

# Isolation and Characterization of Lignin from Rubber Wood in Ionic Liquid Medium

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## Abstract

In this study, lignin was isolated from rubber wood by means of ionic liquid (1,3-dimethylimidazolium methylsulfate). The optimum parameters to isolate lignin were investigated which include the concentrations of ionic liquid (0.1, 0.2, 0.3, 0.4, 0.5 moles), isolation durations (30, 60, 90, 120, 150 minutes), and isolation temperatures (25, 40, 60, 80, 100°C). The optimum parameters for lignin isolation are 0.5 moles concentration, 120 minutes and 100°C. Lignin could be isolated from rubber wood up to 13.03wt.% by ionic liquid however, in comparison with acid and alkaline isolation, they give better yields than ionic liquid which 22.15wt.% and 18.56wt.% of lignin, respectively. Lignin preliminary structures isolated by ionic liquid have been confirmed based on Fourier transform infrared (FTIR) results. From these results, isolated lignin by ionic liquid provides lignin without other related isolative. Apart from that, ionic liquid also could be recycled up to three times compared to acid and alkaline isolation methods.

**Keywords:** Lignin, Ionic liquid, Rubber wood, Isolation, Waste biomaterial

## 1. Introduction

Rubber wood (*Hevea brasiliensis*) becomes raw material for sawmills and wood product factories, such as furniture, kitchenware, wooden apparatus etc, when they turn out to be matured rubber trees. This natural waste biomaterial contained lignin that is the second in abundance after the cellulose. Lignin is part contained in the middle and lamella cell wall that acts as the adhesive between cells. The amount of lignin in plants varies widely, and is typically contributing in the range of 20-30% in weight (wt.%) dry wood depending on the nature of woods used (Sundqvist, 2004). Lignin serves as a raw starting material for many useful products. The macromolecular and multifunctional nature of lignin makes it very suitable for newer industrial compounds and a new class of polymers. Pure lignin is basically an amorphous form consisted of aromatic ether backbone that can be potential substrates for the production of aromatic chemicals (Chakar & Ragauskas, 2004). Lignin can be isolated in large quantity from lignocellulosic materials by a strong dilute acid, alkaline treatment, organosolve isolation or sulphite pulping process (Pouteau, et al., 2003; Fernandez-Bolanos, et al., 1999). However, the drawbacks in the case of the above processes are toxicity, high cost, difficulties in recovery, severe side-reaction, and instability in processing. During isolation of lignin, many potentially toxic chemicals such as sulfuric acid or sodium hydroxide may often be found to modify lignin, cellulose or other related compounds by change chemical structure due to the breakdown of some inter unit linkages under the acid or alkaline isolation conditions (Lundquist, 1992; Csiszár, et al., 2006). In addition, none of the isolation processes that have been previously attempted facilitates the production of lignin.

Ionic liquids are promising environmentally benign indicated some intriguing properties such as negligible vapour pressure, good dissolving, and reusable (Welton, 1999; Hoffmann, et al., 2003; Poole, 2004; van Rantwijk &

Sheldon, 2007). Ionic liquids have been hailed as the green alternative to conventional volatile organic solvents (VOSs) in chemistry due to their novel properties, while maintaining the ability to dissolve a wide range of organic and inorganic compounds. Ionic liquids are capable of dissolving complex macromolecules and polymeric materials such as lignocellulose with high efficiency (Liu, et al., 2005; Xie, et al., 2005). Recently, ionic liquids that being considered as green solvents have been successfully used to dissolve and process lignocellulosic materials without derivatization (Wu, et al., 2004). Since ionic liquids offer a potentially clean method for carrying out isolation, chemical reactions or processes biopolymers materials, more attention have been paid on cellulose (Feng & Chen, 2008), starch and protein (Biswas, et al., 2008). The aim of this study was concentrated on lignin isolation that obtained from rubber wood. The lignin isolation process was carried out in ionic liquid system and the effects of varying isolation conditions were determined and studied. The results regarding the weight percentage of isolated lignin were investigated by comparison with the earlier reports, acid and alkaline isolation processes. The functional groups of preliminary structures of isolated lignin were determined spectroscopically by using Fourier transform infrared (FTIR). The capability of ionic liquid of being able to recycle especially after isolation of lignin from rubber wood was also reported in this study.

