

PAPER • OPEN ACCESS

Integrating ILS in dissolving palm fruit cellulose

To cite this article: K N Mansor *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **702** 012013

View the [article online](#) for updates and enhancements.

You may also like

- [Ionic liquid glasses: properties and applications](#)
Mikhail Yu. Ivanov, Nikolay V. Surovtsev and Matvey V. Fedin
- [Preparation and Characterization of Titanium Tetrachloride-Based Ionic Liquids](#)
Lixia Gao, Lina Wang, Tao Qi et al.
- [Physicochemical Properties of Tungstate-Based Room-Temperature Ionic Liquids](#)
Yunxiang Qiao, Jun Hu, Huan Li et al.



The advertisement features a dark teal background. On the left, the ECS logo is displayed above the text 'The Electrochemical Society Advancing solid state & electrochemical science & technology'. Below this, the '242nd ECS Meeting' is announced for 'Oct 9 - 13, 2022 • Atlanta, GA, US', with a note that it will present 'more than 2,400 technical abstracts in 50 symposia'. In the center, a portrait of M. Stanley Whittingham is shown next to a Nobel Prize medal. To the right of the portrait, text identifies the 'ECS Plenary Lecture featuring M. Stanley Whittingham, Binghamton University Nobel Laureate - 2019 Nobel Prize in Chemistry'. A 'Register now!' button with a checkmark icon is positioned above a photograph of a large audience seated in a lecture hall. In the bottom right corner, a person is shown holding a glowing hexagonal grid of icons representing various scientific fields.

ECS The Electrochemical Society
Advancing solid state & electrochemical science & technology

242nd ECS Meeting
Oct 9 - 13, 2022 • Atlanta, GA, US
Presenting more than 2,400
technical abstracts in 50 symposia

ECS Plenary Lecture
featuring
M. Stanley Whittingham,
Binghamton University
Nobel Laureate -
2019 Nobel Prize in Chemistry

Register now!

Integrating ILS in dissolving palm fruit cellulose

K N Mansor¹, M A M Halim¹, N Ali², W Z W A Rahman² and S B Abdullah^{1*}

¹ Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Gambang, Kuantan, Pahang, Malaysia

² D3 Technology Solutions Sdn Bhd, Gebeng Industrial Area Phase II, 26080 Kuantan, Pahang, Malaysia

*E-mail: syamsul@ump.edu.my

Abstract. Ionic liquids (ILs) are introduced to improve the production of palm oil in industry in order to extract more thus lowering the oil losses. The main objective of this study is to investigate the potential of sulfate-based ILs in dissolving palm fruit cellulose. The interpretation study was done in lab scale by dissolving cellulose (that directly extracted from palm fruit) in ILs and percentage of cellulose dissolved is calculated based on weight percentage dissolved. The highest average percentage dissolving of cellulose was 10.67 wt% with condition concentration of sulfate-based ILs is 9000 ppm and the mixing time was two hours. In a nutshell, the higher concentration of ILs and more mixing time will achieve higher percentage of cellulose dissolved.

1. Introduction

World production of palm oil, the most widely traded edible oil, has also seen significant leaps in production and planted areas; production had almost doubled from 1990 to 2001, with Malaysia and Indonesia contributing to most of the increased production. This had been achieved mainly by opening of new land for oil palm plantations [1]. Although the production was increased year by years, but the production counted many losses in term of residue oil that still left during pressing process and slightly in empty fruit bunch though trenching [2]. The highest mean is the oil loss in the pressed fiber and the lowest mean is the oil loss in the wet nut [3].

It can be seen the major of palm oil production was producing about 74 wt% for palm oil-based products, from food to cleaners to cosmetics. Huge production also comes with huge losses of palm oil by considering the amount of oil loss from 5 – 10 wt% during pressing step to be exactly. Hence, oil losses can be reduced during the pressing process by introducing the ionic liquids (ILs) as pre-treatment right before pressing of palm fruit. Perhaps by applying ILs before pressing process will breakup more oil cell within cellulosic structures of fresh fruit bunch for palm oil. Many researchers agreed that pre-treatment was the key to unlock the recalcitrance of lignocellulose for cellulosic structure containing oil palm hence gives proof that using ILs can also help to dissolve the cellulose. In general, a palm oil mill extracts 20 wt% of oil from the fresh fruit bunch while also produces 23 wt% of empty bunch, 15 wt% of fiber, and 12 wt% of nut. Oil losses occur in various by-products, including the fiber, which remains after the mesocarp oil is extracted by a screw press [3-5]. In order to accommodate above palm oil problem, the objective in improving palm oil is to investigate potential sulfate-based ILs in dissolving cellulose whereas directly extracted from palm fruit. Several process parameters have been identified to evaluate the dissolution of palm fruit cellulose in sulfate-based ILs such as temperature, concentration and mixing time.



