# PHYSICAL PROPERTIES OF PALM-BASED METHYL ESTER SULPHONATE (MES) SURFACTANT

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**ABSTRACT.** Palm-based methyl ester sulphonate (MES) is an anionic surfactant derived from renewable resources by sulphonation of palm methyl ester with sulphur trioxide and can be used as an alternative to the conventional linear alkylbenzenesulphonate. MES has been shown to possess favourable environmental characteristics, water hardness tolerance, with excellent detergency and foaming properties that are useful in detergent industry. Due to its great potential as surfactant in consumer products, it is crucial to determine its physical properties to protect consumer safety and the ecosystem. Physical properties are important aspects of a chemical because they play a significant role in determining its possible applications. Therefore, this study aims to determine the physical properties of various homologues of palm-based MES, such as C<sub>12</sub>-, C<sub>14</sub>-, C<sub>16</sub>-, and C<sub>16:18</sub>-MES. The selected physical properties included physical appearance (form, colour, and odour), functional groups via Fourier-transform infrared (FTIR) spectroscopy, density, melting point, and flammability. The sulphonation process to produce MES was confirmed through sulphonationcation interactions from the FTIR spectra. A better understanding of the properties and applicability of MES in different areas can be assessed through the study of their physical properties. Information on these properties is important, whereby the specifications of palm-based MES can be used to find the best formulation for its applications and support the regulatory requirements of importing countries, e.g., Registration, Evaluation, Authorisation and Restriction of Chemicals (REACh), to facilitate market penetration.

KEYWORDS. Anionic surfactant; Palm-based; methyl ester sulphonate; Detergent; properties.

## INTRODUCTION

The number of detergent products that contain methyl ester sulphonate (MES) is rapidly increasing worldwide. In the area of surfactants, MES, which is an anionic surfactant derived from palm oil, has been commercially used as the main active ingredient for detergents (Parveez *et al.*, 2020). In 2015, the global market size was valued at USD 0.57 billion and is likely to reach USD 2.2 billion by 2024 (Global Market Insight, 2020). Today, MES is found in detergent formulas, including granules or powders, liquids, and even soap bars. The commercial success of these detergent products demonstrates that MES has tremendous potential to be used as a surfactant in consumer products. MES is produced by sulphonation of methyl esters with sulphur trioxide and can be used as an alternative to conventional surfactants, such as linear alkylbenzene sulphonate (LAS). The

product proves to be an environmental-friendly and feasible alternative to the presently used surfactant, linear alkyl benzene sulphonate (Parveez *et al.*, 2020). The use of MES as an alternative surfactant to LAS is governed by prices and their availability on a sustainable basis. In addition, the ecotoxicological behaviour of a surfactant is considered as important as its technical capabilities, and MES was found to be readily biodegradable, comparable to LAS (Ghazali, 2002). MES has attracted considerable attention as a sustainable material that can be obtained from renewable resources, such as palm oil (Maurad *et al.*, 2006; Maurad *et al.*, 2017). It has been shown that MES derived from renewable resources have favourable environmental properties, good surface-active properties, water hardness tolerance, excellent detergency, and good foaming and wetting properties (Satsuki *et al.*, 1992; Ghazali *et al.*, 2004). Furthermore, MES was successfully formulated into light-duty and heavy-duty liquid detergents, in addition to powdered detergents (Maurad *et al.*, 2006; Maurad *et al.*, 2017).

Most industrialised countries now require substances to be registered or notified with the local government in accordance with existing legislation (Ghazali *et al.*, 2019). One example is the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACh), a European Union Regulation (Regulation (EC) No 1907/2006, 2006). It was established to increase the protection of consumers, health, and the environment from hazardous substances exposure (Cihák, 2009). All manufactured chemical substances in the European Union (EU) or imported into the EU in quantities of one tonne or more per year are required to be registered with the European Chemicals Agency (ECHA). The specific information required by REACh consists of three endpoints: physico-chemical, environmental, and human health (Agency, 2012). Various properties are required under the physico-chemical endpoint, such as state of the substance (appearance / physical state / colour), melting point / freezing point, boiling point, density, flash point, flammability, solubility, and viscosity. The requirements for environmental and human health endpoints can be found elsewhere (Agency, 2012).