## 2. Materials and methods

### 2.1 Materials

1,3-Dimethylimidazolium methylsulfate and methanol were purchased from Merck. Sulfuric acid was obtained from Sigma Aldrich. Sodium hydroxide was purchased from Acros. Rubber wood was kindly supplied by sawmill and wood processing industries, it has taken from a local forest in Malaysia. All chemicals purity  $\geq 98.0\%$  and were used as received.

### 2.2 Isolation of lignin by ionic liquid

In this study, rubber wood was grinded, sieved (200 $\mu\text{m}$ ), and kept dried in a vacuum oven at 100°C for overnight to eliminate moisture before isolation. Batch isolation experiments were conducted in glass flasks that were filled with unvarying rubber wood (5.25g). 1,3-Dimethylimidazolium methylsulfate was poured in glass flask to prepare lignocellulose solution with 0.1 moles concentration of ionic liquid, followed by mole rising (0.2, 0.3, 0.4 and 0.5 moles) separately. The glass flask was allowed to be stirred (500 rpm) at 25°C for 30 minutes. The insoluble component in the ionic liquid-soluble lignin was then separated from solution by filtration under reduced vacuum pressure from the corresponding solution. Then, the solubilized lignin was isolated from ionic liquid by precipitation with methanol. Upon completion, the obtained precipitated residue was filtered off. The isolated lignin was purified by washing thoroughly four times with distilled water. The lignin was collected and then rigorously dried into vacuum oven at 85°C for at least 24 hours until dryness. The lignin was weighed and the isolated fibres were quantified and for the contents of lignin and other related isolative. Further adjustments in isolation duration (60, 90, 120 and 150 minutes) were made based on the higher lignin yield that obtained from appropriate isolation concentration. The isolation temperature remained nearly constant for 25°C in that condition before slowly elevated (40, 60, and 80) to a final value of 100°C by immersed in oil bath respectively and it is based on the higher lignin yield that acquired from suitable isolation duration. The isolations were repeated in quintuplicate (five times) and the average yields were computed. The isolated lignin was also kept under silica gels prior to further analysis.

### 2.3 Isolation of lignin by acid and alkaline

The rubber wood used for acid and alkaline isolations was also in dried fine powder form. Acid isolation was done according to method as described elsewhere (Pouteau, et al., 2003) with some minor modifications, 5.25g of the rubber wood was heated in a flask containing 72% sulfuric acid, rubber wood to solvent ratio of 1:28 (by weight). The mixture was then heated under reflux for three hours. After filtration the solvent was removed under vacuum, the lignin was then washed with distilled water until the filtrate was neutral. While, isolation of lignin by alkaline following literature procedure (Jiebing, et al., 2009) 5.25 g rubber wood was mixed with 1.5 mol/L sodium hydroxide at 70°C for 120 minutes in a rubber wood to solvent ratio of 1:10 (by weight). This was followed by filtration, the solid residues were removed, and then the liquor lignin was neutralized to pH 4 by addition of sulfuric acid, and precipitated with distilled water. The precipitate was filtrated then, purified and dried as analogous to the ionic liquid procedure.

### 2.4 Characterization

In order to characterize the preliminary structures of isolated lignin the optimum lignin yield that obtained by ionic liquid isolation have been evaluated by Fourier transform infrared (FTIR) spectrometry. Perkin-Elmer (RX 1 model) FTIR system instrument was used to record FTIR spectra to determine the functional groups present in the

isolated lignin. The FTIR spectra were obtained on applying a horizontal attenuated total reflectance (HATR) cell by direct transmittance lignin solution using identical ionic liquid as a solvent (1wt.% lignin). All spectra were rationed against background spectra. The analyses were performed in quintuplicate and the average spectra were taken. Each spectra was 20 scans, in the frequency range of 4000-400 $\text{cm}^{-1}$  and resolution of 4 $\text{cm}^{-1}$ .

