



## **The Effect of Imidazolium-Based Ionic Liquids on the Tensile Properties of Gellan Gum/KCF Biocomposite Films - A Prelude Study**

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### **Authors' contributions**

*This work was carried out in collaboration among all authors. Authors AAS and KA designed and conceived the study. Author AAS performed most of the experiments. Authors AAS and TK collected and analyzed the data. All authors contributed to the writing of the manuscript, read and approved the final manuscript.*

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### **ABSTRACT**

In this prelude study, the gellan gum/kenaf core fiber (KCF) biocomposite films were fabricated with the addition of imidazolium-based ionic liquids such as 1-butyl-3-methylimidazolium chloride (Bmim Cl), 1,3-dimethylimidazolium methylsulphate (Dmim MeSO<sub>4</sub>), 1-ethyl-3-methylimidazolium acetate (Emim Ac), and 1-ethyl-3-methylimidazolium trifluoromethanesulfonate (Emim OTf). The fabrication of the biocomposite films was carried out by mixing KCF and the ionic liquids in an aqueous solution, followed by dissolution of gellan gum in the same solution at a temperature of 90°C. The mixed solution was gelled at room temperature, and the formed gel was dried in an oven at 40°C for 48 hours to obtain a freestanding biocomposite film. The fabricated films were characterized by using a universal testing machine to acquire tensile properties. The tensile test results indicated that the biocomposite film added with Emim Ac possesses a higher tensile extension at maximum (up to 24%) than the biocomposite film without ionic liquid (added with glycerol). In addition, the

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biocomposite film added with Emim OTf has a higher tensile modulus at maximum (up to 758%) compared to the biocomposite film without ionic liquid. In conclusion, the tensile properties of the gellan gum/KCF biocomposite films can be improved with the addition of Emim-based ionic liquids with different counter anions.

*Keywords: Ionic liquid; gellan gum; kenaf core fiber; biocomposite; film.*

## 1. INTRODUCTION

Ionic liquids are salts that have a lower melting point than the boiling point of water, thanks to their bulky cations and/or anions [1,2]. They are non-volatile since they have a very low vapor pressure [3,4]. Ionic liquids are commonly regarded as environmentally friendly solvents because of their recyclability [5,6]. They can be recycled by removing unwanted solvent through evaporation under reduced pressure, followed by freeze-drying under vacuum [7,8]. Moreover, ionic liquids have remarkable solvent properties [9,10] such as high stability, non-flammability, high polarity, good electrical conductivity, high thermal stability [11,12], and good miscibility with many organic solvents, including water [13,14]. They also have the capability to dissolve most organic and inorganic materials, as well as some biopolymers [15,16]. Besides, ionic liquids can be customized according to the desires of the applications [17,18].

Preceding studies have proven that the ionic liquids can be employed as solvents [19,20], plasticizing agents [21,22], compatibilizers [23,24], and coupling agents [25,26] for improving the thermomechanical properties of polymer blends and polymer composites. In addition, ionic liquids are also capable of interacting with many organic and inorganic compounds, including polymers, biopolymers, and natural fibers [27,28]. Therefore, they could provide intermolecular interactions between component phases of polymer blend and polymer composite systems. Among numerous types of ionic liquids, imidazolium-based ionic liquids were selected for investigation in this prelude study. This is because they are widely used, easily found, reasonable price, and non-flammable [29]. Moreover, the most important feature of imidazolium-based ionic liquids is that they have higher stability compared to the pyrrolidinium- and piperidinium-based ionic liquids [30].

On the other hand, biocomposite films have been received considerable attention from academics and industries [31,32]; this is because they can

simply be disposed of without polluting the environment [33,34]. Furthermore, the production cost of the films can be reduced [35,36] through the consumption of natural fibers as fillers [37,38]. The use of natural fiber such as kenaf core fiber (KCF) as a filler in various polymer composites has been extensively studied [39,40] because it possesses several beneficial properties, for example, abundant in nature, low-cost, non-toxic, environmentally friendly, and renewable as well [41,42]. On top of that, biocomposite films can be fabricated using a biopolymer like gellan gum as a polymer matrix. The utilization of gellan gum for such films is advantageous due to this biopolymer typically owns some useful properties, for instance, non-hazardous, natural abundance, renewable, biodegradable, and so on. Additionally, gellan gum is frequently used as a gel, and the gel can effortlessly form a freestanding film upon dried [43,44].