Fourier-transform infrared (FTIR) spectroscopy is widely used in substance identification. It is able to provide accuracy, reproducibility, rapid, non-destructive, and time-saving method that can detect a range of functional groups of a substance. Besides, FTIR is sensitive to changes in molecular structure and can provide information based on chemical composition and possible to detect small absorbance changes on the order of 10<sup>-3</sup>, which helps to perform and distinguish the structure state of the whole substance (Cocchi et al., 2004). Meanwhile, the determination of physical state, colour, and odour could be used in substance identification, particularly in incidences, whereby its identification is not immediately possible (Vanderley José et al., 2016). In general, substances of higher purity produce stronger characteristics, such as more pungent odour. Some substances of high purity (an odour might be due to a single chemical or mixture of chemicals) concern affect the taste, odour or appearance of samples that would generally lead to acceptance of the sample at concentrations significantly lower than those of concern for sensory evaluation. Chemicals vary in their ability to produce odours and people vary in their ability to smell them. Tastes or odours may be detectable by consumers at lower or higher levels, depending on individual and local circumstances. Relative density is calculated from a ratio between mass of a substance volume, determined at 20 °C, and the mass of the same volume of water determined at 4 °C. This parameter is not used for classification and labelling. The information on relative density is used in the determination of viscosity, as required for the classification criteria for aspiration hazard (REACh, 2019).

Melting point of a substance can be measured by various methods. For instance, the melting point of oils and fats can be determined by using Wiley melting point *via* AOCS Cc 2-38 method, open capillary slip point, softening point *via* AOCS Cc 3-25 method, and automated techniques such as differential scanning calorimetry (DSC) (American Oil Chemists' Society *et al.*, 2004; Tolstorebrov *et al.*, 2014). The melting point of a compound is related to its physical properties,

such as hardness and thermal behaviour. The DSC thermogram gives valuable information on the melting profile of a substance and can also be used to monitor changes in thermal characteristics, such as the total melting enthalpy, crystallisation, and thermal properties (Mat Dian *et al.*, 2006).

The flammable property is intended to classify a substance into the appropriate hazard class. The flammable properties include pyrophoricity, flammability, and flammability upon contact with water. As described elsewhere, a substance is pyrophoric if it ignites spontaneously within 5 minutes of exposure to air under a standardised test conditions (REACh, 2019). For flammability property, a flammable gas has a flammable range with air at 20 °C and 101.3 kPa. A flammable liquid has a flash point that is below the upper limit set in the classification and labelling criteria, while a flammable solid is a readily combustible solid; powdered, granular, or pasty substance, which can be easily ignited by brief contact with an ignition source (REACh, 2019). Meanwhile, flammability in contact with water means a substance, which upon contact with water, becomes spontaneously flammable or emits flammable gases in dangerous quantities (REACh, 2019). The flammability of a sample could be related to its flash point, the lowest temperature at which causes the vapours of the sample to ignite. The test was performed at room temperature. It is a screening test conducted to determine if the reaction of a substance with water or damp air leads to the development of dangerous amounts of gas or gasses which may be highly flammable. Currently, there is a limited database on the physical properties of these compounds available in the market especially the flammable property. Due to the nature of substance / surfactants that may pose some adverse effects on humans or animals, it is important in future, all new surfactants that will enter the market to have a product safety datasheet, which includes the environmental protection parameters such as physical property.

Currently, available information on palm-based MES in terms of physico-chemical data is insufficient to fulfil the requirements of REACh. The main objective of this study is to determine selected physico-chemical properties of palm-based MES, such as physical appearance (form, colour, and odour), functional groups *via* FTIR, density, flammability, and melting point. Various homologues of MES samples were tested, including C<sub>12</sub>-MES, C<sub>14</sub>-MES, C<sub>16</sub>-MES, and C<sub>16:18</sub>-MES. The availability of information will ensure compliance with the regulatory requirements of importing countries and assist MES market penetration into the European and other potential markets.

## MATERIALS AND METHODS

# Materials

The MES samples of  $C_{12}$ -MES (69.70 % purity),  $C_{14}$ -MES (77.15 % purity),  $C_{16}$ -MES (85.95 % purity), and  $C_{16:18}$ -MES (88.72 % purity) were obtained from KLK Oleomas (Malaysia). The purity of MES samples was analysed according to active ingredient based on ASTM D4251-89 (2016) standard test method for active matter in anionic surfactants by potentiometric titration.