### 2.5 Recyclability Test

Ionic liquid was recycled for at least three times through similar isolation procedure rigorously. The recyclability was measured by compared percentage of isolated lignin with first time isolation by intact ionic liquid. All recycled ionic liquid were proper filtered and dried prior to measurement. The recovery ionic liquid obtained was utilized for the estimation of mass balances of recycled ionic liquid. The measurement was calculated as previously, and, if the results appeared unsatisfactory, purification and isolation was repeated in quintuplicate. The average yield of isolated lignin was being used as comparison.

## 3. Results and discussion

### 3.1 Isolation of Lignin

All weights and calculations were made on an oven-dried (85°C, 24 hours) basis. The conditions of isolation to get the highest solubility of lignin were optimized by varying concentrations of ionic liquid. The results given below (Figure 1), the total yield of soluble lignin increased from 0.123 to 0.224g with mole increases from 0.1 to 0.5 moles isolation concentration of ionic liquid at room temperature (25°C) for 30 minutes. The best concentration for highest lignin yield was found at 0.5 moles with lignin solubility value of 0.224g. In this lignin isolation study, higher concentration of ionic liquid showed more soluble rubber wood lignin than less concentration of ionic liquid due to the decrease amount of lignin saturation.

Isolation duration of the lignin with maximum concentration of ionic liquid was evaluated. In general, increase of isolation duration showed higher soluble lignin yields than less time-consuming isolation. Higher lignin soluble yield is 0.298g, which obtained at 120 minutes isolation in room temperature (25°C). Lignin solubility values retained for this isolation condition. After that, solubility of the lignin in rubber wood samples became almost similar (see Figure 2). No significant differences in their yields were observed in prolong durations. These results suggested that under the appropriate extension condition of ionic liquid isolation duration corresponds with increasing of ionic liquid concentration favoured the lignin released from the cell walls of rubber wood.

In a series of isolation studies with varying temperatures, the low temperature condition soluble less lignin than the higher ones. Lignin solubility was increased from 0.298g (25°C) to 0.578g (60°C), and up to 0.684g for 100°C, with 0.5 moles of ionic liquid and at 120 minutes. The rates solubility of the lignin at high temperature condition was found to be more accelerated isolation than the low temperature samples in ionic liquid for couple of hours (Figure 3). These results indicated that lignin only fully isolated from rubber wood at optimum condition of isolation and it did not fully soluble in the ionic liquid except the presence of elevated temperature that may increase the interaction between lignin and ionic liquid.

Table 1 gives the yields (wt.%) of total isolated lignin obtained by ionic liquid, acid and alkaline isolation procedures. Yield of lignin is given on a dry weight basis related to the rubber wood. As can be seen, isolation of the rubber wood (wt.% dry starting material) with 0.5 moles ionic liquid at 100°C for 120 minutes could be isolated only 13.03wt.% lignin of the rubber wood. Meanwhile, acid and alkaline isolation yielded 22.15wt.% and 18.56wt.% lignin, respectively. The initial filtration with ionic liquid removes the residual or non-isolatable lignin fibre material which is mainly due to the presence of cellulose and other related compounds which a part of rubber wood, and it was calculated from the isolated yield corresponding to the release of 78.45wt.% non-lignin fibre material. For acid and alkaline isolation, the related unsolubilized fibre materials were accounted only 64.22wt.% and 74.01wt.% of the total solubilized rubber wood lignin, respectively. The amounts of fibre materials were higher in all cases and slightly lower fibres for acid isolation. As shown in Table 1, the ionic liquid isolation process resulted in much lower lignin isolation yield from rubber wood although the process conditions were optimum for the highest lignin yield. The total of isolated lignin yields from rubber wood are approximately between 13wt.% compared to acid and alkaline isolation processes even after experiment repeated (consistency was 88%). This study established that conventional acid and alkaline does isolated lignin from rubber wood in a good yield under strong conditions. Nevertheless, the lignin precipitated from liquors regarded as acid-soluble and alkali-soluble lignin compounds are not completely free from impurities owing to non-homogenous appearances of liquors though after centrifugation (data not shown) so, they probably contained relatively higher amounts of other modified compounds thus, it tend to result in more yields. In other words, it is no longer identical to the lignin found in the rubber wood. Moreover, this caused inaccuracy in the estimation, due to the additional contamination

