



Effect of high pressurized sterilization on oil palm fruit digestion operation

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Abstract

Sterilization and digestion are two important steps in the palm oil milling process prior to oil extraction. After sterilization, palm fruitlets are stripped and then fed into a digester where the fruitlets are heated with atmospheric pressure steam to soften and loosen the mesocarp fiber. The effect of different pressures of sterilization on digestion operation needs to be investigated. To convey better understanding of the process, several tests were performed on the fruitlets sterilized under different sterilization conditions, i.e., 40 and 70 psi. Oil released, sludge formation and water absorption during the digestion process were analyzed. It was found that fruitlets sterilized at 70 psi contained more sugar content in the condensate due to more hydrolyzed sterilized fruit. It was also found that fruit sterilized at higher pressure released more oil into the condensate and absorbed more water.

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Introduction

The effect of the sterilization and digestion processes on oil palm fruit properties have been investigated in several studies. These treatments affect various characteristics and properties of the fruits, such as physical, mechanical and chemical properties. These properties are of important consideration since they determine treatments in the downstream milling process of oil palm fruit. Several studies determining the properties of fresh and processed oil palm fruit have been conducted by a number of researchers, such as Akpanabiatu *et al.* (2001); Bora *et al.* (2003); Owolarafe *et al.* (2007a); Owolarafe and Faborode (2008) and Sauyee *et al.* (2011). Owolarafe *et al.* (2007b) observed the physical properties for two main varieties, Dura and Tenera.

Furthermore, both sterilization and digestion steps in palm fruit milling processes greatly affect the recovery and quantity of crude palm oil that is extracted from the mesocarp fiber (Babatunde *et al.*, 1988; Sukaribin and Khalid, 2009; and Mat Jusoh *et al.*, 2013). Though sterilization is the most crucial stage in the palm fruit milling process (Kamal, 2003), digestion is also another important stage.

Even when fruits are well-sterilized, optimum oil extraction cannot be achieved unless the sterilized fruit are well-digested. This is because through digestion process, oil cells are ruptured and hence oil flows readily during pressing operation (Owolarafe and Faborode, 2008). Thus, both processes promote cell disintegration further to facilitate oil expression. However, Owolarafe *et al.* (2002) stated that over-digestion may result in higher fiber content in crude oil which may lead to oil loss to sludge after clarification. Though the current commercial process of sterilization of FFBS (fresh fruit bunches) of oil palm is commonly carried out at 131°C and a pressure of 40 psi, some palm oil mills still carry out the operation at atmospheric pressure (steam temperature 100°C) using a continuous sterilizer. Aside from the function of facilitating loosening the fruits from the bunch, proper sterilization also inhibits free fatty acid (FFA) formation. Whether it is at 131°C or at 100°C, these conditions are sufficient enough since the lipase enzyme, which deteriorates oil quality (accelerates FFA formation), is inactivated at about 55°C (Matthaus, 2012). However, in practice, this process is still seen to possess some drawbacks. Sterilization of fruits at 100°C reduces stripping

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efficiency as considerable numbers of fruitlets are not stripped during the process. Thus, this eventually contributes to a low oil extraction rate (OER).

In another study, Babatunde *et al.* (1988) compared the effect of sterilization process conducted at two different pressures, i.e., at atmospheric pressure (1 atm.) and 2.66 atm. with varied time from 15 to 120 minutes on stripping efficiency and oil recovery. It was found that sterilization conducted at the higher pressure; at each time, reduced the number of unstripped fruits from bunches thus increasing oil recovery compared to that conducted at atmospheric pressure. This is due to better steam penetration to the inner part of the bunch. Since most of the oil-bearing cells are plant tissue, the heat and water presence enhanced the possibility of hydrolysis, by which oil-bearing cells were ruptured. Chemical changes, such as hydrolysis, take place during the sterilization process (Kuntom and Ariffin, 2012). In such a reaction, with the presence of water accompanied by high temperatures and usually high pressure, polysaccharides (cellulose, hemicellulose, etc.) will be broken down into di- or monosaccharides, such as glucose, xylose, etc., as it might occur during sterilization. Furthermore, Owolarafe and Faborode (2008) found the evidence that an increase in oil-containing cell disintegration enhanced the potential of oil release. This finding is in agreement with Mahidin (1998) who stated that one of the functions of sterilization is to disintegrate the cells where oil globules reside. Owolarafe *et al.* (2008) found that different durations of the sterilization process affected the amount of oil recovered from extraction.

