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FULL PAPER

Compaction, mechanical strength and disintegration of palm oil empty fruit bunch (EFB) carboxymethyl cellulose (CMC) tablets

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Abstract

In this work, the compaction, tablet mechanical strength and tablet disintegration characteristics of carboxymethyl cellulose (CMC) derived from palm oil empty fruit bunches (EFB) were studied. These characteristics were then compared with an established tablet binder, microcrystalline cellulose (MCC). Tablets with 13-mm diameter were formed at various compaction pressures of 37.7MPa, 75.4MPa, 113.0MPa, 150.7MPa, and 188.4MPa using a laboratory universal testing machine inside a rigid die set. The results showed that increasing the compaction pressure increased the tablet elastic energy, plastic energy, mechanical strength and disintegration time in the experimental range adopted in this work. However, CMC tablets exhibited lower plastic work as well as lower mechanical strength and longer disintegration time compared to MCC tablets. In conclusion, the CMC tablet displayed lower performances in comparison to the more established tablet binder MCC as evaluated in this study.

1. Introduction

Palm oil empty fruit bunches (EFB) are the most abundant and readily available organic waste materials generated from palm oil mills in Malaysia. Each year, more than 15 million tonnes of EFB were generated by the palm oil industry in Malaysia (Rahman *et al.,* 2007). EFB is valuable biomass residue which can be converted into energy and also as a raw material for products of higher value such as pulp and paper, composites, compost and also bio-fertilizer (ref). EFB can be considered as the largest source of lignocellulosic biomass made up of 18-23% (w/w) lignin, 35% (w/w) celluloses and 25% (w/w) hemicellulose (Rosnah *et al.*, 2009).

Additionally, this cellulose content in the EFB can be chemically modified to produce carboxymethyl cellulose (CMC) (Rosnah *et al.*, 2004, Eliza *et al.*, 2015). Indeed, the production of CMC from EFB is beneficial since the raw material is a by-product that can be processed to a higher value product. According to Boruvkova and Wiener (2011), CMC is a derivative of cellulose which can be obtained by the chemical modification of natural cellulose. Commonly, it can be prepared through the reaction of alkali cellulose together with monochloroacetate. For CMC derived from EFB, the cellulose must first be recovered from EFB (Rosnah *et al.*, 2004). Carboxymethyl cellulose (CMC) is known as one of the water soluble cellulose derivatives with methyl group that has huge application in the food and pharmaceutical area owing to its viscosity, non-toxicity and hypoallergenic nature. It is an important ingredient in the formulations of many pharmaceutical products. Upon dissolution, it is commonly used as a film-coating agent for coating tablets (Amin *et al.*, 2007). The use of CMC derived from EFB for coating tablets has been extensively reviewed by Amin and co-workers (Amin *et al.*, 2007), where the properties of the tablets coated with CMC derived from EFB exhibited lower quality in comparison to the commercially available CMC.

Nowadays, tablets represent the most popular drug delivery systems which are manufactured by compressing a powder formulation in a die (Harbir, 2012). Tablet can be defined as a solid dosage form in which it consists of one or more active ingredients along with a series of other substances which known as excipient included in the formulation (Aulton and Taylor, 2017). Normally, tablets are in powder form, pressed or compacted to form into a solid dosage. Tablets are commonly preferred as a popular and

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versatile dosage form today due to the ease of manufacturing and convenience in administration. In addition, tablets provide the accurate amount of dosage and also chemically and physically stable by having good tolerance to changes in temperature and humidity. However, the effectiveness of such dosage forms is dependent on the dissolution of the drug in the digestive fluids prior to absorption into the systemic circulation, thus, the rate of disintegration of the tablet is critical. In addition, the formed tablets must be capable of withstanding handling throughout its lifetime without experiencing loss of mechanical integrity. Apart from the active ingredient, tablets contain excipients that act to improve the tablet properties such as binders to improve tablet strength. A previous work has shown that the addition of CMC can increase the tensile strength of cassava-starch based films (Tongdeesoontorn *et al.*, 2011). Therefore, in this current work, the suitability of CMC derived from EFB as a tablet binder is sought through extensive comparison with an existing commercial binder. The existing commercial pharmaceutical binder chosen is MCC due to its suitability and proven usage as a binder for direct compression in the pharmaceutical industry (Thoorens *et al.*, 2014).