of lignin yields in the precipitate. The lignin yield that isolated by ionic liquid was rather homogenous and the contamination relatively small.

It can also be seen from Table 1 that in all isolations the total amount of isolated lignin and fibre is less than 100% of the rubber wood. It is thus obvious that there is a large loss of rubber wood lignin in the waste liquid, possibly due to their high water solubility and/or their tiny molecule dimensions stays in the waste liquid. When attempting to wash the isolated lignin, this lignin was found to be washed away, if avoiding washing, the precipitate contaminated by the organic or inorganic salts, and therefore, the isolated lignin was washed after filtration, to eliminate as much as possible of the some organic or inorganic salts. For ionic liquid isolation, the amount was 8.52wt.%, somewhat strangely, an alkaline isolation gave 7.43wt.% water-soluble yield. The water-soluble yields for both isolation processes from rubber wood are noticeably almost close to each other. Whereas, acid isolation was extremely yielded high water-soluble compounds (13.63wt.%) due to the fact that some isolative escaped from the precipitation by acidification that altered the polysaccharide within the cell walls of rubber wood during this isolation by hydrolyzation of cellulose fibre produced some water-soluble monosaccharides (Kim, et al., 2001). This observation implied that the use of ionic liquid isolation under the optimum conditions may be significantly offered best isolation option with strong delignifying properties devoid of derivatization. Additionally, the rapid process by one-step precipitation process in the ionic liquid isolation makes lignin easy to obtain from rubber wood.

### 3.2 FTIR Analysis

The corresponding assignments of FTIR spectra of the ionic liquid-soluble lignin are given in Table 2. Eventhough the spectra contains numerous absorbtion bands not all can be designated explicitly to structural groups of lignin, the region from the 4000 to 3500 $\text{cm}^{-1}$  did not show any useful information of isolated lignin by ionic liquid was not considered further. As can be seen from Table 2, the magnification region, 3500–900 $\text{cm}^{-1}$  was far more informative however, the hydroxyl, carbonyl, methoxyl and carboxyl groups are the most important chemical functional groups in lignin and sufficiently they can be used for identification of lignin (Gosselink, et al., 2004). Similar observations were clearly found in this study of rubber wood lignin isolated by ionic liquid. The FTIR spectra of the lignin samples possess strong intensity band at 3420 $\text{cm}^{-1}$  that attributed to the hydroxyl groups, methoxyl groups is showed by medium intensity absorption bands at 2860–2850 $\text{cm}^{-1}$ , carbonyl groups are assigned at 1740–1660 $\text{cm}^{-1}$  stretching frequencies with strong intensity. The medium intensity absorption at 1715 $\text{cm}^{-1}$  demonstrated C–O nonconjugated and strong intensity at 1712 $\text{cm}^{-1}$  illustrated carboxyl groups. Strong intensity absorption at 1640 $\text{cm}^{-1}$  is indicative of carbonyl stretching conjugated with aromatic ring skeleton. The strong intensity bands in the range of 1610–1503, 1422 $\text{cm}^{-1}$  could be assigned to the C–C bounds group of the aromatic skeleton compounds present in the isolated lignin. The range of bands for rings conjugated with carbonyl group is found at 1500–1400 $\text{cm}^{-1}$ . The medium intensity band at 1462–1420, 743 $\text{cm}^{-1}$  were attributed to the vibration of the C–H deformations and aromatic ring group. A noticeable strong absorption centred observed at 1331–1325 and 1270–1260 assigned to syringyl and guaiacyl revealed that the rubber wood lignin are composed of high syringyl and guaiacyl units congruent with hardwood lignin that has been reported in several instances in the literature (Akiyama, et al., 2005). The medium intensity at 1230–1215 $\text{cm}^{-1}$  band is associated with aromatic phenyl C–O stretching. The strong intensity broad peak centred at 1137–1033 $\text{cm}^{-1}$  confirmed the aromatic C–H in-plain deformation appear from the syringyl units (Sun, et al., 1999a). The medium intensity at 852–837 $\text{cm}^{-1}$  that resulted from aromatic group C–H out-of-plain deformation vibration as confirmed previously (Sun, et al., 1999b). The FTIR results indicated the congruency between isolated lignin and the some previous reports in term of functional group content. Despite the structure of lignin not verified by NMR, nonetheless, a qualitative analysis by NMR cannot be made accurately owing to the isolated lignin insoluble in deuterated DMSO therefore, the content of isolated lignin is below the detection limit to perform this spectroscopical instrument. Apart from that, FTIR revealed that the absence of absorption peak of aliphatic C–H (2925–2850 $\text{cm}^{-1}$ ), celluloses (1620, 1430 and 1317–1315 $\text{cm}^{-1}$ ), hemicelluloses and silicates (1030–620 $\text{cm}^{-1}$ ), these non-lignin materials are mainly responsible for contamination of isolated lignin, so it clear demonstrating that the isolated lignin nearly pure.

