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CHEMISTRY CONCEPTS IN NANOCELLULOSE ISOLATION AND ITS POTENTIAL FOR GREEN CHEMISTRY LEARNING

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Abstract

As a plant that is easy to grow, banana tree (*Musa spp.*) is an abundant tree in tropical countries. Banana tree is only able to bear fruit once in its lifetime and the waste of banana pseudo-stems are also existed in a large number. The cellulose content in the banana pseudo-stems can be utilized as a source of raw material for the manufacture of nanocellulose. This study aims to develop the new chemistry learning context which introduces the process of nanocellulose isolation from banana pseudo-stem through a dissolve method using ionic liquid. The used research method was the qualitative method with content analysis using the content validation format as the research instrument. The result of this research is based on the content validation result where in the nanocellulose isolation process, there are several school chemistry concepts that can be learned by high school students as such as water evaporation, hydrolysis process, factors influencing reaction rate, intermolecular interaction, and colloid and most of the principles of green chemistry can be introduced to high school students as such as Prevention, Atom Economy, Less Hazardous Chemical Syntheses, Designing Safer Chemicals, Safer Solvents and Auxilliaries, Design for Energy Efficiency, Use of Renewable Feedstocks, Reduce Derivates, Design for Degradation, Real-time Analysis for Pollution Prevention, and Inherently Safer Chemistry for Accident Prevention. Thus, the conclusion of this research is that the nanocellulose context has a potential to be integrated into school chemistry learning. Beside that, it also has a potential to introduce the principles of green chemistry except catalysis principle.

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INTRODUCTION

The need for learning science and principles of green chemistry through technologies is important in Indonesia because there are development and transformation of the education sector at international level. Indonesian students have participated in PISA held by Organisation for Economic Co-operation and Development, they obtained the scientific literacy skill's ranking at 62 from 70 participants with the average score in science is 403, lower than all participants' average score in science (OECD, 2016). Based on that, the development of new curriculum is needed. The developed curriculum is Kurikulum 2013. Kurikulum 2013 has a purpose to create the students into human resources who can compete with human resources around the world (Kemendikbud, 2014). Beside on developing the new curriculum, learning context that used in learning science and principles of green chemistry must be based on technologies which are relevant in life nowaday. One of the relevant contexts is nanotechnology which has been using in the various technology fields.

Nanotechnology is the study of materials that have dimention about 1 until 100 nanometers (Xiao et al., 2015). One of materials which has many benefits in technology such as electronic devices and optical devices, and renewable medicine is nanocellulose (Salas et al., 2014). Nanocellulose is cellulose that has diameter range between 1 until 100 nanometers and has length about tens to thousands nanometers, has relatively high aspect ratio, and has a high crystalline structure properties (Xiao et al., 2015; Effendi et al., 2015; Wei et al., 2014). Nanocellulose has increase in popularity in last decade because it has unique properties such as low density, high tensile strength, high Young modulus, easy modificated-surface properties, and has biodegradable property (Islam et al., 2014; Xiao et al., 2015; Wei et al., 2014).

Nanocellulose has been applicated in various industries such as thickener in cosmetics, stabilizer in food industries, quality enhancer in paper and furniture, as composite material for constructions and vehicles, optical devices, stabilizer in multyphase systems, and also as reinforcing material in nanocomposite using the principles of colloidal science (Salas et al., 2014). Nanocellulose is a material that has usability potential as a water filtration membrane (Karim et al., 2016; Carpenter et al., 2015) It is based on the result of experiment conducted by Yu *et al.* (2013) that membrane of cellulose nanocrystalline (one kind of nanocellulose) which the hydroxyl groups of its chain have been reacted with succinic acid significantly can increase binding efficiency of Pb^{2+} and Cd^{2+} metal ions in solutions (Carpenter et al., 2015). Nanocellulose is also a material that has usability potential in biomedical application such as in medical implants, soft-tissue implants and cartilage replacement, drug delivery, wound healing, tissue engineering, antibacterial activity, and cardiovascular (Jorfi & Foster, 2015).