Hence, the main drive in conducting this current work is to provide data on the CMC powder derived from EFB in terms of its compaction behaviour, mechanical strength and disintegration characteristics, which is unfortunately currently unavailable in the literature. These data will be beneficial to the researchers currently using CMC derived from commercial sources that would then allow them to have information on the CMC derived from palm oil empty fruit bunches (EFB) in terms of their tableting properties.

2. Materials and methods

2.1 Raw materials

In this research study, carboxymethyl cellulose (CMC) powder derived from palm oil empty fruit bunches (EFB) was obtained from Waris Nove Sdn Bhd. The commercial tablet binder used to benchmark the performance of the CMC powders as tablet binder was microcrystalline cellulose (MCC) type Avicel PH-102 (FMC Biopolymer, U.S.A).

2.2 Sample preparation

The mass of each tablet mass was specified to be 0.001 kg (1.0 g) (Mohammed *et al.,* 2005). Therefore, 1±0.1 g of sample powder was weighed using electronic balance (A & D Weighing ER-120A, Tokyo, Japan) to make tablets each consisting of pure CMC and MCC powders.

2.3 Tablet formation: uniaxial die compaction method

In this study, 13 mm diameters of tablets were produced by compressing accurately weighed samples of the materials using a universal testing machine (Model 3382, Instron, Canton MA, U.S.A) inside a 13-mm stainless steel die set (Specac, UK). A typical laboratory uniaxial die compaction process involves four consecutive stages which are known as filling of the die with the powder (die filling stage), the application of stress onto the powder bed, hence, forming a tablet (loading stage), the removal of stress (unloading stage) and ejection stage where the formed tablet is ejected from the die.

The tablets were compacted with 37.7MPa, 75.4MPa, 113.0MPa, 150.7MPa, and 188.4MPa compaction pressures with a cross-head speed of 0.1mms -1 (Mohammed *et al.,* 2006) during the loading stage. Same cross-head speed was also applied for unloading stage (Anuar and Briscoe, 2009). The force-displacement data was recorded during loading and unloading stages by Bluehill computer software program (Canton, MA, U.S.A). The data was then used to calculate the plastic and elastic energies of the tablets. The plastic and elastic works were determined by integrating the corresponding areas under the force-displacement curve (Mohammed *et al.,* 2005) recorded during the loading and unloading stages of the compaction process. Additionally, the maximum ejection pressure of each tablet was also recorded during the ejection stage.

2.4 Mechanical strength test

The tablet mechanical strength in this work was determined to apply a certain load along the diameter of the tablet body in order to determine the required force to break the tablet into two halves (Figure 1). This test will give the tablet indirect tensile strength (Fell and Newton, 1971). This indirect tensile strength of the tablets was determined by using a diametrical compression test whereby the tablets are fractured along their central lines. The indirect tensile strength of tablets was determined by utilizing the maximum fracture force, tablet thickness, and tablet (Fell and Newton, 1971):

$$\sigma_t = \frac{2P}{\pi DT}$$

Where σ_t (Pa) represents the tensile strength, P (N) is the breaking force of the tablet, D is the tablet diameter (m), and T (m) is the tablet thickness. The breaking force of the tablets was measured using the same universal testing machine that was for the compaction process. Each tablet was fractured diametrically between two parallel platens at a speed of 0.0116 mm/s (Mohammed

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et al., 2006).

Figure 1. Tablet placed diametrically during the mechanical strength test

2.4 Tablet disintegration test

Disintegration test for the tablets was conducted to determine the disintegration time of the tablets. In this study, the disintegration test was performed using a paddle apparatus dissolution tester (Pharma Test, Germany). 900 mL of distilled water was filled in all vessels to serve as the disintegration medium. The disintegration medium was held at a temperature of 37°C (Hamad, 2013).

