

Sains Malaysiana 47(6)(2018): 1123–1129
<http://dx.doi.org/10.17576/jsm-2018-4706-07>

Effects of Soaking in Glycerol and Conventional Solvents on the Grinding of *Kempas Wood (Koompassia malaccensis)*

(Kesan Rendaman dalam Gliserol dan Pelarut Konvensional pada Pengisaran Kayu Kempas (*Koompassia malaccensis*))

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ABSTRACT

Glycerol, the main by-product in biodiesel manufacturing, is a useful and environment-friendly solvent for many organic and inorganic substrates. This study investigates the effects of soaking using glycerol, silicone oil, dimethyl sulfoxide (DMSO) and Organosolv (1:1 water: ethanol) on energy consumption in the process of grinding kempas wood and on the particle size of ground kempas. Kempas wood chips were soaked in various solvents at 90°C for 1 h. The structural characterisation of untreated and treated kempas was analysed using CHNS, ATR-FTIR and XRD. Meanwhile, the grinding energy was calculated based on power per unit time while particle size was analysed using nested column sieves. Glycerol has high stability, good compatibility with other chemicals and is environmentally friendly. Glycerol-soaked kempas consumed less energy which led to energy saving of up to 0.015 W h and exhibited the smallest average particle size (263 µm) close to that of untreated kempas due to glycerol lubricating properties. Therefore, glycerol can be used as an alternative to conventional solvents in reducing the grinding energy consumption and particle size of lignocellulosic biomass.

Keywords: Glycerol; grinding energy; kempas woods; lignocellulose biomass; Organosolv

ABSTRAK

Gliserol, hasil sampingan utama dalam pembuatan biodiesel merupakan pelarut berguna dan mesra alam bagi kebanyakan substrat organik dan bukan organik. Kajian ini mengkaji kesan perendaman menggunakan gliserol, minyak silikon, dimetil sulfoksida (DMSO) dan Organosolv (1:1 air: etanol) terhadap penggunaan tenaga dalam proses pengisaran kayu kempas dan saiz zarah kempas kisaran. Serpihan kayu kempas direndam dalam pelbagai pelarut pada suhu 90°C selama 1 jam. Pencirian struktur ke atas kempas tidak dirawat dan dirawat dianalisis menggunakan CHNS, ATR-FTIR dan XRD. Manakala tenaga kisaran dikira berdasarkan kuasa per unit masa dan saiz zarah dianalisis menggunakan penapis kolum bersarang. Gliserol mempunyai kestabilan yang tinggi, keserasian yang baik dengan bahan kimia lain dan mesra alam. Kempas yang direndam dalam gliserol menggunakan tenaga yang lebih rendah dan membawa kepada penjimatan tenaga sehingga 0.015 W h dan mempamerkan purata saiz zarah yang paling kecil (263 µm) yang sangat hampir dengan kempas yang tidak dirawat disebabkan oleh sifat pelincir gliserol. Oleh itu, gliserol boleh digunakan sebagai pelarut alternatif kepada pelarut konvensional dalam mengurangkan penggunaan tenaga kisaran dan saiz zarah biojisim lignoselulosa.

Kata kunci: Biojisim lignoselulosa; gliserol; kayu kempas; Organosolv; tenaga kisaran

INTRODUCTION

Lignocellulosic biomass residue is one of the most abundant wastes in Malaysia and can be considered an important feedstock for biofuel application such as power generation and transportation. In a modern bio-refinery, lignocelluloses are converted through a number of different processes into a range of products, including bio-fuels, valuable chemicals, heat and electricity as stated by Binder and Raines (2010). In Malaysia, wood-based products can be obtained in large quantities. The commonly available woods are *kempas*, jelutong, jati, rubber wood, cengal batu, cengal emas and balau. In this study, *kempas* was used due to its popularity in the Malaysian timber industry,

especially as material for flooring, desirable structural properties and good treatability with wood preservatives (Wong 1988).