2. Materials and Methods

2.1. Materials

Cellulose fiber which directly extracted from the palm fruit was supplied by D3 Technology Solutions Sdn. Bhd. Pure 1-butyl-3-methylimidazolium methylsulfate ILs was prepared accordingly in Faculty of Chemical and Process Engineering Technology (FTKPP) Laboratory.

2.2. Breakdown of cellulose and dilution

The cellulose fiber was separated and breakdown into powder before immersing in sulfate-based ILs namely 1-butyl-3-methylimidazolium methylsulfate. But before immersion process is taking place, the ILs was diluted into three different concentrations and the experiment was prepared for three different mixing times. After immersion, drying process was conducted to remove moisture from the sample for weighing purposes.

2.3. ILs dilution process

Pure 1-butyl-3-methylimidazolium methylsulfate ILs was diluted using deionized water to obtain three different concentrations of 5000 ppm, 7000 ppm and 9000 ppm. The total of 6 ml each of diluted ILs were used to immerse cellulose powder for heating with stirring processes [6].

2.4. Sample preparation

Experiment was started with 0.05 g cellulose powder was added into 50 ml beaker contain 6 ml of diluted ILs solution. 150 ml of tap water as water bath was heat up until 70°C using hotplate. Then the solution in the 50 ml beaker was placed into the water bath on the hotplate and the stirrer was adjusted at 800 rpm while the temperature remains at 70°C. The solution was placed in covered beaker to avoid contamination and impurities. The heating and stirring process was carried out for two hours (changes follow the time set). The solution was rinsed with 25 ml of deionized water before filtered using filter paper for 20 min. After that, rinsed process was proceeded to remove the remaining ILs. Then, the filtered sample was dried in an oven with the temperature of 70°C for 24 hours and was put at room temperature for 20 min of cooling process. Bear in mind, the filter paper was weighed before filtering the solution to prevent error in mass change of cellulose. Then, the dried sample was weighed, and the data was recorded [7].

2.5. Dissolution performance

Change in mass of cellulose fiber was calculated after the drying process. The percentage dissolution was calculated using equation (1) below:

$$\text{Percentage dissolution (\%)} = \frac{\text{Weight Before (g)} - \text{Weight After (g)}}{\text{Weight Before (g)}} \times 100 \quad (1)$$

3. Result and Discussion

3.1. Percentage of dissolution

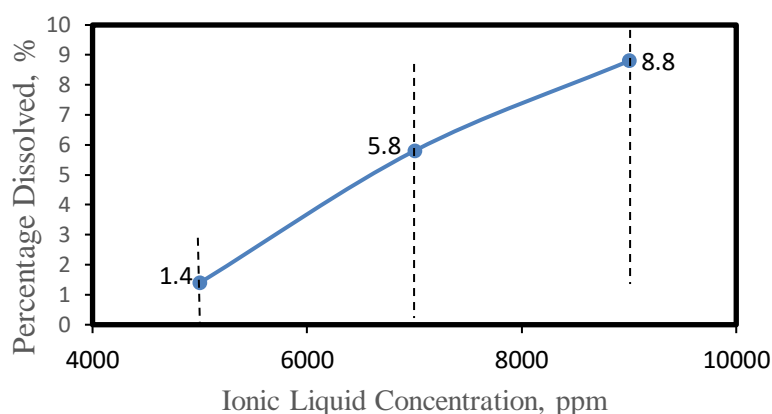
The percentage of dissolved cellulose fiber at various process parameters was summarized in table 1. The overall result obtained depicts the highest average percentage for dissolved cellulose at 9000 ppm ILs solution for two hours mixing time. In many studies with ILs, it was foreseen that both cation and anion plays great importance and responsible for the dissolution of cellulose especially for sulfate-based ILs [6].

Table 1: Summary of cellulose dissolved percentage

Concentration of ILs (ppm)	Cellulose Dissolved (wt%)
1 hour	
5000	1.4
7000	5.8
9000	8.8
1.5 hours	
5000	4.4
7000	8.4
9000	10.8
2 hours	
5000	7.0
7000	10.4
9000	12.4

3.1.1 Effect of ILs concentration

Based on figure 1 to 3, the ILs solution with concentration of 9000 ppm has better dissolution percentage compared to ILs solution with concentration of 5000 ppm and 7000 ppm. For ILs solution with concentration of 9000 ppm, the average dissolution percentage is 10.67 wt% higher compare to the other two ILs concentrations which merely has the average dissolution percentage of 4.27 wt% and 8.2 wt% for 5000 ppm and 7000 ppm, respectively. As the concentration of the ILs increases from 5000ppm to 9000ppm, the percentage of cellulose dissolved also increased. The reason behind the improvement of electro spinnability of cellulose with the addition of ILs as solvent is that ILs penetrates into the gaps between the chains and the ILs enhancing the chain mobility due to weaker interaction with the chains. As a result, cellulose chains can be easily elongated and fibrillated during the process [7]. This can deduce that increased of concentration ILs brings high dissolution percentage of cellulose because the chain of cellulose become weaker after mixing with solvent [8].