One tablet sample was placed at the bottom of each vessel and the paddle was programmed to rotate at 100 rpm as shown in Figure 2. The disintegration time was measured using a stopwatch until the tablets were completely disintegrated.

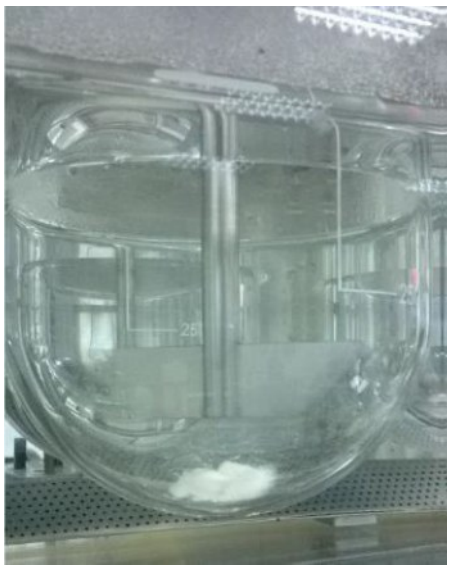

Figure 2. A disintegration test in progress showing the tablet disintegrating into the primary particles.

3. Results and discussion

3.1 Plastic work during the compaction process

The plastic work value was obtained during the loading stage of the compaction process. It represents the apparent work done during the loading stage, where the particulate materials are compacted into tablet form (Alderborn and Nyström, 1996). Based on Figure 3, tablets made from MCC exhibited relatively higher plastic work during tablet formation in comparison to the CMC tablets. Previous works utilizing MCC also showed that the tablets exhibited higher values of plastic work for MCC in comparison to sodium starch glycolate, lactose, stevia and sweet potato tablets (Shamsudin *et al.*, 2012; Zahraa *et al.*, 2013). The high values of plastic works for MCC tablets were attributed to the extensive plastic deformation of the MCC particles during the loading stage of the compaction process, reflecting the high ductility of the MCC particles. Meanwhile, CMC tablets showed relatively lower plastic work values. Therefore, the extent of the plastic deformation of the CMC particles was also relatively lower in comparison to MCC particles, thus CMC particles exhibited lower ductility within the compaction pressures adopted in this current work. In addition, the increase in the compaction pressures used during the loading stage increased the tablet plastic work for both MCC and CMC tablets. It can also be observed in Figure 3 that the difference in the plastic work values was higher between 37.7 MPa to 75.4 MPa, compared to compaction pressures above 75.4 MPa. This trend has also been observed in other works albeit different ranges of compaction pressures used in forming the MCC tablets (Shamsudin *et al.*, 2012) and urea fertilizer tablets (Shamsudin *et al.*, 2014). A logarithmic increase in the plastic work values as the compaction pressures increased indicated that there is a hypothetical maximum limit of the plastic work value when the tablet density approaches the particle true density at very high compaction pressures.

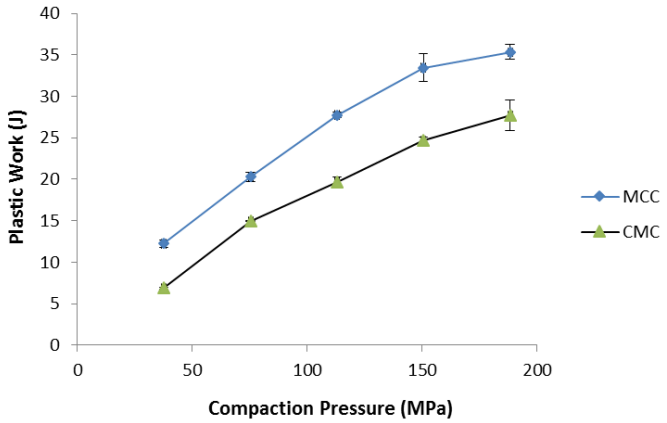

Figure 3. Plastic work values for the MCC and CMC tablets.