### 3.3 Recyclability Test

Recently, ionic liquid have been shown recyclable properties up to several times by separation under reduced vacuum pressure (Feng & Chen, 2008). From Table 3, the recyclability test results showed that the first cycle of ionic liquid was very significant in isolate the lignin. The results exposed that second and third cycle are decreased lignin yield probably due to the reduction efficiency of ionic liquid. The mass balances of recycled ionic liquid also decrease because of loss during purification however, the decreasing was not very significant. The recyclability test indicated that ionic liquid was able to recycle and isolate lignin from rubber wood up to three times. Therefore,

ionic liquid is expected to be a potential solvent for industrial to isolate lignin owing to the recyclable properties as opposed to common acid and alkaline processes.

#### 4. Conclusions

Isolation of lignin from rubber wood in ionic liquid medium and the effect of concentration, duration and temperature on optimization of the isolation were studied. The rate of isolation is strongly influenced by isolation temperature conditions, which swollen fibres under elevated temperature conditions and assist to remove lignin from the fibres. It is also known that the isolation duration of the ionic liquid increased the lignin yield especially at high concentration which lignin easily bonded with free ionic liquid. It is possible to conclude that the yield of lignin isolated by ionic liquid highly depends to the isolation temperature, subsequently concentration of ionic liquid and isolation duration. The optimum parameters of isolation lignin by ionic liquid are 0.5 moles concentration, 120 minutes and 100°C given about 13.03wt.% of the quantity of solid lignin from rubber wood. The FTIR spectra data has shown functional group isolated lignin are on par with previous studies, FTIR spectra were also found isolated lignin are nearly pure because it shown no discernible of absorption peak of celluloses, hemicelluloses, silicates and other non-lignin materials spectra suggested that it is free from other organic compounds.

Noticeably, the main advantages of ionic liquid over acid and alkaline isolation, is simple to use in isolation process, the lignin of the rubber wood can be easily precipitated and isolated without further neutralization so, it can speed up production of lignin. In conclusion, compared with the conventional isolation by acid and alkaline, ionic liquid can be used as low-cost isolation method hence, this study indicated the approach probably can be applied economically to large-scale processes for the industrial application. Besides, they are very effective due to the recyclable behaviour and environmental friendly. As already mentioned, for acid and alkaline isolation, they have more lignin yields mainly caused by some hydrolyzation of cellulose, present of impurities and other organic compounds that contaminated their yields. These findings further verified ionic liquid are potential chemicals used as inert solvent based for isolation of rubber wood lignin since it possess many benefits than other delignifying agent based on acid and alkaline. For this reason, it is therefore likely that the convenient method proposed in this study may be preferred for most studies on lignin although a more complete study of lignin and ionic liquid interaction is required. These data will be used later in producing aromatic compounds from the isolated lignin.