Based on the findings from several previous studies, sterilization greatly affects both the digestion and oil extraction processes. During sterilization, a number of oil-bearing cells are disintegrated, while the digestion process enhances the function of the sterilization in cell disintegration. So far, the digestion has been varied in duration. Likewise, the sterilization process has been conducted at different durations as well as different pressures and temperatures, i.e., 100°C (1 atm.) and 130°C (2.66 atm.). Sterilization at 130°C greatly improves the oil recovery since more oil-bearing cells are ruptured thus more oil is released. Yet, the current process, which has been conducted at 40 psi (about 2.76 atm.), still leaves a number of unstripped bunches. Therefore, it is necessary to investigate the possibility of further increase of the pressure during sterilization. It is also important to analyze its effect on the properties of the fruit and investigate its impact on the digestion performance.

Material and Methods

Material

Fresh oil palm fruit bunches (FFBs) of the Tenera species were harvested from the UPM Oil Palm Estate (Taman Pertanian Universiti Putra Malaysia), Serdang, Selangor, Malaysia. The harvesting method was based on the criteria of the ripeness standard (PORIM, 1995). Without delay, the harvested FFBs were immediately put in a large horizontal vessel for the batch sterilization process.

Sterilization process

The fresh fruit bunches (FFBs) were subjected to two different sterilization conditions at the Pilot Scale Sterilizer, Faculty of Food Science and Technology, UPM, Serdang, i.e., at 131°C, 40 psi for 1.5 hours as commonly practiced at palm oil mills and sterilized at 70 psi (temperature of about 150°C) for thirty minutes. After sterilization was complete, the fruits were immediately detached from the bunches manually. The detached fruits were collected for the soaking treatment and digestion process.

Soaking treatment

The current oil palm fruit digestion process is conducted at atmospheric pressure with operational temperatures ranging from 90 - 96°C. This elevated temperature is reached by injecting live steam from a boiler into the digester vessel through a pipe network. However, since it is at ambient pressure, the steam immediately condenses into hot liquid water in the digester. Therefore, to simulate a digestion process phenomenon, a number of sterilized fruitlets (40 psi and 70 psi sterilized fruits), which were categorized into two different conditions, i.e., intact fruits and ruptured ones, were subjected to a hot water soaking treatment for 10, 20, 30 and 60 minutes in a 500 ml beaker glass containing 95 - 96°C hot DDI water settled on a hot plate.

After soaking, the fruitlets were then separated from the liquid. As soon as it reached room temperature, hexane was poured into the liquid to bond the droplets of oil within the liquid mixture. Then, the water was separated from the oil-hexane solution using the gravity settling method. After separation, the water was filtered using filter paper, Whatman no. 5 (pore size 2.5 µm) to obtain a solid-free aliquot. Ten microliters of the aliquot was diluted with 0.99 ml of DDI water to make up 1 mL of diluted aliquot. Then, about 1 mL aliquot sample from the procedure mentioned above was mixed with 3 mL of concentrated sulfuric acid in a test tube and shaken for 30 s. To reduce the heat, the tube was

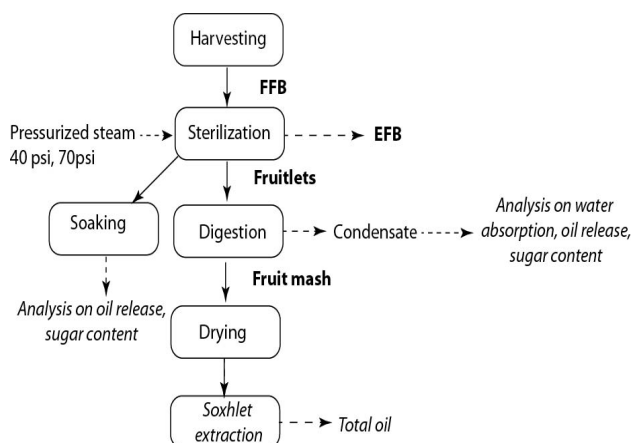


Figure 1. Method of experiment and analysis

then immersed in cold water to allow it to reach room temperature. The sugar content of the soaking liquid was determined using the sulfuric acid-UV method as proposed by Albalasmeh *et al.* (2013). The sample was analyzed using a UV spectrophotometer Perkin Elmer Lambda 35. In analyzing the sugar concentration, this method relies on the dehydration of the hydrolyzed sugar into furfural derivatives when it reacts with the concentrated sulfuric acid (Lima *et al.*, 2010).