Generally, nanocellulose is obtained from various sources such as sea animals and natural fibers (Li et al., 2012) like wood chips (Rebouillat & Pla, 2013), oil palm empty fruit bunch (Lani et al., 2014), and cotton (Wang et al., 2013), but this study wants to raise one of thematerials that easy to be found in Indonesia which can be used as nanocellulose resource as a context for learning of scienceand the principles of green chemistry, that is banana plants. This is needed because Indonesian students are expected to be able to learn the science and principles of green chemistry through the contextof nanotechnology especially nanocellulose from materials that can be found in their daily lives. So far, there has been no research on the development of the learning context through the context of nanocellulose. There are several studies on the context of nanotechnology in education that have been carried out, such as a research on curriculum development that inserts the context of nanoscience and nanotechnology (NST) to enhance students' scientific literacy skills (Laherto, 2012), a survey of science teachers' perception on nanotechnology teaching and professional development (Lin et al., 2015), and a research that aims to describe advancements that have been made in science and nanotechnology, and challenges that educate students and the public about critical nanoscience concepts through reviews of current research on nanotechnology education including curricula (Jones et al, 2013).

Banana (*Musa spp.*) is one kind of plant that almost grows across the world. Each year, about 120-150 million tons of bananas are grown in the world, and this makes it as fourth most important food product in the world (Reddy & Yang, 2015). As in Indonesia, it is the most abundant plant that grows in plains of indonesia. There are 30 kinds of bananas that grow in Indonesia such as Ambon, Raja, Kepok, and many more the kinds of banana (Valmayor et al., 1991). Initially, banana is discovered in humid tropics, but it can adapt in various climates. Currently, banana has been planted in 150 countries with an area of 4.84 hectares that has produced 95.6 billion tons of bananas (Singh et al., 2011).

Banana plants can breed in generative and vegetative ways. Breeding in generative way (seed) usually occurs on wild bananas, while breeding in vegetative way (shoot growth) is for cultivation bananas, and it is the only one way for preserve them (Singh et al., 2011). During the growth process, plants that grow with vegetative way require to eliminate the old part of the plants (in this case is cultivation bananas) with reason for rejuvenation (Sandor, 2007). Felling of banana plants that have been harvested will produce banana pseudo-stem waste with a large number.

Many efforts have been made for resolving banana pseudo-stem waste problems, one of them is making paper from these waste fibers (Singh & Bandyopadhyay, 2013) as an alternative material to replace wood. Banana pseudo-stem contains one of the same materials present in wood. This material is cellulose. Cellulose (figure 1) is a linear homopolysaccaride, unbranched chain, consists of 10000 to 15000 D-glucose units (Sawatdeenarunat et al., 2015; Xu et al., 2013).

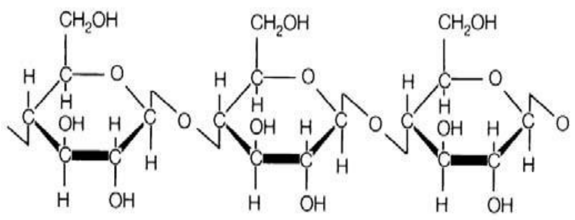

Figure 1. Structure of cellulose

About 72.71 percent of holocellulose (the mixture of cellulose and hemicellulose) is contained in banana pseudo-stem, and holocellulose is contained about 39 percent of cellulose (Li et al., 2010), therefore, banana pseudo-stem can be used as a raw material for nanocellulose isolation.

In nanocellulose isolation process, there are several methods that can be used, one of them is ionic liquids method (Effendi et al., 2015; Xiao et al., 2015; Isik et al., 2014). Ionic liquid is a salt with [R]X formula that has melting point below 100°C, this definition is used because ionic liquid is liquid at room temperature (Singh & Kumar, 2008; Khupse & Kumar, 2010; Curnow, 2012, Matic & Scrosati, 2013). Mostly ionic liquids consist of organic cation $[R]^+$ and inorganic or organic anion X^- (Khupse & Kumar, 2010; McGarrigle et al., 2011; Curnow, 2012; MacFarlane et al., 2014). In nanocellulose isolation from natural materials, ionic liquid method is better than other methods like acid method because the fabulous characteristics from ionic liquid such as environmentally friendly, high chemical and thermal stability, low vapour pressure, nonflammable, and can be recycled, therefore ionic liquids are usually called as "green solvents" (Xiao et al., 2015; Rauber et al., 2017).

How nanocellulose can be obtained by using ionic liquids allows for the chemistry concepts that can be learned by high school students, so the process of nanocellulose isolation from banana pseudo-stem waste using ionic liquid should be demonstrated in chemistry learning at high school. Demonstration of nanocellulose isolation from banana pseudo-stem by using ionic liquid as a solvent is also a manifestation of environmental concern. Chemical process in nanocellulose isolation using conventional chemicals that can bring negative effects on the environment can be avoided by using non-pollutant alternative materials such as ionic liquids, and it is a representation of "green chemistry" (KARAGÖLGE&GÜR, 2016).