Adapa et al. (2011) and Zhu et al. (2010) found that the particle size reduction of biomass helps to increase the surface area, pore size and number of points of contact for inter-particle bonding in compaction operations such as densification process, which is an important step in the transformation of feedstock into bio-energy in the field of renewable energy obtained from renewable biomass-based resources. While from the studies conducted by Brandt et al. (2012) on the energy consumption during the grinding process, it was found that the wood pre-soaked in ionic

liquids or lubricants was able to be ground to a smaller particle size compared to the air-dried wood. However, there are still many challenges in putting ionic liquids into practical use due to their high cost and the need for regeneration techniques (Park & Kazlauskas 2003).

Glycerol (CH₂OH.CHOH.CH₂OH), the main by-product of biodiesel is a colourless, odourless, viscous and hygroscopic liquid with a high-boiling-point (290°C) (Da Silva et al. 2009). Glycerol is insoluble in hydrocarbons, moderately soluble in common solvents like ether and dioxane and completely soluble in water and alcohols (David & Henry 1996). Its solubility in water is due to the presence of three hydrophilic alcoholic hydroxyl groups of glycerol and formation of intra- and intermolecular hydrogen bonds between solvent (David & Henry 1996). In addition, Pagliaro and Rossi (2010) showed that glycerol is highly stable under normal storage conditions, compatible with many other chemicals, virtually non-irritating in its various uses and has no known negative environmental effects. Therefore, glycerol is widely used in the food and pharmaceutical industry and as a chemical intermediate as reported by Sun and Chen (2008).

Due to glycerol stability and negligible environmental effect, this study aimed to investigate the utilisation of cheap and easily available glycerol as a soaking agent. Besides, this study also intends to compare energy demand, particle size and crystallinity for wood grinding of wood chips with or without pre-soaking in glycerol or other conventional solvents such as dimethyl sulfoxide (DMSO), 1:1 water: ethanol mixture (Organosolv) and silicone oil.

MATERIALS AND METHODS

CHEMICALS AND MATERIALS

Glycerol, silicone oil, DMSO, ethanol and methanol were purchased from Sigma-Aldrich and used as received. The lignocellulosic biomass used in this study was *kempas* (*Koompassia malaccensis*) provided by the Forest Research Institute of Malaysia (FRIM).

PREPARATION OF *KEMPAS* WOOD CHIPS

Kempas was cut into wood chips with an average size of 1 × 0.1 × 0.5 cm. The wood chips were then dried overnight in an oven at 105°C and kept in a desiccator before use.

SOLVENT-SOAKING OF WOOD CHIPS

The wood chips (1.00 g) were placed in four wide-mouthed Pyrex culture tubes with screw caps and 10 mL of glycerol, silicone oil, DMSO and Organosolv were added into each tube, respectively. The tubes were placed in an oven and kept at 90°C for 1 h (Brandt et al. 2012). After that, they were allowed to cool down to room temperature.

STRUCTURAL CHARACTERIZATION

CHNS instrument (Thermo-Finnigan/EA1112) was used specifically for simultaneous determination on the amount of carbon, hydrogen, nitrogen and sulphur elements present in the soaked *kempas* wood chips. Meanwhile, the attenuated total reflectance of Fourier transform infrared (ATR-FTIR) was performed using Perkin Elmer Spectrum 400 to observe the appearance and disappearance of the functional group present in the untreated and treated woods chips. About 5 mg of the soaked *kempas* wood chips were placed on the diamond window. Scans were conducted in the range of 650 – 4000 cm⁻¹ band.

To identify the crystallinity of untreated and treated *kempas* wood, X-ray diffraction (XRD) was performed using a D8-Advance (BRUKER, Germany) XRD system. Patterns were collected from 10 to 60 (2) with a step size of 0.025 at 8 deg/min. Crystallinity index (CrI) of the samples was computed by using (1) (Segal et al. 1959).

$$\text{CrI} = \frac{I_{002} - I_{\text{am}}}{I_{002}} \times 100 \quad (1)$$

where I_{002} is the maximum intensity of crystalline portion in the sample at about $2\theta = 22.6^\circ$ and I_{am} is the intensity attributed to the amorphous portion of *kempas* soaked samples at $2\theta = \sim 16$.

