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Effect of multi walled carbon nanotube (MWCNT) and mixing conditions on unsaturated polyester (UP) physical properties

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Abstract. Unsaturated polyester resins (UP) are widely used for thermoset composite application, but to increase its mechanical and physical properties, fillers are required. In this study, UP was mixed with multi-walled carbon nanotube (MWCNT) using mechanical stirrer to improve the mass change after water absorption (WA) and the value of coefficient of friction (COF). The MWCNT was varied at 0.3 and 0.6 wt%, the stirring parameters were also varied with the mixing time at 30 and 90 minutes, with mixing speed at 500 and 1500 rpm, respectively. The results showed that the mass change after WA test giving 41% increase and COF improved 100%. The improvement on WA and COF might be influenced by high shear energy density, produced by high mixing speed, which might facilitate complete separation of MWCNT.

1. Introduction
UPs are extremely versatile in properties, applications and have been a popular thermoset used as polymer matrix in composites [1]. However, thermoset can not cure before heated. Therefore, an accelerator or promoter is needed to accelerate curing process [2]. UP has lower water absorption (WA) value than epoxy [3], but if it mixed with wrong filler, the WA property can be decreased [4]. Therefore the right filler must be used to get the best WA property.

One filler that can improve the WA in UP is multi-walled carbon nanotube (MWCNTs) better than glass fiber, plant fiber and fur [4-7]. Moreover, previous studies reported that MWCNT could improve UP’s coefficient of friction (COF) values [5]. The improvement COF caused by its sliding interface mechanism [8] and the improvement WA caused by its barrier performance (hydrophilic behavior) [9]. The improvement of these properties also depends on the preparation parameters [10].

There are various methods to prepare MWCNT composites, such as solution mixing, bulk mixing, melt mixing and in situ polymerization [10]. The solution mixing method is more efficient than others because multi-walled carbon nanotubes dispersing in composite can be done directly. Moreover, the solution processing method is the best method when the product is in the form of paste on solution. There are two ways that are considered in solution mixing, namely mechanical stirrer and sonication...
In mechanical stirrer, the parameter, time and speed, could be set, while in sonication, only the time could be set [11]. The mixing process is important to discuss, because the dispersion of MWCNT can influence the composite properties.

Due to this reasons, the research of UP/MWCNT composite is necessary to be done. In this research the effect of composition percentage of MWCNT and mixing condition will be investigated by using mechanical stirrer to produce the UP 150 HRN-EX/MWCNT composites with the best properties.

2. Experimental method

2.1. Materials
UP 150 HRN-EX, Methyl Ethyl Ketone Peroxide (MEKP) and Cobalt Naphtenatel (Co-NAP) were used as matrix, catalyst and accelerator/promoter respectively and purchased from PT. Justus Sakti Raya, Indonesia. MWCNT with outer diameter 30 - 50 nm and length 10 - 20 µm, purity >95wt% and ash <1.5wt% was supplied by Cheaptube.

2.2. Preparation of nanocomposites
The variation of MWCNT that used was 0.3 wt% and 0.6 wt%; and they were identified as low and high composites respectively. Initially, UP and MWCNT were mixed manually for 1 minute, then continued by solution mixing in mechanical stirrer. Mechanical stirrer was set at 500 rpm as the lowest speed and 1500 rpm as the highest speed and mixing time for 30 minutes as the low duration and 90 minutes as the high duration with various compositions (Table 1) [12, 13]. Co-NAP was then added and mixed manually for 10 seconds. MEKP was also added after that, and mixed manually for 10 seconds. The solution was poured into mold with a dimension 30 x 30 x 0.3 cm.

2.3. Testings
WA test was carried out in Water Bath Gesellschaft fur Labortechnik mbH D-30938 Burgwedel typ 1004 no 10966099 I made in Germany based on ISO 62 standard. The temperature and soaking duration were set at 23°C and 24, 48, 96, 192 hours. COF test was conducted in an OTS FOOR societa per azioni vignate–mi–italy s/n: 20006498 according at ASTM D 1894 standard.

3. Results and discussion
Table 1 shows the WA and COF test results related to MWCNT compositions and stirring conditions (duration and stirring speed). The lower value of the increased weight represents the better property of WA and the higher value of COF exhibits the better anti-slip property that could be found in the nanocomposites [3-14]. The lowest value of weight change is (0.48 ± 0.08) %, or 41% reduction on WA compared to the control value. Moreover, in COF testing, the highest value COF is 0.29 ± 0.010 or 100% increasing compared to the control value.