Furthermore, the fabricated gellan gum/KCF biocomposite film could possibly be used for preparing biodegradable film. Nevertheless, the biocomposite film is quite fragile, inflexible, and brittle, as perceived in this prelude study causing its usage to be constrained. Thus, imidazolium-based ionic liquids are added to investigate their effect on the tensile properties of the biocomposite films. Moreover, the study regarding the addition of ionic liquids for improving the tensile properties of the biocomposite films has not yet been reported so far. Additionally, the gellan gum/KCF biocomposite films added with imidazolium-based ionic liquids may potentially be applied in the production of eco-friendly films for barring dimensions, dividing sections, holding things, wrapping substances, and so forth.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Gellan gum, 1-butyl-3-methylimidazolium chloride (Bmim Cl) ( $\geq 98\%$ ), 1-ethyl-3-methylimidazolium acetate (Emim Ac) (97%), and 1-ethyl-3-methylimidazolium trifluoro-

methanesulfonate (Emim OTf) ( $\geq 98\%$ ) were purchased from Sigma-Aldrich (M) Sdn. Bhd., Malaysia. Glycerol ( $\geq 99\%$ ) [45] was procured from Fisher Scientific (M) Sdn. Bhd., Malaysia. 1, 3-dimethylimidazolium methylsulphate (Dmim MeSO<sub>4</sub>) (97%) was obtained from Merck (M) Sdn. Bhd., Malaysia. KCF (420  $\mu\text{m}$ ) was acquired from the National Kenaf and Tobacco Board (NKTB), Malaysia. All obtained materials were used without any further modification [46]. Chemical structures of glycerol, Bmim Cl, Dmim MeSO<sub>4</sub>, Emim Ac, and Emim OTf are shown in Fig. 1.

## 2.2 Fabrication of Gellan Gum/KCF Biocomposite Films Added with Ionic Liquids

The biocomposite films were fabricated by pouring 2.4 g of ionic liquid into a 100 mL beaker, followed by 0.28 g of KCF and 56 g of distilled water. They were then stirred using a magnetic stirring apparatus. 1.6 g of gellan gum was gradually added to the mixture, followed by heating at 90°C and stirring at 1500 rpm until all gellan gum dissolved [43]. The mixed solution was immediately cast into a 14 cm internal diameter glass Petri dish and allowed to cool to room temperature (25°C) for the gelation process [17]. Later the gel was slowly dried in an oven at a temperature of 40°C for 48 hours [25] to obtain a freestanding biocomposite film. The content of KCF was fixed to 15 wt. % relative to the weight of gellan gum, while the weight ratio of gellan gum and ionic liquid was fixed in 2:3. The gellan

gum/KCF biocomposite film added with glycerol at the same weight ratio was also fabricated for comparison reason.

## 2.3 Tensile Test of Biocomposite Films

The tensile properties such as tensile extension at maximum and tensile extension at break, as well as tensile stress and tensile modulus at maximum were measured according to the ASTM D638-10 [47,48] by using a universal testing machine, UTM (Instron, model 5567, Norwood, MA, USA) equipped with a 30 kN load cell [49,50]. The crosshead speed was 5 mm/min with a 30 mm gauge length [43]. The tensile test was carried out in the temperature range of 21 to 25°C and a relative humidity range of 40 to 60%. Ten samples from each biocomposite film were tested to determine the mean values.

Before testing, the biocomposite films were cut by using a die cutter into a rectangular shape with a length and width of 60 mm and 12.7 mm, respectively. The film samples were conditioned in an oven at a temperature of 40°C for at least 24 hours prior to the tensile test [51]. The test results were analyzed by utilizing a Microsoft® Excel® [52] of Microsoft 365 statistical software through a single-factor analysis of variance (ANOVA) technique. A statistically significant difference in the tensile extension, tensile stress, and tensile modulus mean values of the biocomposite films was ascertained at a 95% confidence level [53,54].