MEASUREMENT OF ENERGY CONSUMPTION

The wood chips were weighed prior to grinding using a grinder (Model: DF-15; Voltage: 110 V/60 Hz). Each sample was ground for 5 s as this was sufficient to grind the untreated wood chips into powder as shown by Brandt et al. (2012). The energy consumption (E_c) was calculated as the energy E in joules (J) consumed during 1 h per g of sample, divided by the time period t in seconds (s) according to (2). The energy saved (E_s) was calculated relative to the energy requirement of untreated *kempas* wood according to (3), where $E_{\text{untreated}}$ is the average energy required to grind 1 g of the untreated and dried wood chips.

$$E_c, (\text{W h}) = \frac{E(\text{J})}{t(\text{s})} \quad (2)$$

$$E_s, (\text{W h}) = E_{\text{Untreated}} - E_c \quad (3)$$

PERCENTAGE OF GROUND *KEMPAS* PASSING THROUGH THE SIEVE

The percentage of ground *kempas* soaked with various solvents that passed through the sieves after grinding was calculated according to (4).

$$\text{Percent passing (\%)} = \frac{\text{Weight of ground kempas passed through the sieve (g)}}{\text{Weight of kempas wood chips after soaking (g)}} \times 100\% \quad (4)$$

PARTICLE SIZE ANALYSIS

After grinding, the wood chips were soaked overnight in 20 mL methanol as indicated in the study by Brandt et al. (2012). They were then filtered and rinsed again with methanol and left to air-dry overnight. Afterwards, the powder was passed through a nested column of sieves with decreasing pore sizes (1000-125 μm) by shaking on a vibratory sieve shaker for 8 min. The percentage weight of material retained by each sieve was measured and from this data, the log-normal distribution mass median diameter or average particle size (D_{50}) was calculated, i.e. 50% of the smaller particle size of the sample by mass. It was used in this paper as a measure of the average particle size and calculated by linear interpolation as presented by Davis (2009) using (5).

$$\text{Percent passing (\%)} = \frac{\text{Weight of ground kempas passed through the sieve (g)}}{\text{Weight of kempas wood chips after soaking (g)}} \times 100\% \quad (5)$$

where x_1 and x_2 are the pore sizes of the sieves which allowed just less than and more than 50% of the sample to pass through by weight, respectively, y_1 and y_2 are the percentages of material passing through those sieves.

RESULTS AND DISCUSSION

PHYSICAL APPEARANCE OF GROUND *KEMPAS*

Figure 1 shows the images of ground and untreated *kempas* after soaking in different solvents, respectively. It can be seen that there was no significant difference in terms of particle size. However, the colour intensity of soaked *kempas* was different than the untreated *kempas*. Glycerol, organosolv and DMSO soaked *kempas* gave a lighter brown colour while silicon oil soaked *kempas* had darker brown colour than the untreated *kempas*. This corresponded to the amount of solvent absorbed (Table 3) where DMSO soaked *kempas* was the highest, followed by glycerol, organosolv and silicone oil. The amount of absorbed solvent correlated to the leaching of *kempas* wood gave the lighter brown colour. Silicon oil absorbed by *kempas* was the lowest 0.042 mL, therefore, silicon oil only wetted or lubricated the surface of *kempas* wood and gave it the darker brown colour rather than leaching.

STRUCTURAL CHARACTERIZATION OF SOAKED *KEMPAS* WOOD CHIP

All woods are composed of cellulose (40% - 50%), hemicellulose (15% - 25%) and lignin (15% - 30%) and