For reference solutions, monosaccharide stocks were dissolved in DDI water to make aliquot. The stocks, glucose ($C_6H_{12}O_6$) and xylose ($C_5H_{10}O_5$), were obtained from Sigma Aldrich. The solution was made by dissolving a known volume of glucose in a 1 mL solution. The concentrations of the standard solution used in the study were 0.00 mg/L, 0.02 g/L, 0.04 g/L, 0.06 g/L, 0.08 g/L and 0.1 g/L. Then, 1 ml of the sugar solution was mixed with 3 ml of concentrated sulfuric acid to make 4 ml of aliquot. The concentrated sulfuric acid (H_2SO_4) was obtained from J.T. Baker. For the blank solution, DDI water was mixed with sulfuric acid at a ratio of 1:3 (v/v). The standard solution of xylose was made using the same steps as glucose. The sample was analyzed using a UV spectrophotometer Perkin Elmer Lambda 35.

Digestion process

After detachment from the bunch, the fruitlets were weighed and the mass was recorded. They were afterwards transferred into a lab-scale digester in batch method. The temperature of digestion process was set at 95 - 96°C. This elevated temperature was reached by injecting live steam from the boiler Casoli. The stirrer in the digester was set to rotate at 25 - 27 rpm counter clockwise. The time of the operation of the digestion process was varied, i.e., 10, 20 and 30 minutes for each 40 psi and 70 psi sterilized fruits. The steam injected from the boiler was at the rate

of 0.156 L/min. Prior to the experiment, preliminary study was carried out to investigate the performance of two types of blades, i.e., parallel and zigzag blades. The type of blade which would have better performance would be used in the main experiment. The other parameters, such as rate of blade rotation, temperature, and mass of samples, were fixed for each batch.

During the digestion process, both mass transfer and heat transfer occurred, involving water (moisture content and steam), solid (fiber, sludge) and oil within the system (digester vessel). The digestion process produced condensate consisting of water, oil and solid (sludge). This condensate was collected at the bottom of the digester through the outlet pipe and its volume was measured. The condensate was transferred into conical tubes and kept overnight for it to settle and aid separation of sludge, water and oil separation. Afterwards it was put into a water bath to elevate the temperature so that the separation could be easily observed. The digested mash was transferred into a cup for the oil pressing process.

Result and Discussion

Physically, the samples sterilized at 70 psi (temperature of steam about 150°C) for 30 minutes had a darker color due to the high temperature compared to that of the sample sterilized at 40 psi. The sterilization of oil palm fruit bunches at 40 psi is commonly practised in palm oil mills.

Soaking treatment

Effect of different sterilization processes

From study result, it was found that the sterilization conditions affected the sugar dissolved and the oil released during soaking with hot water. The sample of fruit which was sterilized at 70 psi yielded higher sugar content than samples sterilized at 40 psi. Correspondingly, the rate of oil released from 70 psi sterilized samples was also higher. Since dissolved sugar comes from hydrolyzed cell walls, the concentration of sugar correlates to how much oil bearing cells (lignocellulosic materials) are damaged. Consequently, it promotes the oil release into the soaking liquid. The presence of sugar in the liquid solution indicates that the oil bearing cell walls were being hydrolyzed and being ruptured in both the sterilization and digestion processes. Consequently, the broken cell walls allow the oil globules to flow out and accumulate in the liquid solution. This finding is in agreement with Rangnekar *et al.* (1982) and Baharudin *et al.* (2013), who came into conclusion

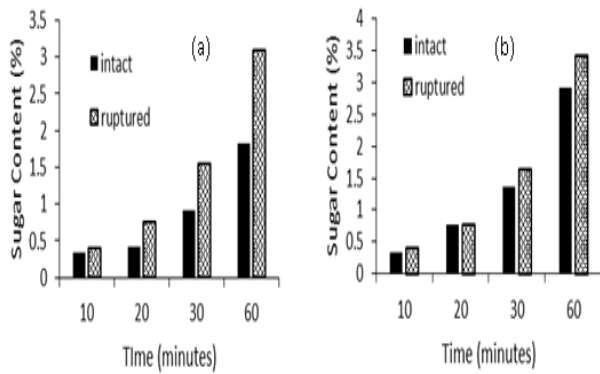


Figure 2. Effect of cell rupture to sugar content of the samples, (a) sterilized at 40 psi and (b) sterilized at 70 psi

that higher pressure obtained more hydrolysed lignocellulosic materials.