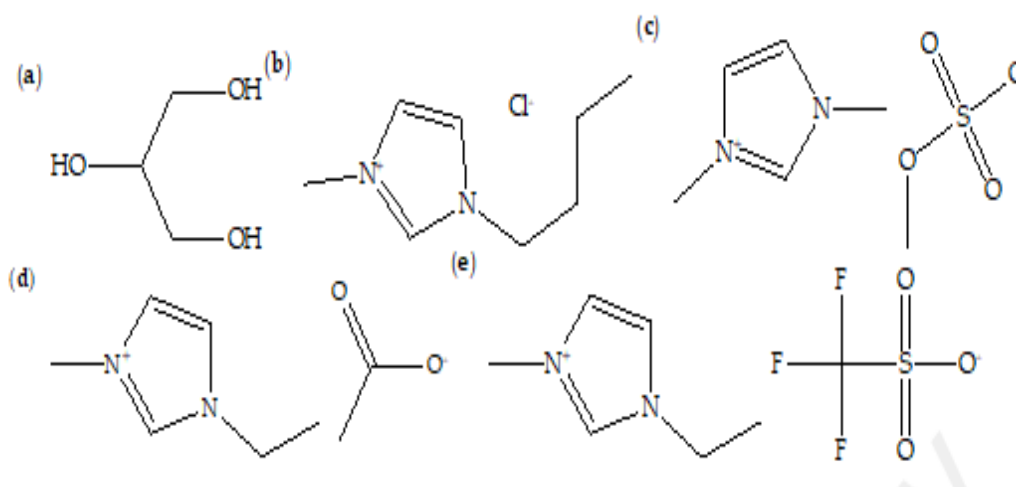


Fig. 1. Chemical structures of (a) glycerol, (b) Bmim Cl, (c) Dmim MeSO<sub>4</sub>, (d) Emim Ac, and (e) Emim OTf

### 3. RESULTS AND DISCUSSION

Fig. 2(a) and (b) demonstrate the tensile extension at maximum and tensile extension at break of the gellan gum/KCF biocomposite films without and with the addition of ionic liquids. It can be seen that the tensile extension at maximum of the biocomposite film without ionic liquid (added with glycerol) is lower than the biocomposite films added with Bmim Cl and Emim Ac ionic liquids. This indicated that the capability of the ionic liquids to increase the elongation property of the biocomposite films is much better in comparison with glycerol. The substantial increase of the tensile extension is due to the plasticizing effect of Bmim Cl and Emim Ac that could act as efficient plasticizers for the biocomposite films. Furthermore, the

addition of Emim Ac increased the tensile extension at maximum of the biocomposite film up to 24% compared to glycerol. Nevertheless, the tensile extension at maximum of the biocomposite films added with Dmim MeSO<sub>4</sub> and Emim OTf are lesser in comparison with the biocomposite films added with glycerol, Bmim Cl and Emim Ac. This is because the Dmim MeSO<sub>4</sub> and Emim OTf could not act as plasticizers for the biocomposite films. Therefore, the addition of imidazolium-based ionic liquids is not necessarily linked to the increase of the tensile extension of the gellan gum/KCF biocomposite films. On the other hand, the tensile extension at break result exhibited a similar trend as the tensile extension at maximum result, whereby the highest tensile extension at break was also found at the biocomposite film added with Emim Ac.