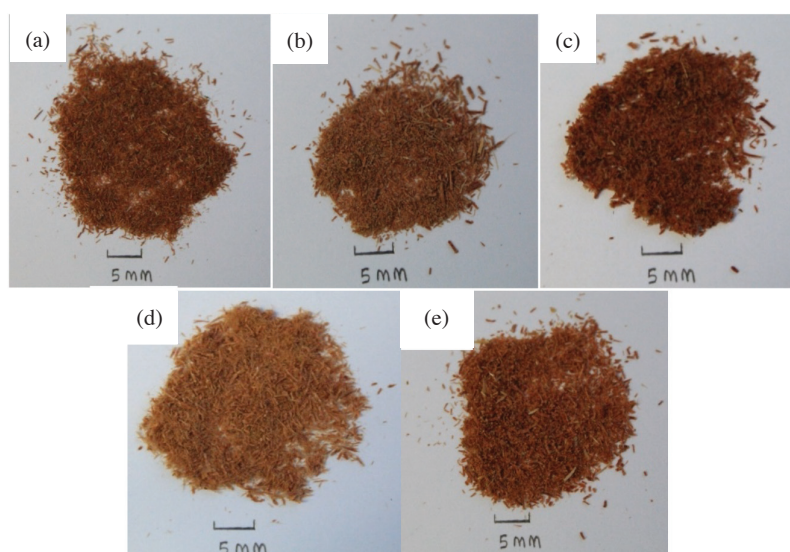


FIGURE 1. (a) Untreated ground *kempas* and ground *kempas* after soaking at 90°C for 1 h with various solvents: (b) glycerol, (c) silicon oil, (d) DMSO and (e) Organosolv

TABLE 1. CHNS analysis data of untreated *kempas* wood and treated *kempas* wood soaked in glycerol, DMSO, silicon oil and organosolv (ethanol/water)

Treatments	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Sulphur (%)
Untreated	44.64 \pm 3.69	6.46 \pm 0.68	0.93 \pm 0.28	0.04 \pm 0.02
Glycerol	42.51 \pm 2.68	6.39 \pm 0.23	1.44 \pm 0.36	0.23 \pm 0.05
DMSO	42.70 \pm 1.30	6.28 \pm 0.28	1.44 \pm 0.34	3.19 \pm 0.11
Silicon Oil	42.22 \pm 0.41	6.38 \pm 0.49	1.42 \pm 0.17	0.26 \pm 0.03
Ethanol/H ₂ O	41.42 \pm 4.32	5.90 \pm 1.26	1.04 \pm 0.16	0.23 \pm 0.05

basically contain elements such as carbon, hydrogen, nitrogen, sulphur and other elements. CHNS analysis was applied to identify whether the soaking of *kempas* wood in various solvents could lead to a chemical modification. From Table 1, the CHNS data analysis shows that there was no significant difference between the treated and untreated *kempas* wood in terms of carbon, hydrogen, nitrogen and sulphur content except for the sulphur content in DMSO-soaked *kempas* wood. The wood sulphur content of DMSO-soaked *kempas* increased up to 3.19% due to the sulphur content of DMSO. This showed that there was no modification of *kempas* wood occurring.

ATR-FTIR spectroscopy was used to observe any structural change in the *kempas* wood composite upon pre-treatment. Figure 2(a) shows the ATR-FTIR of untreated and treated *kempas* wood which displays the appearance of several peaks similar to the basic structure of all wood samples. The peaks observed were the strong broad peak at 3349 cm^{-1} representing the OH stretching of cellulose, hemicellulose and lignin, C-H stretching at 2902 cm^{-1} of methyl and methylene group (Poletto et al. 2012; Popescu et al. 2009) and C=O stretching of carbonyl in carbohydrate (Poletto et al. 2012; Popescu et al. 2009). There were also several peaks representing the lignin such as peaks at 1597, 1511 and 1235 cm^{-1} depicting the C=C, C-O stretching or bending vibration and peak at 1455 cm^{-1} representing the asymmetric bending of CH_3 while methoxy ($-\text{OCH}_3$) group presence in the lignin. Lastly is the strong absorption peak at 1029 cm^{-1} which corresponded to the C-O stretching vibration in cellulose, hemicellulose and lignin (Labbe et al. 2005).