Green chemisty was originally an idea in academic studies that later evolved into a common practice to be undertaken, supported by industrial and academic fields (Hjeresen et al., 2000). Green chemistry is the design of chemical products and processes that can reduce the negative impacts to human health and the environment (Hjeresen et al., 2000). Green chemistry is also defined as the design of chemical products and processes for reducing and eliminating the use of hazardous substances (Anastas & Eghbali, 2010). There are twelve principles of green chemistry:

1) **Prevention**. It is better to prevent waste then to treat or clean up waste after it has been created.
2) **Atom Economy**. Synthetic methods should be designed to maximize the incorporation of all materials used in the process into the final product.
3) **Less Hazardous Chemical Syntheses**. Wherever practicable, synthetic methods should be designed to use and generate

substances that possess little or no toxicity to human health and the environment.

4) **Designing Safer chemicals**. Chemical products should be designed to effect their desired function while minimizing their toxicity.
5) **Safer Solvents and Auxiliaries**. The use of auxiliary substances (solvents, separation agents, etc.) should be made unnecessary wherever possible and innocuous when used.
6) **Design for Energy Efficiency**. Energy requirements of chemical processes should be recognized for their environmental and economic impacts and should be minimized. If possible, synthetic methods should be conducted at ambient temperature and pressure.
7) **Use of renewable Feedstocks**. A raw material or feedstock should be renewable rather than depleting whenever technically and economically practicable.
8) **Reduce Derivates**. Unnecessary derivatization (use of blocking groups, protection/deprotection, temporary modification of physical/chemical processes) should be minimized or avoided if possible, because such steps require additional reagents and can generate waste.
9) **Catalysis**. Catalytic reagents (as selective as possible) are superior to stoichiometric reagents
10) **Design for Degradation**. Chemical products should be designed so that at the end of their function they break down into innocuous degradation products and do not persist in the environment.
11) **Real-Time Analysis for Pollution Prevention**. Analytical methodologies need to be further developed to allow for real-time, in-process monitoring and control prior to the formation of hazardous substances.
12) **Inherently Safer Chemistry for Accident Prevention**. Substances and the form of a substance used in a chemical process should be chosen to minimize the potential for chemical accidents, including releases, explosions, and fires (Anastas & Eghbali, 2010).

Based on those, there is a relationship between green chemistry and practicum activities in chemistry learning, especially in the use of chemicals. Most chemicals used in laboratories are substances that can damage health and potentially damage the environment, so the principles of green chemistry are important to be inserted in chemistry curriculum to make chemistry inherently green (Kitchens et al., 2006), especially in the practicum. As an example at Westminster College, the principles of green chemistry are inserted in general chemistry, organic chemistry, and organic laboratory activity (Kennedy, 2015). The experiment of nanocellulose isolation from banana pseudo-stem waste using ionic liquid has a potential to be introduced to high school students as a green chemistry based-chemistry concepts learning. Therefore, this research aims to develop the new learning context for learning chemistry concepts and the principles of green chemistry through nanocellulose isolation process.

METHODS

The used research method was the qualitative method with content analysis technique. One reason why a researcher might want to do a content analysis is to obtain descriptive information about a topic. Content analysis is a very useful way to obtain information that describes the issue or topic (Fraenkel et al., 2012). This research has done with identifying the chemistry concepts and principles of green chemistry using the validation sheets for the conformity of nanocellulose and ionic liquid contexts with chemistry concepts, learning objectives, and principles of green chemistry in the demonstration of nanocellulose isolation from banana pseudo-stem waste using cis-oleilimidazolinium acetate ionic liquid. The development of the practicum kit of nanocellulose isolation has done through two steps i.e. preparation of banana pseudo-stem waste and nanocellulose isolation from banana pseudo-stem waste using cis-oleilimidazolinium acetate and its testing. Cis-oleilimidazolinium acetate ionic liquid which is used in this experiment is a reaction result from imidazoline, cis-oleic acid, and $Pb(CH_3COO)_2$.

Preparation of banana pseudo-stem waste

Banana pseudo-stem waste is cutted into small pieces, and the pieces of banana pseudo-stem waste are dried in the sun for a week until they are dry. After that, the pieces of dried banana pseudo-stem waste are bleached with bleaching agent that consists of $NaClO_2$ 2,5% b/v, acetic acid 4% v/v, and aquades by volume ratio 1:1:2. Bleaching process is performed by comparison between dried banana pseudo-stem waste (in gram) and volume of blaching agent (in mL) of 0,1 : 40 for 2 hours. After that, as a result of bleaching process, the pieces of banana pseudo-stem waste are rinsed by aquades, and they are dried in the sun to evaporate water contained in them. The pieces of dried banana pseudo-stem as a bleaching result are mashed to a size of 100 meshes.