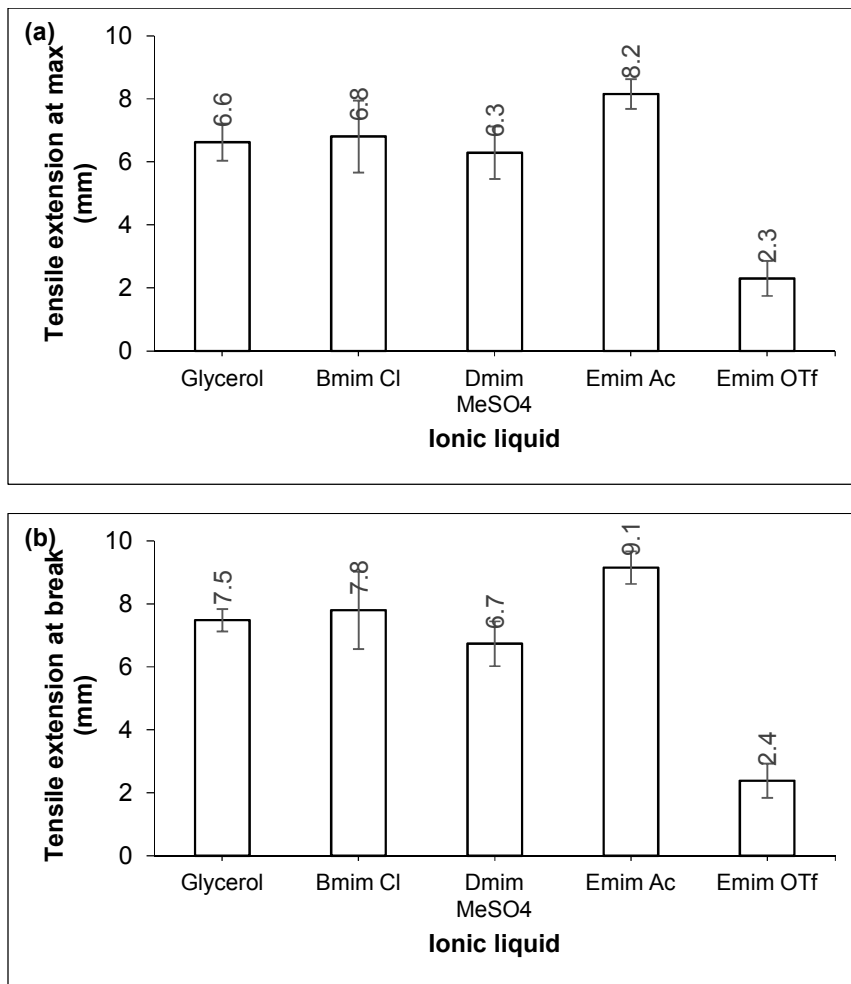


Fig. 2. (a) Tensile extension at maximum and (b) tensile extension at break of the gellan gum/KCF biocomposite films without and with the addition of ionic liquids

Fig. 3(a) and (b) show the tensile stress and tensile modulus at maximum of the gellan gum/KCF biocomposite films without and with the addition of ionic liquids. It can be perceived that the tensile stress of the biocomposite films increased with the addition of Dmim MeSO<sub>4</sub> and Emim OTf ionic liquids. A considerable increase could be seen for the biocomposite film added with Emim OTf, whereby the increase is up to 97% compared to glycerol. A similar trend was also detected for the tensile modulus, which increased up to 758% with the addition of Emim OTf. The obtained results displayed that the addition of certain imidazolium-based ionic liquids could also improve the stiffness and rigidity characters of the biocomposite films. However, in the previous study [21], the addition of Emim OTf had decreased the tensile stress and tensile modulus of the HDPE/KCF biocomposites but increased the tensile

extension. Hence, the Emim OTf ionic liquid can behave quite differently in gellan gum/KCF and HDPE/KCF biocomposites. This demonstrated that the different polymer matrices for the biocomposites added with Emim OTf had provided distinct improvements in the tensile properties. On top of that, the tensile stress and tensile modulus of the biocomposite films decreased with the addition of Bmim Cl and Emim Ac ionic liquids. This is due to the decrease in stiffness of the biocomposite films, which is caused by the increase of extensibility. From the results, it can be noticed that the different types of imidazolium-based ionic liquids could affect the tensile properties of the biocomposite films. Additionally, the significant improvement and decrement could be sighted for the biocomposite films added with Emim-based ionic liquids.