The peak appearance of most treated *kempas* wood was similar to the untreated *kempas* wood but with a varied intensity which implied that the cellulose hemicellulose and lignin remained in the matrix but varied the intensity of the peaks due to higher solvent ratio compared to the *kempas* wood sample (10 mL/g). However, for *kempas*

wood treated with silicon oil, the appearance of additional strong peaks at 2958, 1257 and 789 cm^{-1} represented the methyl stretching, CH_3 deformation in Si-CH_3 and Si-C stretching in Si-CH_3 of silicon oil. According to Ang et al. (2012), the band at around 800 cm^{-1} is sensitive to the amount of amorphous cellulose present in the regenerated material and the broadening of this band indicates the higher amorphousity of regenerated cellulose. In the DMSO-soaked *kempas* wood and organosolv spectra (Figure 2(a)), the peak observed at 895 cm^{-1} was broad as the untreated *kempas* wood implied the similar amount of the disordered cellulosic structure. The disordered cellulosic structure was probably due to the deformation of glycosidic linkage and hydrogen bond rearrangement (Proniewicz et al. 2001). Meanwhile, there was no significant peak observed at 895 cm^{-1} for glycerol and silicon oil treated *kempas* wood. The disappearance of the peak was possibly due to that both solvents only acted physically on the surface of the *kempas* wood with no hydrogen bonding occurrence. The observation on amorphousity of the *kempas* wood was supported by XRD data analysis.

The crystallinity of lignocellulose of treated and untreated *kempas* wood was investigated using XRD. Figure 2(b) shows the diffraction patterns of untreated and treated *kempas* wood. It is known that the change in crystalline structure is commonly affected by the hydrolysis and the extent of lattice order. The slight increase in crystallinity index of glycerol, DMSO and silicon oil-soaked *kempas* to 70.7%, 67.7% and 67.0% can be due to the small quantities of hydrolysed cellulose during solvent treatment which suggests that the solvent hydrolysed portion of *kempas* cellulose is mainly amorphous cellulose rather than crystalline cellulose (Awadel et al. 1999). In addition, the properties of the solvents itself might contribute to the high degree of lateral order or reorientation of hydrogen bonding on cellulose as suggested by Milichovsky (1990).

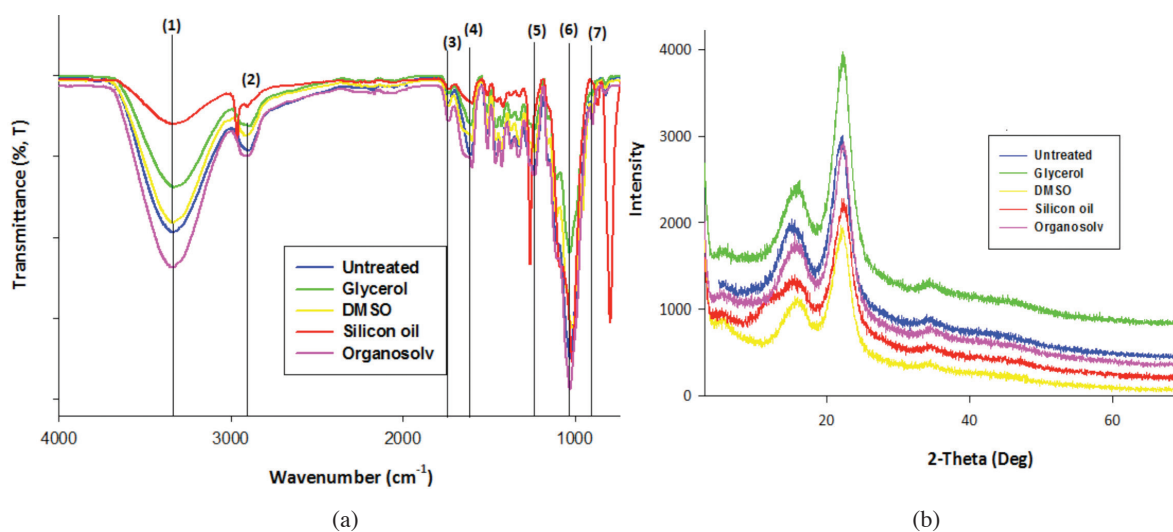


FIGURE 2. (a) ATR-FTIR spectral analysis of untreated *kempas* and treated *kempas* in glycerol, DMSO, silicon oil and organosolv. FTIR spectra bands (cm^{-1}): (1) 3349 cm^{-1} ; (2) 2902 cm^{-1} ; (3) 1731 cm^{-1} ; (4) 1597 cm^{-1} ; (5) 1235 cm^{-1} ; (6) 1029 cm^{-1} ; (7) 895 cm^{-1} (amorphous) and (b) XRD analysis of untreated and pretreated *kempas* in glycerol, DMSO, silicon oil and organosolv (ethanol/water)