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Table 1. The yields of lignin from rubber wood isolated by ionic liquid, acid and alkaline

	Ionic liquid isolation	Acid isolation	Alkaline isolation
Yield (wt.% in rubber wood lignin)	13.03	22.15	18.56
Fibre related in the isolated lignin (wt.%)	78.45	64.22	74.01
Other water soluble compounds (wt.%)	8.52	13.63	7.43

Table 2. Assignment of IR bands in the spectra

Wavenumber (cm <sup>-1</sup> )	Functional group assignment	Intensity
3420	hydroxyl vibrations	strong
2860–2850	methoxyl groups	medium
1740–1660	carbonyl stretching	strong
1715	C–O nonconjugated	medium
1712	carboxyl groups	strong
1640	carbonyl stretching conjugated with aromatic ring skeleton	strong
1610–1503, 1422	C–C bounds of aromatic ring skeleton vibrations	strong
1500–1400	rings conjugated with carbonyl group	medium
1462–1420, 743	C–H deformations and aromatic ring skeleton vibrations	medium
1331–1325	syringyl	strong
1270–1260	guaiacyl	strong
1230–1215	aromatic phenyl C–O stretching	medium
1137–1033	aromatic C–H in-plane deformation	strong
852–837	aromatic C–H out-of-plane deformation	medium

Table 3. The yields of isolated lignin from rubber wood and mass balances of recycled ionic liquid

	First cycle	Second cycle	Third cycle
Yield (wt.% in rubber wood lignin)	13.03	12.36	10.57
Mass balances (wt.%)	85.79	80.35	76.97

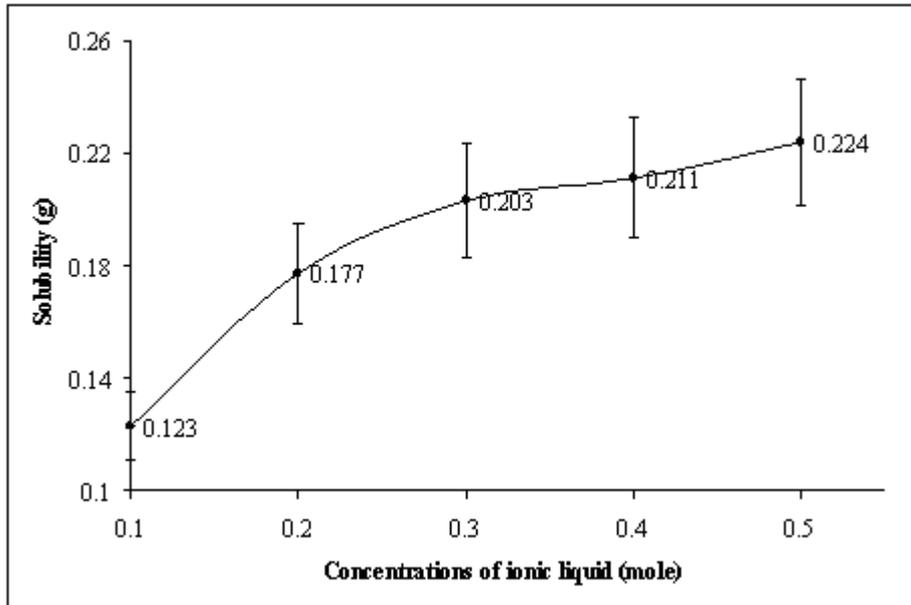


Figure 1. Solubility of lignin yields from rubber wood in ionic liquid at room temperature (25°C) in different isolation concentration at 30 minutes