# Methods

All analyses were conducted in six replicates. The results on physico-chemical properties of MES were expressed as mean value and standard deviation. Prior to analysis, the MES samples were pretreated through overnight drying at 30 °C using drying oven to remove moisture content. It is believed that moisture content could affect MES properties.

#### **Physical appearances of MES samples**

The physical appearances of MES samples (form, colour, and odour) were examined through odour and visual observation at 25  $^{\circ}$ C.

#### Functional groups via FTIR spectra

The IR spectra were recorded by using a Perkin Elmer (Beaconsfield, Buckinghamshire, United Kingdom) 1725 series FTIR spectrometer, which was equipped with a deuterated triglycine sulphate detector and controlled by a PerkinElmer 7300 PC. The instrument was purged with dry N<sub>2</sub>, and automatic dehumidifiers were used to protect from interference by  $CO_2$  and water vapour, respectively. The prepared samples were placed between NaCl windows, and the transmission path was fixed at 100 µm with a polytetrafluoroethylene spacer. The cell was then placed in the cell holder and the sample was scanned at room temperature. Calibration spectra were obtained by co-addition of 40 scans at 4 cm<sup>-1</sup> resolution and a gain of 2.0 with strong Beer-Norton apodisation over the frequency region 4000 cm<sup>-1</sup> – 650 cm<sup>-1</sup>. The spectra were rationed against a background air spectrum.

#### **Determination of Density**

The analysis was based on the Organisation for Economic Cooperation and Development (OECD) guideline for testing of chemical methods TG 109, density of liquids and solids. The determination of sample density was conducted by using Anton Paar DMA density meter 4500 M with an accuracy of 0.00005 g/cm<sup>3</sup>. The oscillating U-tube sensor (measuring cell) was filled with 1 mL of sample. The U-tube sensor instrument was electronically excited to simultaneously oscillate at the fundamental resonant frequency and its harmonics. A mechanical oscillator, constructed in the form of a U-tube, was vibrated with the oscillator resonance. Parameters for the measuring procedure were set in "measurement settings", and the "measurement finished by equilibrium" was selected for the highest accuracy results. DMA 4500 determined the density and concentration after complete temperature equilibrium was reached.

#### **Determination of Melting Point**

The melting point was analysed based on OECD guideline for testing of chemicals method TG 102, melting point/melting range. The test substances and a reference material were subjected to the same controlled programme. The difference in energy input is necessary to maintain identical temperatures between the test substance and reference material was recorded. The melting point of MES samples was measured by using differential scanning calorimetry (DSC) and capillary tube method. It can also be used to monitor the physical interaction of substances in blends from changes in thermal characteristics, such as total melting. DSC total melting of a substance is the total energy required to bring the substance from solid state to a complete melt. DSC partial melting of an oil or fat is the energy required to bring a substance from solid state in the system was more noticeable at 30 and 20 °C than at lower temperatures to a complete melt (Md. Ali & Dimick, 1994).

In the first method, DSC was coupled with a microscope (Mettler Toledo, 6000 System), with N<sub>2</sub> carrier gas at 20 mL/min. Approximately 5 mg to 8 mg of precisely weighed ( $\pm$  0.005 mg) sample was placed in a sealed DSC pan and transferred to the DSC head. When a sample underwent a phase transition, the corresponding change of enthalpy gave a departure from the base line of heat flow record. The samples were heated at 70 °C for 15 min to destroy crystal memory, cooled at 0 °C for 90 min and at -50 °C for 5 min. An empty covered sample pan was used as reference. The analysis was measured from -30 °C to 300 °C at a rate of 2 °C/min. It was important

to start the measurement at low temperature as there may be some thermal events taking place at such low temperature.

As for the capillary tube method, new capillary tubes were broken in half, and samples were inserted into the tube. The quantity of samples should be such that the height does not exceed 1 mm. Three tubes with the same sample were inserted in the tube guide. The electrothermal digital melting point unit was switched on and the instrument component was kept in stable condition. The melting process was observed through a magnifying lens. The reading was noted at every 2 °C/min. Temperature was recorded when the first droplet of liquid was seen. The end temperature was recorded when the entire sample was liquefied, whereby the entire sample changed from opaque to transparent.