TABLE 2. Crystallinity index (*CrI*), average particle size (D_{50}) of ground *kempas* wood powder, energy consumption and energy saving for grinding of untreated *kempas* and treated *kempas* with glycerol, DMSO, silicon oil and organosolv of *kempas* wood chips

Solvents	CrI Values (%)	D_{50} (μm)	Energy consumption (W h)	Energy saving (W h)
Untreated	65.5	238	0.153	0
Glycerol	70.7	263	0.138	0.015
DMSO	67.7	354	0.153	0
Silicon oil	67.0	331	0.138	0.015
Organosolv (Ethanol/water)	62.3	417	0.138	0.015

Table 2 shows the crystallinity index (*CrI*) of both untreated and treated *kempas*. Generally, the lower *CrI* indicates a higher amount of amorphous cellulose presents in the regenerated cellulose (Kuo & Lee 2009). The *CrI* of untreated *kempas* was 65.5%. Among the regenerated *kempas* wood, *kempas* wood treated with organosolv has a slightly lower crystallinity index value which shows that organosolv is capable of reducing the ordering of cellulosic fibres (Sannigrahi et al. 2010).

ENERGY CONSUMPTION

Table 2 shows the energy consumption and saving for ground *kempas* wood chip soaked with various solvents. The running empty grinder was found to consume 0.138 W h over the measured 5 s. The energy required to grind untreated wood chips was 0.153 W h. It was observed that the energy consumption for ground wood chips soaked with glycerol, silicone oil and Organosolv (0.138 W h) was reduced compared to that for ground untreated wood chips with energy savings up to 0.015 W h. Meanwhile, the energy consumption for DMSO-soaked ground wood chips was the same as for the untreated wood chips and led to no energy saving. The energy savings might be a consequence of the specific properties of the various solvents, for example, in terms of viscosity and density, glycerol was the highest (1499 c.p, 1.26 g/mL) followed by silicone oil (100 c.p, 0.967 g/mL), DMSO (1.996 c.p, 1.10 g/mL) and ethanol (1.074 c.p, 0.79 g/mL). The high viscosity led to low penetration abilities which enhanced the glycerol and silicone oil lubricating properties. Meanwhile, organosolv-soaked *kempas* wood has lower energy consumption due to its wetting effect.

PERCENTAGE OF GROUND *KEMPAS* PASSING THROUGH SIEVE

Table 3 shows the amount of various solvents absorbed by *kempas* wood chips and the respective percentage of ground wood powder passing through the sieve. It can be seen that the solvent absorbed in the highest amount by the wood chips was DMSO (0.945 mL), followed by glycerol (0.607 mL), Organosolv (0.361 mL) and silicone oil (0.042 mL). Samios and Durov (2004) reported that DMSO has high penetration ability as it is a bi-functional molecule, has both polar and non-polar groups and thus exhibits hydrophilic and hydrophobic effects simultaneously. DMSO also has a low viscosity which enhances its penetration ability.

In comparison with DMSO, glycerol showed lower absorption by *kempas* wood chips. This might be due to the lower penetration ability and the fact that the viscosity of glycerol (1499 c.p. at 20°C) was higher than that of DMSO (1.996 c. p. at 20°C). The viscosity was found to have a pronounced effect in the form of resistance to the spreading of liquids on solid surfaces and therefore, the spreading of highly viscous solvents tended to be lower than that of solvents with low viscosity (Sikalo et al. 2002).