Nanocellulose isolation from banana pseudo-stem waste using cis-oleilimidazolinium acetate and its testing

Bleached banana pseudo-stem waste powder is entered into cis-oleilimidazolinium acetate which is melted in warm water. The process of nanocellulose isolation from banana pseudo-stem waste using cis-oleilimidazolinium acetate ionic liquid is done for 2 hours. The testing of nanocellulose that has been formed can be done by comparing Tyndall effect on 3 test tubes that each tube contains only ionic liquid, mixture of ionic liquid and dried banana pseudo-stem, and mixture of ionic liquid and bleached banana pseudo-stem powder from nanocellulose isolation result.

RESULTS AND DISCUSSION

Chemistry concepts in preparation sample process of banana pseudo-stem waste

In the preparation sample of banana pseudo-stem waste, the pieces of banana pseudo-stem waste are dried in the sun for a week. It is intended for removing water content in them through the water vaporization process. Water vaporization process is a chemical process on the change of water in liquid phase into gaseous phase water. Thermodinamically the water vaporization process is endoterm where the process of changing water in liquid phase into gaseous phase water requires energy (+ sign on enthalpy) (McMurry & Fay, 2004).

$$H_2O(l) \rightarrow H_2O(g) \quad \Delta H = +44 \text{ kJ/mol}$$

Next the pieces of dried banana pseudo-stem waste are bleached. This process aims to gain purified cellulose by removing components other than cellulose such as lignin and hemicellulose through hydrolysis reaction using acid catalyst. Hydrolysis reaction is a reaction of substances with water which causes ionized water into H^+ (or H_3O^+) and OH^- (Whitten et al., 2004). Acetic acid in bleaching agent acts as acid catalyst to facilitate the hydrolysis process of components other than cellulose. The Illustration of hydrolysis mechanism of hemicellulose is shown in Figure 2.

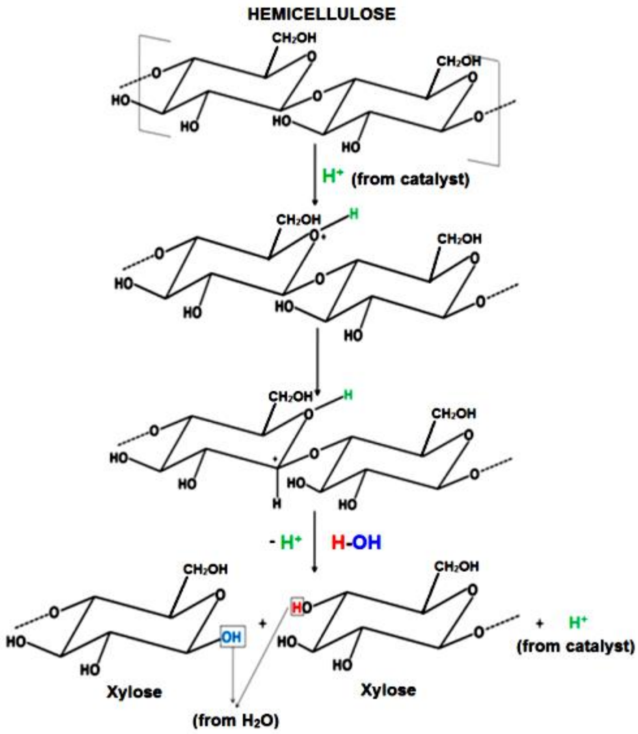

Figure 2. Illustration of hydrolysis mechanism of hemicellulose

Chemistry concepts in the process of nanocellulose isolation using cis-oleilimidazolinium acetate ionic liquid

The process of nanocellulose isolation from banana pseudo-stem waste using cis-oleilimidazolinium acetate ionic liquid is done by adding bleached banana pseudo-stem powder in a size of 100 meshes into ionic liquid. The small size of banana pseudo-stem powder aims to accelerate the reaction of nanocellulose isolation because the touch surface area of the substance is one of the factors that can accelerate the reaction rate. Particles with large touch surface area will increase the possibility of them to collision effectively, so the reaction will be faster than particles with small touch surface area (Jespersen et al., 2011). The illustration of the influence of touch surface area to the reaction rate is shown by Figure 3.