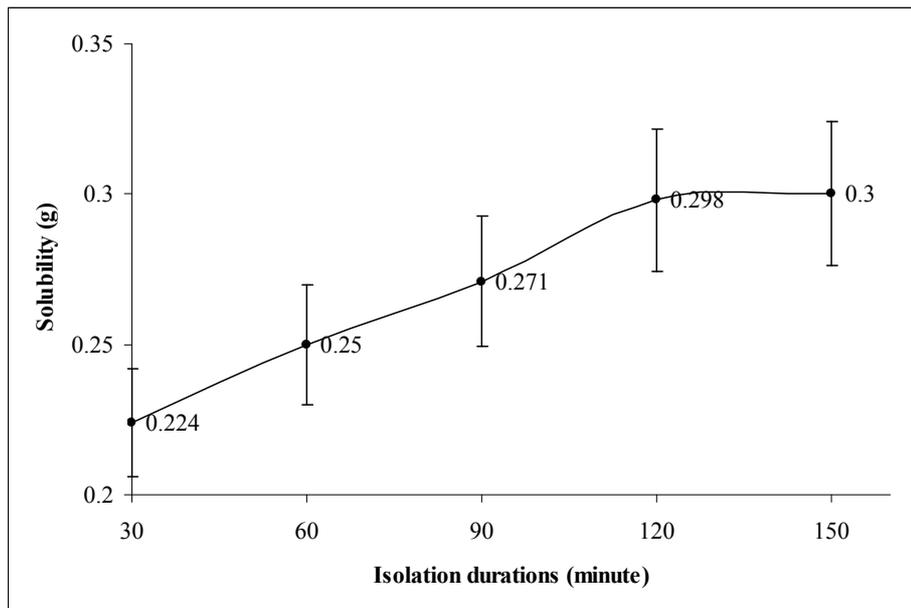


Figure 2. Solubility of lignin yields from rubber wood in ionic liquid at room temperature (25°C) in different isolation durations at 0.5 moles of ionic liquid

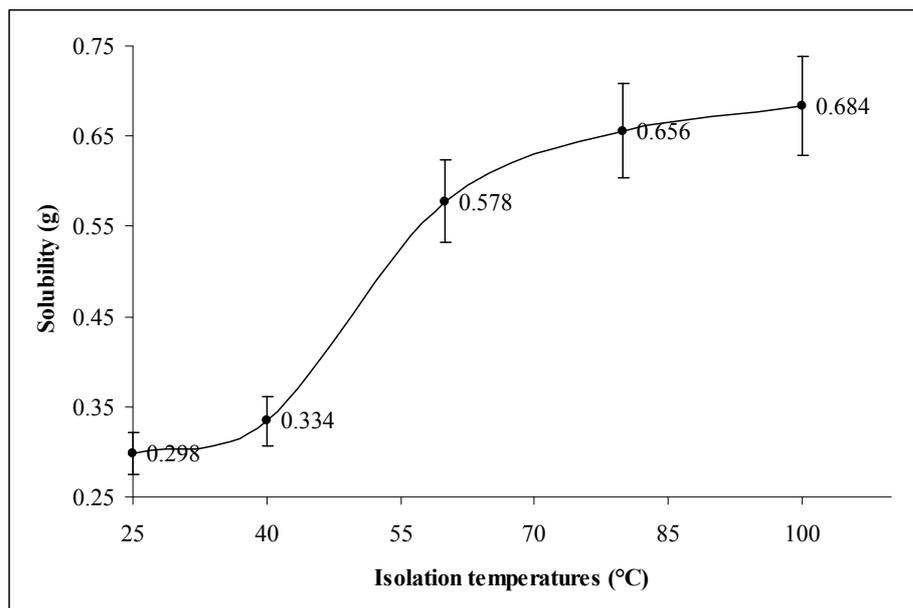


Figure 3. Solubility of lignin yields from rubber wood in ionic liquid at 0.5 moles of ionic liquid in different isolation temperatures at 120 minutes