3.2 Elastic work during the compaction process

In contrast to the plastic work, the elastic work is the recovered work of compaction during the unloading stage of the compaction process. Physically, the elastic work is represented by the axial tablet body expansion of the newly formed tablet still confined within the die cavity after the unloading of the compaction pressure. Therefore, higher elastic expansion or recovery depicted by the elastic work means that the particles incurred relatively higher elastic deformation during the loading stage. Based upon Figure 4, MCC and CMC tablets exhibited relatively similar elastic work values and the values increased exponentially with the compaction pressure, similar to those found for other materials such as sweet potato and stevia tablets (Shamsudin *et al.,* 2012) and urea tablets (Shamsudin *et al.,* 2014).

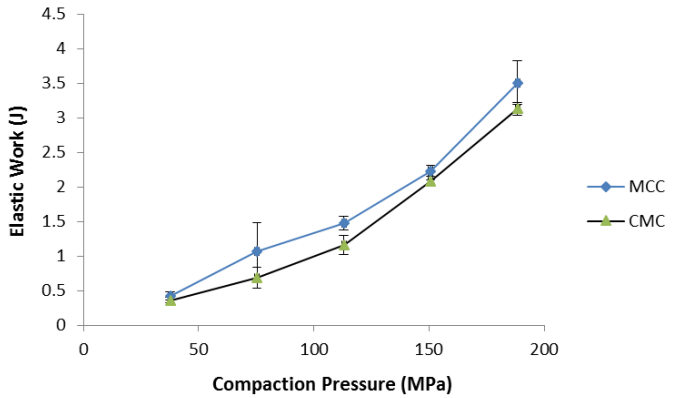

Figure 4. Elastic work values for the MCC and CMC tablets.

3.3 Maximum ejection pressure during the compaction process

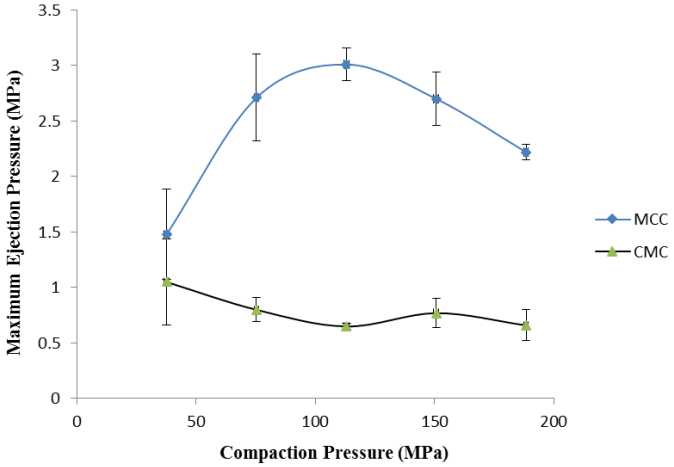

Figure 5. The maximum ejection pressure of the MCC and CMC tablets at different compaction pressures.

The maximum ejection pressure gives the quantitative indication of the difficulty of extruding the formed tablet from the die cavity. A relatively high maximum ejection pressure reflects the occurrence of relatively high static frictional effects due to the common occurrence of high maximum ejection pressures at the start of the ejection stage where the tablet starts to move inside the die cavity (Anuar and Briscoe, 2009). The frictional effects were apparently lower for the case of CMC tablets at all compaction pressures used in this work depicted by their lower maximum ejection pressures in comparison to the values obtained for MCC tablets. In addition, it can be clearly observed in Figure 5 that the CMC tablets values of the maximum ejection pressures were apparently uninfluenced by the compaction pressures used in this work. In contrast, MCC tablet maximum ejection pressure increased to a maximum value approximately at 113 MPa before gradually decreased as the compaction pressure increased.