### **Determination of Flammability**

The flammability test was performed according to Dir 92/69/ European Economic Community (EEC) (Official Journal (O.J.) L383 A), upon contact with water. A small quantity of sample was placed at the centre of a filter paper on the petri dish surface. The test was conducted at room temperature. A screening test was performed by dropping a few drops of water on the sample to determine if the reaction of sample with water or damp air caused dangerous amount of gas or gasses to develop, which may be highly flammable. Calcium carbide (CaC<sub>2</sub>) was used as the control substance.

The flash point is an important indicator of flammability of a substance. The flash point of MES was also investigated by using Cleveland open cup tester method, which is equivalent to OECD guideline for testing of chemicals method TG A9, flash point. The method is applicable for viscous materials that have flash points of 79 °C to 400 °C, except for fuel oils. Sample was poured into a test cup so that the top of the meniscus of the test specimen was exactly at the filling mark. The test cup was then placed at the centre of the heater. The sample was heated until it liquefied before being poured into the test cup. The temperature of sample was increased rapidly at first and then at a slower constant rate as the flash point was approached. At specified temperature intervals, a flame was passed across the cup. The flash point is the lowest temperature at which the flame application causes vapours of the sample to ignite.

## **RESULTS AND DISCUSSION**

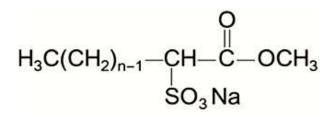
## Physical appearances of MES samples

MES is hygroscopic in nature which has the ability of a chemical compound to absorb or adsorb water. The technology to produce palm-based MES involves sulphonation of a palm oil-based methyl ester with sulfur trioxide/air to produce a dark product which followed by digestion, bleaching and neutralizing to produce a white ivory colour MES  $C_{12}$  and  $C_{14}$  and additional steps through the turbo tube drying and flaking system process to produce dry MES  $C_{16}$  and  $C_{16-18}$  (Salmiah *et al.*, 1998; Smulders *et al.*, 2007; Zulina *et al.*, 2020). During sulphonation, bleaching and neutralization, the ester group in the molecule could be hydrolysed to form a by-product call di-salt. The sources of this odour are from the source of liquid ingredients such as sulfonic acids and salts, and also from sulphates (James, 2015).

Figure 1 shows the chemical structure of MES samples analysed in this study. Physical appearances, including colour and odour of the MES samples are given in *Table 1*. All MES samples had soap-like odour, whereby  $C_{12}$ -MES and  $C_{14}$ -MES were in semi-solid state, while  $C_{16}$ -

MES and  $C_{16:18}$ -MES were flakes. The colour of  $C_{16:18}$ -MES was white to ivory, while other MES samples ( $C_{12}$ -MES,  $C_{14}$ -MES, and  $C_{16}$ -MES) were off-white.

From the analysis, physical appearances including colour and odour of the MES samples are similar as observed for all samples, which is typical for MES samples technical specifications of MES with different carbon chain lengths produced from MPOB's MES plant (Zulina *et al.*, 2020). MES produced from MPOB usually has an active content of 83% to 86% for C<sub>14</sub>-MES, C<sub>16</sub>-MES and C<sub>16:18</sub>-MES and 68% for lower chain C<sub>12</sub>-MES. It has very low colour (<25 Klett colour) and low di-salt content, (<5%) (Zulina *et al.*, 2020). Organosulfur compounds are organic molecules that contain sulphur and are associated with good perfume retention odours' characteristic soaplike odour. Scents or smells are just a blend of chemical compounds released into the air and interact with the scent receptors in our noses. Scents used to fragrance the soaps samples are the same chemicals that give fragrance and plants their distinctive smells to surfactant.