Although the amount of DMSO absorbed by the wood chips was the highest compared to the other solvents, the percentage of ground wood powder soaked in DMSO passing through the sieve was the lowest at 10.9% compared to untreated 45.62%. This was potentially due to the swelling of *kempas* wood after being soaked by DMSO. It was followed in ascending order by silicone oil (29.70%), Organosolv (31.77%) and glycerol (34.46%). Glycerol has a higher percentage of wood passing through a sieve as it does not cause the swelling of ground wood but acts as lubricant due to its high viscosity, density and low penetration ability. As the environmental issue is the main concern of this study, the researchers intend to discover the green alternative solvents to the environment and can be used to reduce the energy consumption. The *kempas* wood chips soaked with glycerol showed the highest percentage of ground wood powder passing through the sieve compared to other solvents. Therefore, glycerol, a green solvent and a by-product of oleochemical industry, has a high potential of being used as a soaking solvent for wood chips in order to reduce energy consumption on grinding.

PARTICLES SIZE AFTER GRINDING

Mani et al. (2004) described that the energy consumption of the grinding process is largely proportional to the ratio between the initial and final particle size. Brandt et al. (2012) hypothesised that energy savings might relate to rougher grinding and larger final particle sizes due to the starting particle sizes being very similar. Therefore, the particle size distribution of the ground wood was measured for each sample and average particle size was calculated in order to determine whether the energy savings were related to less effective grinding.

The average particle size (D_{50}) shown in Table 2 was measured for ground chips after soaking for 1 h in the chosen solvents: glycerol (263 μm), silicone oil (331 μm)

TABLE 3. Amount of various solvents absorbed by *kempas* wood chips and percentage of ground wood powder passing through the sieve

Treatment solvent	Amount of solvent absorbed (mL)	Mass of ground wood passed through the sieve (g)	Percent passing (%)
Untreated	0	0.4566	45.62
Glycerol	0.607	0.5543	34.46
Silicone oil	0.042	0.3100	29.70
DMSO	0.945	0.2121	10.90
Organosolv	0.361	0.4329	31.77

DMSO (354 μm) and Organosolv (417 μm). A lower D_{50} indicates a more effective grinding process. D_{50} of untreated ground wood powder was 238 μm . As mentioned above, there was no energy saving in terms of energy consumption for untreated wood chips. Therefore, the chosen solvent, glycerol, led to energy savings of 0.015 W h and showed almost the same D_{50} as untreated ground wood powder; thus, it shows that glycerol has a potential for being used as a soaking solvent in grinding processes.

Despite the difference in crystallinity index, particle size and different amount of solvent absorbed by *kempas* wood, the energy consumption of glycerol, silicon oil and organosolv was the same. This was the consequence of the lubrication and wetting effect on the ground *kempas* wood. Table 2 illustrates that the energy savings were not due to particle size, crystallinity index, and amount of solvent absorb but lubrication and wetting effects instead.

CONCLUSION

The use of glycerol, silicone oil and organosolv resulted in energy saving of 0.015 W h. Meanwhile, for the average particle size (D_{50}) of grounded *kempas*, glycerol soaked *kempas* D_{50} was closest to that of untreated *kempas* powder compared to silicone oil, DMSO and organosolv (1:1 water: ethanol) soaked ground *kempas* which has larger D_{50} at constant grinding time. There were no significant changes observed in terms of chemical properties as referred to FTIR and XRD analysis. Glycerol-soaked *kempas* consumed less energy which led to energy saving of up to 0.015 W h and exhibited the smallest average particle size (263 μm) close to that of untreated *kempas*. Therefore, glycerol is the best choice to be used as soaking solvent in grinding process. Moreover, glycerol is easily available and comparatively inexpensive and can lower the grinding energy requirements for lignocellulosic feedstock. Thus, this emphasises the glycerol potential applications as a green soaking solvent in industries.

ACKNOWLEDGEMENTS

The authors acknowledge the support by Universiti Kebangsaan Malaysia for the financial support via the research grants GUP-2017-014 and FRGS/1/2014/ST01/UKM/02/1. The authors are grateful to the Forest Research Institute Malaysia (FRIM) for providing the samples.

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Received: 30 August 2017

Accepted: 29 December 2017