3.5 Tablet mechanical strength

The MCC tablet mechanical strength increased with the increased in the compaction pressure as shown in Figure 6. This is in line with the trend of increasing plastic work with the compaction pressure (Figure 3), reflecting the increase in plastic deformation thus the inter-particulate bonding contributing to the increase in the tablet strength. A similar trend was also observed for CMC tablets although a slight drop in the tablet mechanical strength was observed at the highest compaction pressure of 188.4 MPa. The mechanical strength of the CMC tablets was also lower in comparison to the MCC tablets at all compaction pressures. This is expected as MCC is an established tablet excipient used as binders to increase tablet strength. It is also expected that the MCC tablets would exhibit relatively higher mechanical strength due to larger plastic work value (Figure 3) depicting higher plastic deformation hence contributing to a higher inter-particulate bonding in the MCC tablet compared to CMC tablet.

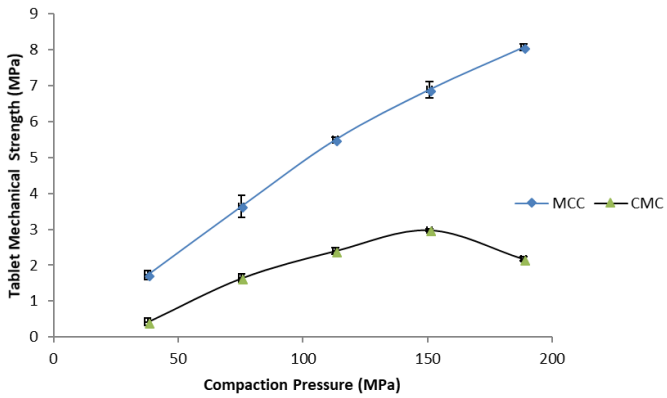

Figure 6. The tablet mechanical strength at different compaction pressures.

3.6 Tablet disintegration

In general, longer time duration for the MCC tablet to disintegrate into its smaller primary particles were observed as the compaction pressures increased. A

similar trend was also observed for CMC tablet disintegration times, where increasing disintegration times with the increased in the compaction pressure (Figure 7). However, CMC tablets exhibited rather slow disintegration times in comparison to the MCC tablets at all compaction pressures. This result indicated that the CMC tablet performed poorly in terms of its capability to disintegrate to smaller primary particles. This was in contrast with the tablet mechanical strength results, where MCC displayed higher mechanical strength hence stronger tablet bonding but easier to disintegrate in water. Although possessing relatively weaker tablet strength, the swelling behaviour of CMC, in general, interrupted the disintegration of the CMC tablet. During the course of the disintegrating experiments, the CMC tablets became gel-like and floated on the water surface.

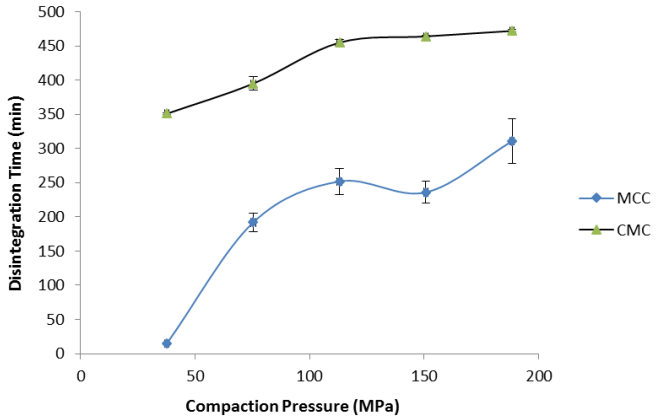

Figure 7. MCC and CMC tablets disintegration times.

4. Conclusion

The performance of CMC derived from EFB in the form of tablets have been evaluated in this study. In general, CMC tablets displayed lower plastic work and mechanical strength in comparison to MCC tablets. In addition, CMC tablets displayed gel-like behaviour during the disintegration test hence having relatively longer disintegration times in comparison to the MCC tablets. However, CMC tablets exhibited rather low maximum ejection pressures indicating relatively easier movement of the CMC tablet during the ejection stage in comparison to the MCC tablet. In conclusion, CMC derived from EFB tablets performed poorly when benchmarked with an established pharmaceutical tablet binder, MCC.

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