Methyl ester sulphonate (MES)

### Figure 1. Chemical structure of MES samples

<b>TABLE 1.</b> Physical Appearances of C <sub>12</sub> -MES, C <sub>14</sub> -MES, C <sub>16</sub> -MES, AND C <sub>16:18</sub> -MES at 25 °C				
(Ambient Temperature)				

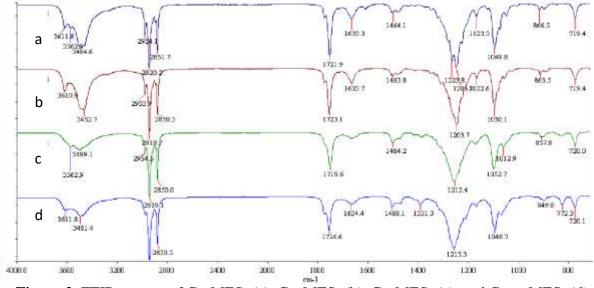
Sample	Appearance /	Form	Colour	Odour
C <sub>12</sub> -MES		Semi-solid/paste	Off-white	Soap-like odour
C <sub>14</sub> -MES		Semi-solid/paste	Off-white	Soap-like odour

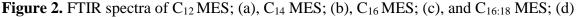
Physical Properties of Palm-Based Methyl Ester Sulphonate (Mes) Surfactant



# Functional groups via FTIR spectra

All MES samples showed similar FTIR spectra (*Figure 2*). The bands in the range of 3500 cm<sup>-1</sup> – 3600 cm<sup>-1</sup> indicated the O-H stretching of carboxylic acid. The symmetric and asymmetric -CH<sub>2</sub> stretching appeared at peaks ~2950 cm<sup>-1</sup> and ~2850 cm<sup>-1</sup>, respectively. The strong band at ~1720 cm<sup>-1</sup> was attributed to the carbonyl group, C=O (Permadani *et al.*, 2018). The sulphonation-cation interactions were confirmed by the major peaks in the range of ~1200 cm<sup>-1</sup> – ~1050 cm<sup>-1</sup> for asymmetric stretch and symmetric stretch, respectively. The characteristic peak at 1050 cm<sup>-1</sup> can be assigned to the S-O stretching vibration of the alkyl sulphonic group present in the ionomeric precursor indicates that both compounds are methyl ester sulfonate (Silverstein *et al.*, 1962, Elraies *et al.*, 2010). The FTIR results demonstrated that the MES are in good agreement from literature and prove that all are MES products (Jin *et al.*, 2016). As reported in other studies, the presence of sulphonate group was indicated at peak 1052 cm<sup>-1</sup> (Khaled Abdalla *et al.*, 2012). In addition, the peak at ~720 cm<sup>-1</sup> was typically for the S-O group (Permadani *et al.*, 2018).





### Density

The density of MES samples is given in *Table 2*. MES with higher number of carbon atoms showed higher density;  $C_{16:18}$ -MES >  $C_{16}$ -MES >  $C_{14}$ -MES >  $C_{12}$ -MES. Compound with higher density shows that the compound is more densely packed than the others. Compounds with higher density implied stronger intermolecular forces. Intermolecular forces are the attractions between molecules, which determine many of the physical properties of a substance. Under appropriate conditions, the attractions between all gas molecules will cause them to form liquids or solids. These forces pull the molecules together, which result in more molecules in one unit of volume. This is due to the formation of strong intermolecular forces; hydrogen bonding (Ariba *et al.*, 2020). From the results, the  $C_{16:18}$ -MES possess higher density compared to other compounds which indicated stronger attractive forces that the carbon for the terminal methyl (CH<sub>3</sub>) appears and the strong band at ~1724.6 cm<sup>-1</sup> was attributed to the carbonyl group, C=O. The carbon directly attached to the sulfonate group (–SO<sub>3</sub>Na–CH–) is observed at peak ~1215.3 cm<sup>-1</sup>. The signal for carbon directly attached to the ester group (–OCH3–) is observed at peak 1048.7 cm<sup>-1</sup>.

**TABLE 2.** Density of  $C_{12}$ -MES,  $C_{14}$ -MES,  $C_{16}$ -MES and  $C_{16:18}$ -MES at 25 °C (Ambient Temperature)

Sample	Density (g/cm <sup>3</sup> ) <sup>a</sup>
C <sub>12</sub> -MES	1.113 + 0.008
C <sub>14</sub> -MES C <sub>16</sub> -MES	$\frac{1.204 \pm 0.002}{1.235 \pm 0.001}$
C <sub>16:18</sub> -MES	$1.240 \pm 0.001$

Note: The <sup>a</sup> values are displayed as mean  $\pm$  standard deviation of six replicates.