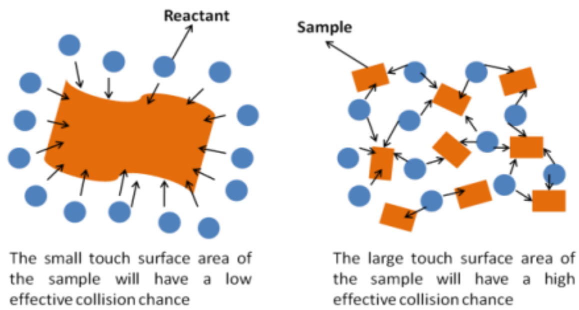

Figure 3. Illustration of the influence of touch surface area to the reaction rate

The principle of nanocellulose isolation of banana pseudo-stem waste using cis-oleilimidazolinium acetate ionic liquid is hydrogen bonding termination of celluloce chains by ion-dipole interaction between -OH groups on celluloce chains and ions from ionic liquid (Isik et al., 2014). It is occured because the ion-dipole interaction is relatively stronger than hydrogen bonding (McMurry & Fay, 2004). The illustration of hydrogen bonding termination of cellulose chains by ionic liquid is shown by Figure 4.

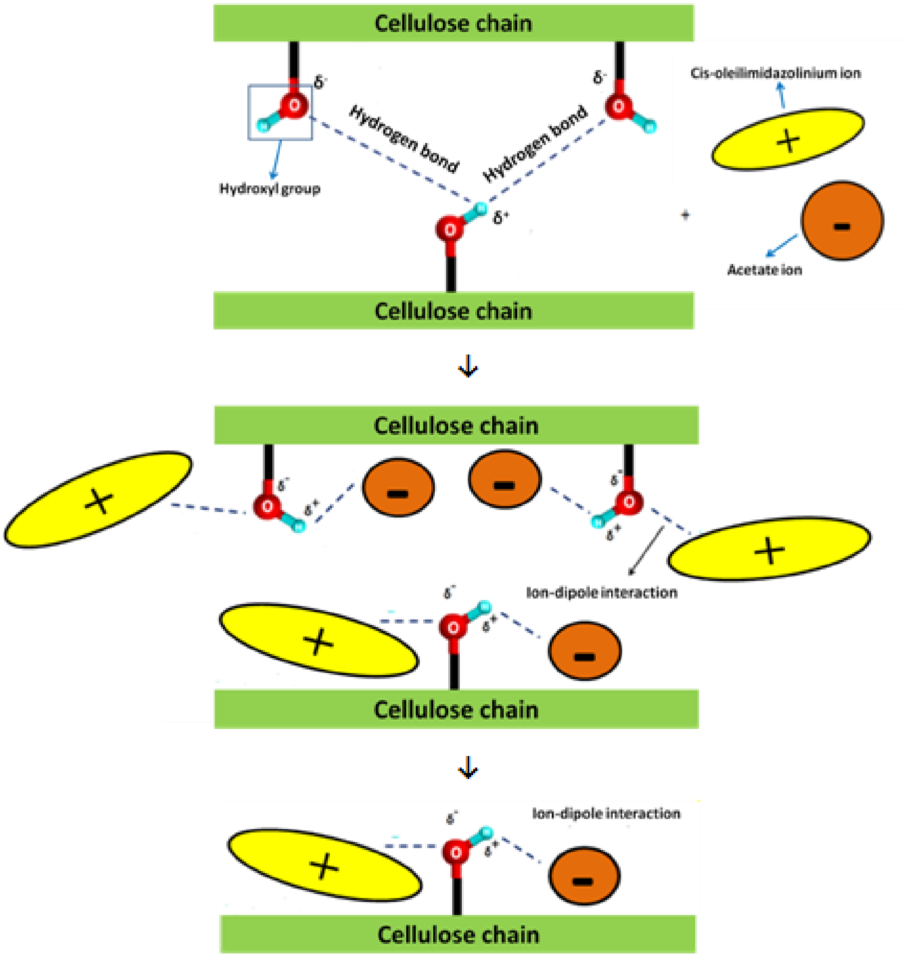

Figure 4. Illustration of celluloce chains hydrogen bonding termination by ionic liquid

The test of obtained nanocellulose can be done by light scattering test (test of Tyndall's effect) on three reaction tubes where each of reaction tubes contains ionic liquid only, a mixture of dried banana pseudo-stem waste with ionic liquid, and a mixture of bleached banana pseudo-stem powder with ionic liquid that has been carried out the nanocellulose isolation process (Figure 5).