**Figure 1:** Effect of ILs concentration for mixing time at 1 hour

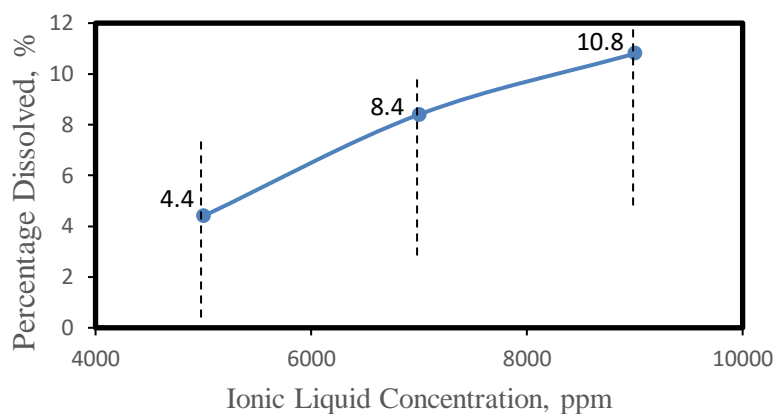


Figure 2: Effect of ILs concentration for mixing time at 1.5 hours

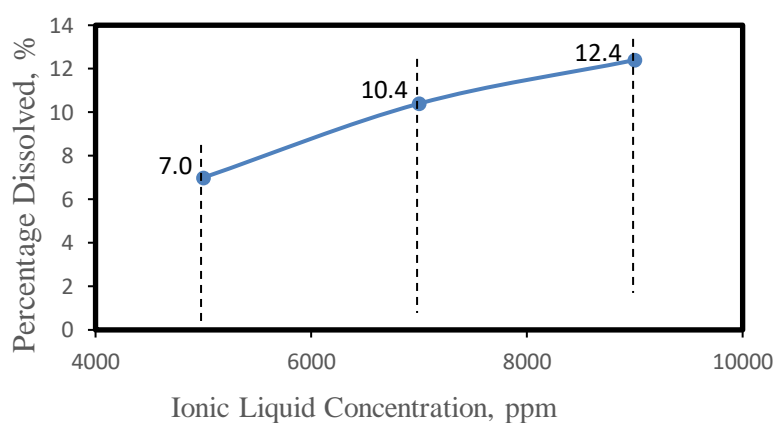


Figure 3: Effect of ILs concentration for mixing time at 2 hours

3.1.2 Effect of mixing time

Mixing time play an important role in determining the rate of cellulose fiber dissolved with ILs whilst mixed up together during allocation time. Based on the graphs shown in figure 4 to 6, mixing time for two hours recorded has the higher rate of cellulose fiber dissolved with the average of 9.93 wt% compared to the one hour with the average of 5.33 wt% and one and half hour with the average percentage of 7.87 wt%. The results showed that increasing of the mixing time for cellulose mixed up with ILs will increase the percentage of cellulose dissolved and the dissolution process generally occurred regarding on the variable of mixing time. Mixing time is important parameter because it significantly shows for substance to react with solvent and how long the time will decide the reaction time. From previous study done in 2017, which had identified the optimum time for the reaction between cellulose and different type of solvent is more than 2 hours at 90°C [9].

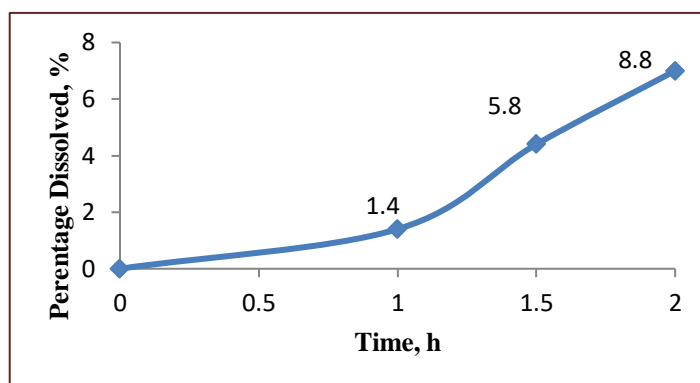


Figure 4: Effect of mixing time for cellulose dissolution at 5000 ppm of ILs concentration