## **Melting point**

The principle of the test involves determination of the phase transition temperature or temperature range from solid state to liquid state. The melting point of MES samples increased in the following order: C<sub>12</sub>-MES < C<sub>14</sub>-MES < C<sub>16</sub>-MES < C<sub>16:18</sub>-MES. As the number of carbon atom increased or the length of carbon chain increased, the melting point also increased due to an increase in the force of attraction between molecules as the molecule became longer. It took more energy to overcome the force of attraction, and cause the melting point to rise. Intramolecular forces keep a molecule intact. Intermolecular forces hold multiple molecules together and determine many of a substance's properties. Dispersion forces that develop between atoms in different molecules can attract the two molecules to each other. The forces are relatively weak, however, and become significant only when the molecules are very close. Larger and heavier atoms and molecules exhibit stronger dispersion forces than do smaller and lighter atoms and molecules. The higher the active content of MES of each chain length, the greater the melting point increase; thus, the intramolecular forces the impurities have been eliminated (Zulina et al., 2020). The melting point of MES-C12, MES-C14, MES-C<sub>16</sub> and MES-C<sub>16-18</sub> has been reported by Zulina et al. with values of 131.0, 116.3, 186.5, and 209.2 °C, respectively. The melting point of alkyl a-sulfopalmitates (C14) and stearates (C16) also has been reported by Weil et al. with values of 180.9-182.8 and 179.8-180.0 °C, respectively but their preparation in a state of purity has not been adequately described (Weil et al., 1953).

The melting point of a substance is considerably affected by impurities. Therefore, melting point determination may serve as a measure of substance purity. In this study, the purity of  $C_{12}$ -MES,  $C_{14}$ -MES,  $C_{16}$ -MES, and  $C_{16:18}$ -MES was 69.7, 77.15, 85.95, and 88.72 %, respectively. Therefore, the melting point results obtained herein were the approximate value. In this study, two different methods were used in determining the melting point of MES samples. Melting points that

were obtained by using DSC and capillary tube methods are presented in *Table 3*. It was suggested that the difference in melting points between the two methods was due to the thermal history inconsistencies of the sample during analysis.

**TABLE 3.** The Melting Point of C<sub>12</sub>-MES, C<sub>14</sub>-MES, C<sub>16</sub>-MES, AND C<sub>16:18</sub>-MES at 25 °C (Ambient Temperature)

Sample	Melting point using DSC (°C) <sup>a</sup>	Melting point using capillary tube method (°C) <sup>a</sup>
C <sub>12</sub> -MES	134.2 <u>+</u> 1.4	123.0 <u>+</u> 1.4
C <sub>14</sub> -MES	140.7 <u>+</u> 1.0	155.5 <u>+</u> 1.2
C <sub>16</sub> -MES	169.2 <u>+</u> 0.8	176.0 <u>+</u> 1.5
C <sub>16:18</sub> -MES	197.5 <u>+</u> 0.7	195.3 <u>+</u> 1.2

Note: The <sup>a</sup> values are displayed as mean  $\pm$  standard deviation of six replicates.

As an example, the DSC thermogram of  $C_{16}$ -MES is depicted in *Figure 3*. The thermogram signified the phase transfer temperatures, whereby the effect of around 60 °C corresponded to a phase transition from solid state to liquid crystal state. At around 120 °C and above, the hydrophobic part of MES started to break down into a less ordered state. The liquid crystal was melted and changed to liquid stage by heat or pressure application, which increased the MES's temperature to the melting point. The melting point for  $C_{16}$ -MES was observed at 169.2 °C. It was observed that moisture, impurities, and purity affected the melting point of a substance. Since the crystal lattice is disrupted by the presence of impurity, melting point is sensitive to substance purity.