a) b)

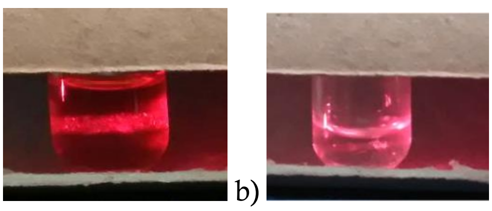

Figure 5. Tyndall's effect occured in a) mixture of bleached banana pseudo-stem powder with ionic liquid that has been carried out the nanocellulose isolation process and b) only ionic liquid

Based on light scattering test (test of Tyndall's effect), a mixture of bleached banana pseudo-stem powder with ionic liquid that has been carried out the nanocellulose isolation process can scatter the light passed on the tube. It is shown that the process of nanocellulose isolation from banana pseudo-stem waste using cis-oleilimidazolinium acetate ionic liquid has been successfully done. Nanocellulose is a nano-sized particle, and particles size 1-1000 nanometers will form a colloidal system when they are dispersed in a medium, and this system has an ability to scatter the light when is passed on it (Jespersen et al., 2011).

Principles of green chemistry in the process of nanocellulose isolation using cis-oleilimidazolinium acetate ionic liquid

The relationship between nanocellulose isolation using cis-oleilimidazolinium acetat and principles of green chemistry is shown by Table 1.

Tabel 1. Relationship between nanocellulose isolation and principles of green chemistry

Nanocellulose isolation process	Principles of green chemistry	Explanation
The use of nanocellulose isolation for introducing chemistry concepts and green chemistry concepts	Design for degradation Designing safer chemical	Nanocellulose has low toxicity (Salas et al., 2014) and biodegradable property, so when the demonstration of nanocellulose isolation has been done, the experimental result can be discharged into the environment.
Nanocellulose isolation from banana pseudo-stem waste using cis-oleilimidazolinium acetate ionic liquid	Prevention Less hazardous chemical syntheses Atom economy Safer solvents and auxiliaries Use of renewable feedstocks Reduce derivates Inherently safer chemistry for accident Real-time analysis for pollution prevention	Ionic liquid is used because it is a new type of solvent that has non-toxic properties, environmentally friendly (Mahlambi et al., 2011; Pham et al., 2010) high chemical and thermal stability, low vapour pressure, nonflammable, and can be recycled. Beside that, ionic liquid has properties that do not form derivates when it isolates the nanocellulose from cellulose (Effendi et al., 2015, 10). Unlike other methods such as acid hydrolysis, steam explosion, mechanical milling, organic solvent and TEMPO-mediated oxidation process, they have disadvantages such as long reaction time, low yield with high time consuming, high cost of production, using toxic solvent, difficulty in separation and they will cause environmental pollution (Xiao et al., 2015). The nanocellulose isolation process using ionic liquid only requires ionic liquids to isolate nanocellulose particels from their raw material (Xiao et al., 2015; Wang et al., 2013) while the other nanocellulose isolation method like acid hydrolysis needs several solvents to isolate nanocellulose particels from their raw material (Ioelovich, 2012).
In the nanocellulose isolation process, it is used a spirits burner	Design for energy efficiency	Ionic liquid is a salt with [R]X formula that has melting point below 100^{o}C, this definition is used because ionic liquid is usually liquid at room temperature. In the nanocellulose isolation process, it is not required a high temperature to melt the ionic liquid because it can melt easily below 100^{0}C, unlike ordinary salts that require high temperatures to melt them. Nanocellulose isolation using other methods such as acid hydrolysis, steam explosion, mechanical milling, organic solvent and TEMPO-mediated oxidation process have high energy demand (Xiao et al., 2015).

CONCLUSION

Based on the result of this experiment, nanocellulose has been obtained from banana pseudo-stem waste using cis-oleilimidazolinium acetate ionic liquid. It is based on the result of Tyndall's effect test that has been done. In the process of nanocellulose isolation, there are several chemistry concepts that can be learned as such as water vaporization process, hydrolysis process on lignin and hemicellulose, factors that can accelerate the reaction rate, concept of intermolecular force on cellulose chains hydrogen bonding process, and colloidal system concept on light scattering test (Tyndall's effect test). The experiment of nanocellulose isolation has potentially introduced the green chemistry on high school students because it has the principles of green chemistry except catalysis principle.

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