there was no cellulose bound water can be present. As a result, the solvent must be carried out in the laboratory with dried condition(Striegel, 1997). The solvent also capable in synthesized the regenerated cellulose. It's also suitable for homogeneous derivatizing of cellulose(Oliveira and Glasser, 1996). However, the solvent system also posed some disadvantages. The cellulose solution in DMAc/LiCl had fail to remain inert while under thermal stress(Bikova and Treimanis, 2002). Moreover, this solvents have some limitation in volatility, high cost and difficult to recovery(Terbojevich *et al.*,1985).

2.3.4. DMSO/TBAF

It's new and varies powerful solvent for cellulose dissolution (Olsson and Westman,2013). It's efficient and could dissolve the cellulose with a DP of up to 1200 within an hour at $60^{O}C$ (Heinze and Kohler,2010). The solvent system had been most used as for analytical purposes for homogeneous reactions for chemical modification of cellulose(Bikova and Treimanis, 2002). Moreover, water content in DMSO/TBAF/Cellulose solution may play a crucial role as the TBAF is hygroscopic salt. Anhydrous TBAF is unstable but the TBAF with water content up to trihydrate are excellent cellulose solvent(Hussain et al.,2004).

2.4. Ionic liquid

The term ionic liquids have been used to describe the salt that melt below 100°C. At room temperature, ionic liquid with melting point of room temperature will undergo melting process (Shamsuri and Abdullah, 2010). Ionic liquids consist of cations and anions, and are commonly understood as green solvents (Kärkkäinen,2007). Ionic liquid considers as the green solvent compare to others due to its wide liquid range, excellent dissolution ability, free from water vapour effect, non-flammable, thermally stable and easy to recycle. Ionic Liquids have been utilized for shaping of cellulose into fibers, films, sponges and beads (Matus, 2012).

Due of cellulose stiff molecules and close chain packing via numerous intermolecular and intramolecular hydrogen bonds, it is extremely difficult to dissolve cellulose in water and most common organic solvents(Zhang *et al.*,2005). That is the reason of ionic liquid synthesized. Using ionic liquid as a solvent for cellulose also implemented the 12 principles of green chemistry. High yield percentage of ionic liquid implemented the green chemistry principle of atom economy (Anastas and Kirchhoff, 2002). The liquid nature of the catalytic phase eliminates the heat and mass transfer problems frequently encountered with heterogeneous Friedel–Crafts-catalysts and produce high yield reaction. Moreover, the preparation of ionic liquid is quite easy and produced no by-products. The high concentration of highly acidic species that can be immobilized in ionic liquids and that leads to a highly reactive catalytic phase allowing for fast reactions under mild conditions. Consequently there has been much interest in finding non-volatile solvents as replacements. Unlike other solvent, Ionic Liquids do not vaporise into the air, effectively eliminating one of the major routes of environmental contamination, making its operations safer and environmentally acceptable (Shamsuri and Abdullah,2010). Other than that, non-volatility properties results in low impact on the environment and human health, and they are recognised as solvents for "green chemistry"(Laus *et al.*,2005).

It has been reported that certain ionic liquids (ILs), molten organic salts with a melting point below 100 °C, can dissolve high amounts of cellulose about 10–20% within short time and without pre-treatment process(Gericke *et al.*, 2012) . Since that time, an increasing number of scientific papers, patents, review articles, and conference abstracts about the use of ILs as cellulose solvents have been published and ionic liquid has become favourite research topic. Moreover, Ionic Liquids might overcome other cellulose solvents weakness, used so far for the homogeneous derivatization, regarding recyclability and cost efficiency. They are intensively studied for the production of commercial bulk derivatives, such as cellulose esters, acetates, propionates, butyrates, and mixed esters (Gericke et al., 2012). ILs have a great variety of chemical and physical properties that can be tuned with cations and anions(Anthony et al.2003).

Ionic Liquids are likewise hard to handle. They show certain weakness that need to be addressed before Ionic Liquids can be applied for the dissolution and processing of cellulose on a commercial scale (Gericke *et al.*,2012). Characterization of ionic liquids was done by determining the thermal stability, the melting point and the crystal structure of each solid ionic liquid. The determination of the liquid range of ionic liquid is necessary in order to know the temperature limits for each ionic liquid(Yang *et al.*,2004).

2.4.1. Synthesis concept for ionic liquid

As mentioned by Wasserscheid and Keim(2000), the principal synthetic concepts for Ionic Liquids which involve quaternisation reactions to generate the cation. Then, followed by anion metathesis, either by the addition of metal salts to precipitate the undesired anion or possibly use of silver salts, the addition of strong Brönsted acids to release the unwanted anion as the volatile corresponding acid, use of ion exchange resins, and lastly, treatment with Lewis acids to form complex anions such as chloroaluminates. Laus *et al.(*2005) reported of efficient synthetic procedures for imidazolium salts were described .

Karkkainen(2007) reported preparation of ionic liquids, is quite straightforward. The first step, and sometimes the only step, is a quaternization reaction that takes a rather long time to accomplish, even when heated with an oil bath(Bourbigou *et al.*, 2003). An attempt has been made to decrease the reaction time by carrying out the synthesis of the ionic liquids in an ultrasonic atmosphere (Huddleston, *et al.*,1998). In the beginning of the 21st century, microwave activation made a breakthrough by shortening the reaction times considerably (Lindström *et al.*,2001). It was decided to study the preparation of ionic liquids under microwave irradiation. The preparations were done according to the principles of green chemistry whenever possible. The aim was to do the reactions atom-efficiently, without a solvent, in an energy- and time-saving manner.

2.4.2. Characterization of ionic liquid

Karkkainen(2007) explained that the production of pure ionic liquids is very important, since impurities have a strong influence on their physical properties and stability. The ionic liquids were characterized with NMR, FTIR and elemental analysis. Their thermal