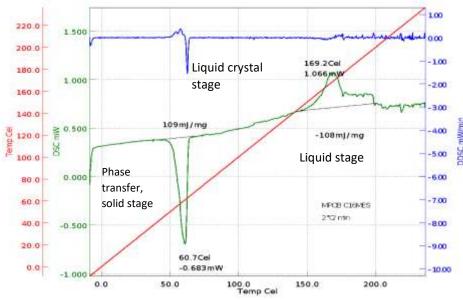


Figure 3. DSC thermogram of C<sub>16</sub>-MES

As the melting point of MES could be affected by its impurities, the reproducibility of melting point determination by using DSC was tested. *Figure 4* shows the DSC thermogram of C<sub>16</sub>-MES analysed at other laboratories (interlaboratory study). From the analysis, the melting point of C<sub>16</sub>-MES was observed at 163.2 °C. Results of the interlaboratory study were comparable (169.2 °C and 163.2 °C), which suggested good repeatability and reproducibility for MES samples measured by using DSC

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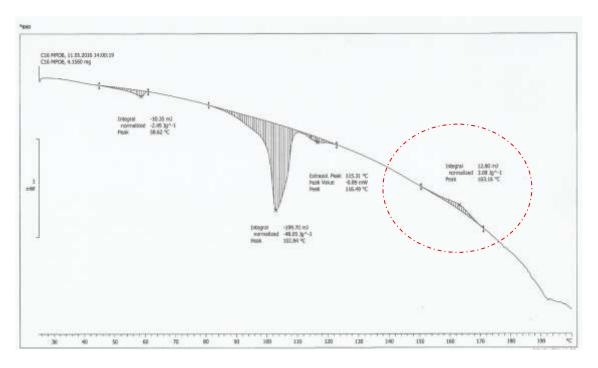


Figure 4. DSC thermogram of C<sub>16</sub>-MES from interlaboratory study

## Flammability

A few drops of water were added to MES samples, and observation was made on whether: (i) any gas was evolved and (ii) if ignition of the gas has occurred. If ignition of the gas occurs, no further testing of MES is needed and it is regarded as hazardous. *Table 4* summarises the results of the flammability test (contact with water) for control and all MES samples. From observation, all MES samples did not spontaneously ignite upon contact with water.

The flammability of a sample can be associated with its flash point. The flash point is related to the safety requirements for handling and storage substance. Thus, higher flash point makes substance safer for handling and storage and could prevent unexpected ignition during combustion. According to REACh, a flash point test does not need to be conducted if the estimated flash point is above 200 °C (REACh, 2006). Due to the high melting point of MES samples (*Table 3*), it was implied that the flash point of MES was also high, most possibly above 200 °C. From the analyses, the flash point of MES samples could not be determined due to their high melting points.

<b>TABLE 4.</b> Observation From Flammability Test (Contact with Water) For C <sub>12</sub> -MES, C <sub>14</sub> -MES,				
$C_{16}$ -MES, AND $C_{16:18}$ -MES				

Sample	Observation <sup>a</sup>
Control $(CaC_2 + H_2O)$	Spontaneously ignites upon contact with water. Hazardous.
Paste samples (C <sub>12</sub> -MES and C <sub>14</sub> -MES) + $H_2O$	No gas ignition occurred.
Flakes samples (C <sub>16</sub> -MES and C <sub>16:18</sub> -MES) + $H_2O$	No gas ignition occurred.

Note: <sup>a</sup> Observations were conducted on six replicates.

## CONCLUSION

The selected physico-chemical properties of several palm-based MES samples, C<sub>12</sub>-MES, C<sub>14</sub>-MES, C<sub>16</sub>-MES, and C<sub>16:18</sub>-MES were determined to help product formulators to find the best applications for these MES based on their physico-chemical properties and to support the regulatory requirements of importing countries. The physico-chemical properties determined included physical appearances (form, colour, and odour), functional groups *via* FTIR, density, melting point, and flammability. In general, palm-based MES samples have soap-like odour, in semi-solid and flake forms, with white to ivory colour. The sulphonation process to produce MES was confirmed through sulphonation-cation interactions from the FTIR spectra. The densities of palm-based MES samples were in the range of 1.113 g/cm<sup>3</sup> to 1.24 g/cm<sup>3</sup>. The melting point obtained from DSC analysis showed comparable results with an interlaboratory analysis, which suggested good reproducibility for melting point determination by using DSC. In addition, the flammability test signified that all palm-based MES were not flammable upon contact with water. The various homologues of palm-based MES compounds and their physical properties can be extended and explored. Information on these physical properties are important, whereby the specifications of palm-based MES can be used to find the best formulation for its applications.

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