Simarani *et al.* (2009) reported that sterilized oil palm biomass had less lignin compared to fresh ones. In addition, in the sterilization process, proteins, in which oil bearing cells are dispersed, are solidified and coagulated; thus, oil will come together and flow more easily (FAO, 2014).

Effect of fiber rupture on oil release

Apart from the chemical process (hydrolysis) occurring in the sterilization process, further cell rupture will take place during the digestion process through mechanical (cutting and shearing) and physical treatments. Intuitively, oil will have an easier way to flow out of the oil-bearing cells which are ruptured.

Figure 2(a) and 2(b) suggest that the action of cutting and shearing the fibers of mesocarp increases the accumulation of sugar dissolved in the soaking liquid for both samples, (i.e., samples sterilized at 40 psi and 70 psi). Similar trends were also observed for oil released (Figure 3(a) and 3(b)). The ruptured fruits for both samples, sterilized at 40 and 70 psi, released almost twice the oil from the intact fruits especially at 40 psi. For samples sterilized at 40 psi, since the hydrolysis of the fiber was not complete, the mechanical actions (ruptured fruits) were more impactful.

Digestion process

After the digestion process, the fruit was found to be all mashed, even after 10 minutes of digestion. After the digestion process was conducted, it can be observed that the samples of 70 psi sterilized had better nut fiber separation than the 40 psi sterilized samples. The other indicator is that the sample of 70 psi sterilized has a darker color of fiber. This is due to the higher sterilization temperature at high pressure.

The blade shape of the digester is one of the key factors in fruit maceration in the digestion process.

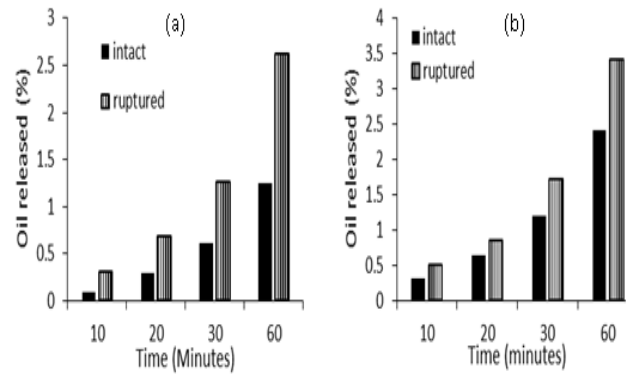


Figure 3. Effect of cell rupture to oil released from the samples, (a) Sterilized at 40 psi, (b) Sterilized at 70 psi

The blade shape should be sharp enough to cut and rupture the mesocarp fiber of the fruit. It also should mix the fruit evenly so that the effect of shear, friction and pressure are distributed well to the whole fruits.

Effect of digestion duration on water absorption, oil release, and sludge formation

The different conditions of the sterilization process affected the performance of the digestion process. Based on preliminary study, the zigzag-shaped blade had a better performance in rupturing fruitlets than the parallel shaped one. Hence, the experiment used this type of blade because of its good performance.

Figure 4(a) shows the profile of the samples sterilized at 40 psi. It could be observed that oil released, sludge and water absorbed by the mesocarp also increased when the time of digestion was extended from 10 to 30 minutes. This may be attributed to the fact that with the time of digestion increased, the mass of the fruit mash increased. However, the increase of the sludge mass was not significant.

Figure 4(b) presents the profile of the digestion process for the sample sterilized at 70 psi. Overall, the total mass of the fruit (including the mass of the fruit, oil released, and sludge) increased with the duration of the digestion process except for the water absorption. In mass balance perspective, the increase of the mass of the oil liberated from the fruit mesocarp and the mass of sludge consequently reduced the total mass of the fruit itself. For instance, after 10 minutes of digestion, the oil released from its fiber accounted for almost 17.3% of the total oil content. Consecutively, the released oil increased up to 35.5% and 41.5% for digestion times of 20 and 30 minutes, respectively. This was due to the fact that more cells were ruptured when the process was extended.