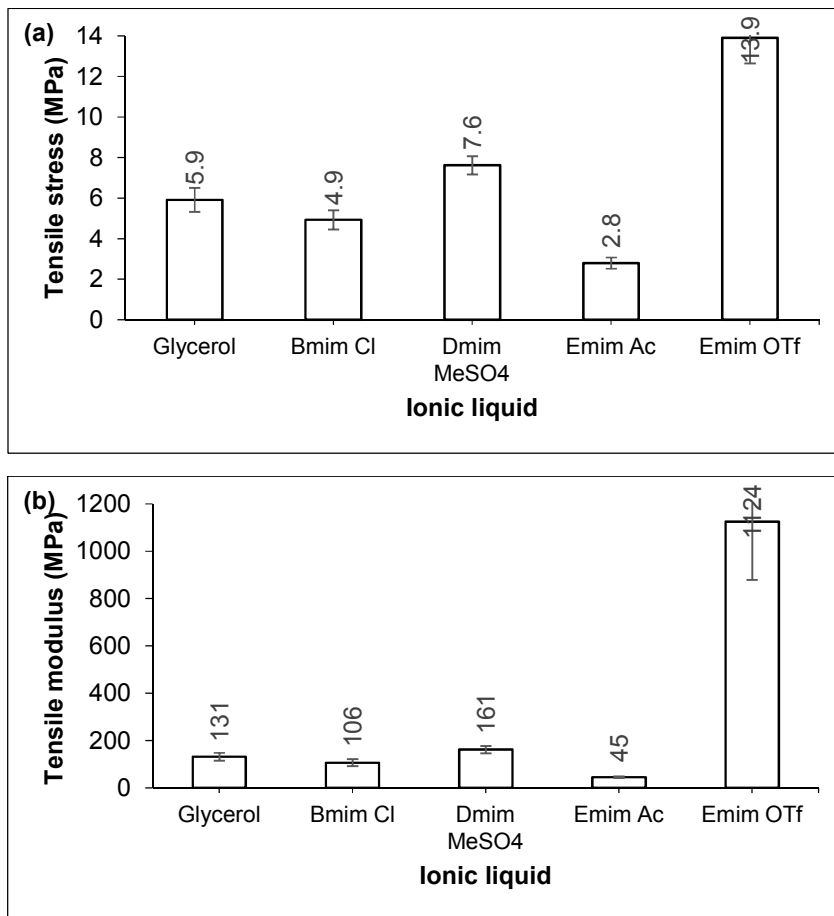


Fig. 3. (a) Tensile stress and (b) tensile modulus at maximum of the gellan gum/KCF biocomposite films without and with the addition of ionic liquids

**Table 1. Single-factor ANOVA results of the tensile properties of the gellan gum/KCF biocomposite films without and with the addition of ionic liquids**

<b>Tensile</b>	<b>Source of variation</b>	<b>SS</b>	<b>df</b>	<b>MS</b>	<b>F</b>	<b>P-Value</b>
Extension at maximum	BG	194.2735389	4	48.56838473	84.58199997	$2.4335 \times 10^{-20}$
	WG	25.83974502	45	0.574216556	-	-
Extension at break	BG	264.7117848	4	66.1779462	122.0646824	$1.4985 \times 10^{-23}$
	WG	24.39696331	45	0.54215474	-	-
Stress at maximum	BG	711.8691013	4	177.9672753	366.7393717	$1.0379 \times 10^{-33}$
	WG	21.83710833	45	0.485269074	-	-
Modulus at maximum	BG	8296666.566	4	2074166.642	169.746806	$1.5767 \times 10^{-26}$
	WG	549863.0643	45	12219.17921	-	-

SS = sum of square, df = degree of freedom, MS = mean square, F = F-value, Number of samples = 5, Number of observations = 50

Statistical analysis was conducted by using a single-factor analysis of variance (ANOVA) to discover statistically significant differences in the tensile properties between the different biocomposite film samples. Table 1 exhibits the single-factor ANOVA results of the tensile properties of the gellan gum/KCF biocomposite films without and with the addition of ionic liquids. The total numbers of the film samples were five, and ten replicates were tested for each sample. The source of variation of the tensile properties had been divided into two categories, namely, between groups (BG) and within groups (WG). F-value is the ratio of the mean square of BG to the mean square of WG. The P-values were less than 0.05 in the Table 1, which rejected the zero hypothesis. Therefore, it can be deduced that there are statistically significant differences in the tensile properties among the different biocomposite film samples at a 95% confidence level.

#### 4. CONCLUSIONS

In this prelude study, the tensile extension at maximum of the biocomposite film significantly increased up to 24% with the addition of Emim Ac compared to glycerol. Besides that, the tensile modulus at maximum of the biocomposite film substantially increased up to 758% with the addition of Emim OTf. Thus, the elongation and stiffness properties of the biocomposite films have been improved with the addition of Emim Ac and Emim OTf, respectively. It can be concluded that the addition of Emim-based ionic liquids with different counter anions could enhance not only tensile extension, but also tensile stress and tensile modulus of the gellan gum/KCF biocomposite films.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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