The addition of increasing CNT composition is related to the higher filler in the materials, as a result, the dispersion might not spread evenly. That phenomenon did not cause any effect on increasing property of WA and COF, which could be observed in WA and COF test results that the increasing CNT composition which is caused by the immersion does not indicate the significant decreasing value. Therefore, the CNT composition is not a significant factor to increase the water resistant and anti-slip property. The results of this study agreed well with previous studies [15, 16]. However, there are another factor that can affect WA and COF property, such as mixing speed and duration [17].

The higher the speed and the higher the mixing time, the lower the WA and COF value. The value of stirring condition given depends on the value of CNT compositions that were applied. The higher amount of CNT composition which was used exhibited the higher amount of stirring condition given [18].

Figure 1 shows MWCNT composition decreases corresponding to the increased stirring duration, the increased weight which shows the reduction indicates the cause of shear stress given that could
disintegrate the bonds of CNT agglomeration and disperse the CNT by visual looking. Otherwise, in a high condition, shear stress cannot disintegrate the bonds of CNT agglomeration, however the higher amount of CNT composition causes the agglomeration that resists the water to be absorbed. That condition also occurred in a high stirring speed in both CNT composition, which could be observed from the test result that was not overly distinct. The increased water absorption is caused by the reduction of CNT length or can be identified as the damage of the CNT bonds [19].

**Table 1.** Water absorption and COF test results.

| MWCNT Composition [wt%] | Speed [rpm] | Time [min] | Sample (MWCNT_Speed_Time) | Weight Change (%) | COF |
|-------------------------|-------------|------------|---------------------------|-------------------|-----|
| Control                 | 500         | 30         | Low_Low_Low               | 0.71 ± 0.10       | 0.029 ± 0.010 |
|                         | 90          | 30         | Low_Low_High              | 0.48 ± 0.08       | 0.005 ± 0.003 |
|                         | 1500        | 30         | Low_High_Low              | 0.52 ± 0.07       | 0.014 ± 0.005 |
|                         | 90          | 30         | Low_High_High             | 0.57 ± 0.01       | 0.031 ± 0.001 |
| 0.6                     | 500         | 30         | High_Low_Low              | 0.70 ± 0.10       | 0.004 ± 0.005 |
|                         | 90          | 30         | High_Low_High             | 0.64 ± 0.07       | 0.012 ± 0.011 |
|                         | 1500        | 30         | High_High_Low             | 0.63 ± 0.10       | 0.002 ± 0.004 |
|                         | 90          | 30         | High_High_High            | 0.68 ± 0.21       | 0.012 ± 0.005 |

**Figure 1.** Weight change vs mixing time during water absorption test, (a) low CNT composition, (b) high CNT composition.

**Figure 2.** COF value vs mixing time, (a) low CNT composition, (b) high CNT composition.
Figure 2(a) represents the similar condition with the Figure 1(a), which shows the CNT has been evenly dispersed that can increasing WA but decreasing COF. The other factor which can affect COF value is the rubbed surface, the more slippery the surface that rubs on the sample causes the lower COF value. The second factor which affects COF value is CNT direction towards the direction of friction, if the CNT direction is perpendicular to the direction of friction, then the COF value will increase [20].

Figure 2(b) exhibits the increasing result and does not show any significant difference. That phenomenon was indicated by the higher number of added filler that causes the molecular weight increased by the agglomeration. The external power of blade rotation and stirring duration does not generate shear stresses which can disintegrate filler aggregates, so that the dispersion does not evenly spread and may cause the homogeneous size [19]. When the CNT composition and stirring duration are similar, but the stirring speed is dissimilar, that causes the decline at the high stirring speed. That phenomenon is caused by the agglomerated CNT which binds the bigger energy than the shear energy density which generates activation energy barrier so that the exfoliation occurs temporary and decreasing that value.

4. Conclusion
The fabrication of UP/MWCNT composite could be mixed together properly using a mechanical stirrer. To obtain a good property, it may need high supply energy density, low binding energy aggregates, and high fracture resistance individual of MWCNT. By increasing amount of MWCNT the stirring condition needs to be higher, so that MWCNT can be well dispersed. It decreased the water absorption, then increased COF value. It is concluded that UP/MWCNT nanocomposites have complex properties which depend on stirring conditions.

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