It is interesting to note that water absorption decreased in the 70 psi sterilized samples at 20 minutes of digestion; while, the oil released and

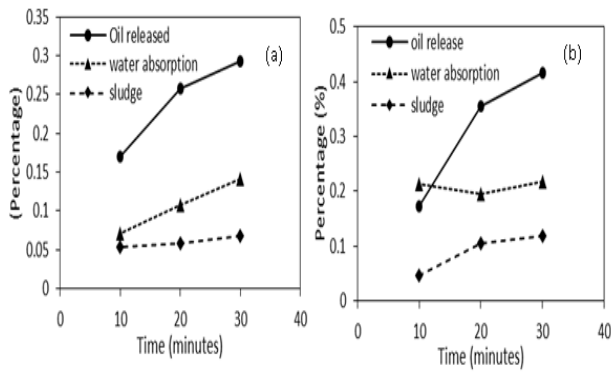


Figure 4. Oil release, water absorption, and sludge in digestion process of (a) sample sterilized at 40 psi (b) sterilized at 70 psi

mass of sludge increased with time. At 10, 20 and 30 minutes of the digestion process, the water absorption was consecutively recorded at 21.4, 19.4 and 21.7%. The water absorption decreased from 21.4 to 19.4% after 20 minutes of the digestion process. This may be due to the effect of the fiber to sludge conversion. Water absorption occurred within the porous fiber. Thus, when the fiber was converted to sludge, there was no void space for water to reside in. Referring to the analysis, there were two types of sludge formed during the digestion process according to its density, i.e., light and high density sludge. The light density lay between the water and oil; while, the heavy density was found below the water. While the oil released and sludge formation decreased the total mass, water absorption; on the other hand, increased the total mass of the fruit mash.

Effect of different sterilization condition on oil yield

Interestingly, it was found that the samples sterilized at 70 psi yielded more liberated oil from their mesocarp than that of the samples sterilized at 40 psi during digestion. This can be observed at all durations of 10, 20 and 30 minutes of the digestion process for the samples sterilized at 70 psi. This concurred with the results from the preliminary experiment on the soaking treatment earlier which reported that the samples sterilized at 70 psi had more oil liberated than that of the samples sterilized at 40 psi (Figure 5). In a previous study, the sterilization process had the effect of degradation on the oil palm biomass (Simarani *et al.*, 2009).

This oil released during the digestion process would facilitate the further oil extraction process in the oil pressing in the next process. While the duration of digestion was extended, more oil was released from both samples, those sterilized at 40 psi and the ones at 70 psi. However, prolonged duration led to more sludge formation that would hinder oil separation.

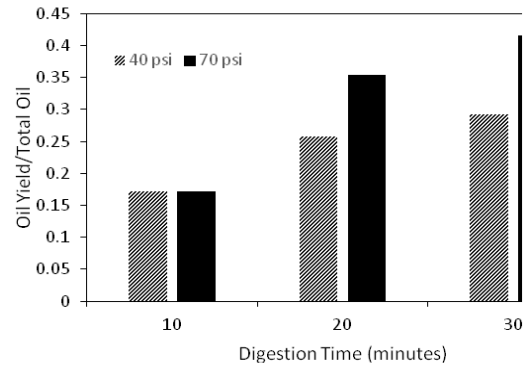


Figure 5. Comparison of oil yield from samples sterilized at 40 and 70 psi

Conclusion

After the sterilization process, the FFBs of the oil palm fruit biomass were degraded due to the pressurized hot steam treatment, which led to the hydrolysis of the lignocellulosic materials to be broken down into simple sugars. The zigzag-shaped blades had a better effect to macerate fruit mash than the parallel blades. During the digestion process, these simple sugars were diluted in the digester condensate (hot water). The study found that the samples sterilized at 70 psi has higher sugar content in the water condensate. It is interesting that the sugar concentration within the water condensate correlates to the amount of oil liberated during the digestion process. The more concentrated the sugar in the water indicated the more cell walls that were ruptured; thus, more oil was liberated in the digestion process. Of both samples, the ruptured ones had more oil released and more sugar dissolved than that of the intact samples. During digestion, samples sterilized at 70 psi released more oil than samples sterilized at 40 psi. In addition, sludge was also formed during digestion. Sludge was collected together in the condensate. Of the two samples, the most sludge was formed from the sample sterilized at 70 psi, which was one of the drawbacks of the sample sterilized at 70 psi.

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