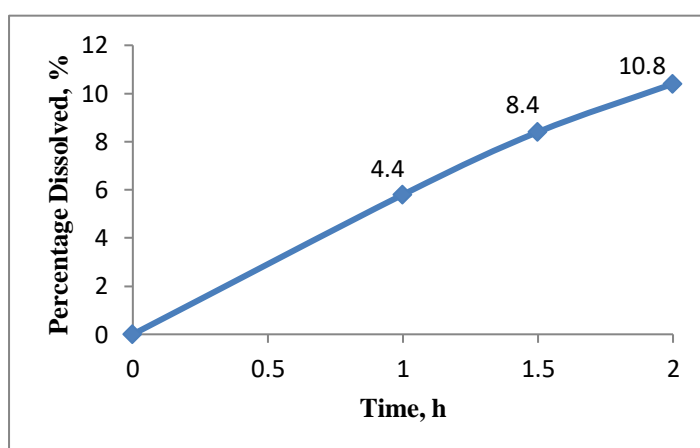


Figure 5: Effect of mixing time for cellulose dissolution at 7000 ppm of ILs concentration

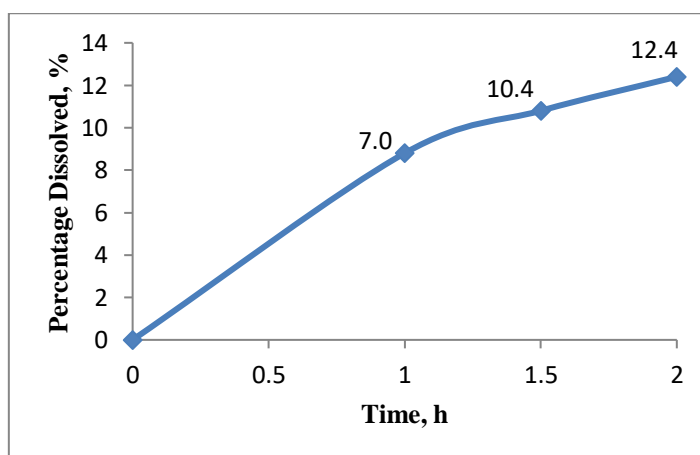


Figure 6: Effect of mixing time for cellulose dissolution at 9000 ppm of ILs concentration

4. Conclusion

In a conclusion, based on the two main parameters which are ILs concentration and mixing time, it shows that the highest percentage of cellulose fiber dissolution occurred for concentration at 9000 ppm during mixing time of two hours. A higher concentration of ILs solution needed for better cellulose fiber dissolution whereas required higher temperature and longer mixing time. The optimum temperature for

this experiment was set up at 70°C because sample will evaporate to the atmosphere when conducting above 70°C.

Acknowledgement

The authors would like to thank the Ministry of Higher Education Malaysia under the Fundamental Research Grant Scheme (FRGS/1/2016/TK02/UMP/03/1 - RDU160128) and D3 Technology Solutions Sdn. Bhd. for the research financial/material supports.

References

- [1] Inyang J, Munot M A and Shazali S T S, Tanjong S J 2019 A Model to Manage Crude Palm Oil Production System *MATEC Web of Conferences* 255 02001
- [2] Lai O M, Tan C P and Akoh C C 2012 *Palm Oil: Production, Processing, Characterization, and Uses* 1st Edition Elsevier
- [3] Zulkefli F, Othman N, Syahlan S, Zaini M R and Bakar M A 2017 Fresh Fruit Bunch Quality and Oil Losses in Milling Processes as Factors that Affect The Extraction Rate of Palm Oil *International Journal of Agriculture, Forestry and Plantation* **5** 99-103
- [4] González B, Gómez E, Domínguez Á, Vilas M and Tojo E 2011 Physicochemical Characterization of New Sulfate Ionic Liquids. *Journal of Chemical and Engineering Data* **56** (1) 14-20
- [5] Azmi S I, Azizan A and Salleh R M 2018 Pretreatment of Oil Palm Frond with Ionic Liquid. *IOP Conference Series: Materials Science and Engineering* 358
- [6] Salvador Â C, Santos M and Saraiva J M A 2010 Effect of The Ionic Liquid [bmim]Cl and High Pressure on The Activity of Cellulase *Green Chemistry* **12**(4), 632-35
- [7] Isik M, Sardon H and Mecerreyes D 2014 Ionic Liquids and Cellulose: Dissolution, Chemical Modification and Preparation of New Cellulosic Materials *International Journal of Molecular Science* **15**(7), 11922-40
- [8] Sashina E S and Novoselov N P 2009 *Effect of Structure of Ionic Liquids on Their Dissolving Power toward Natural Polymers Russian Journal of General Chemistry* **79**(6) 1057- 62
- [9] Mohd N, Draman S F S, Salleh M S N and Yusof B 2017 Dissolution of Cellulose in Ionic Liquid: A Review *AIP Conference Proceedings* **1809**(020035).