Chapter 2

Process for Production of Coconut Oil from Fresh Coconut and Copra
Chapter 2A

Dehydration Studies of Fresh Coconut Grating for Production of Virgin Coconut Oil
2A.1. Introduction

Virgin coconut oil (VCO) is one of the high-value coconut product, sought after for its nutraceutical benefits and as a functional food oil (Marina et al., 2009a). The world demand for it is rapidly increasing and at present most of it is being produced at household, micro- or cottage-scale levels. One major concern is that when produced under unhygienic and improper conditions, the poor quality of VCO does not meet the prescribed standards (Bawalan, 2011). VCO can be produced either by the wet processing method (via coconut milk) or by the dry processing method (via dehydrated coconut gratings) as depicted in Figure 2A.1. In the dry processing method, the fresh coconut gratings are dehydrated at low temperature and the gratings are subjected to cold pressing to yield VCO.

Drying, which involves removal of water, is a complex process. As a unit operation, drying requires simultaneously heat and mass transfer (Aguilera and Stanley, 1999). A variety of dryers are used to dehydrate food products and the selection of drying method is based on the required quality of the final product. The scale of production, energy consumption and environmental issues play an important role in the choice of dryer. A tray dryer is a simple conduction dryer consisting of an insulated chamber into which tray-loads of material to be dehydrated are placed on shelves. Heat is conducted through the tray and into the solid. Air is directed over heating coils and through the spaces between trays in order to remove moisture from material. Tray drying is widely used in food industry due to its versatility and ease of operation (Hui, 2006). Vacuum shelf dryer consists of a vacuum chamber containing heated shelves (by hot water circulating through hollow platens) upon which trays are
placed. The trays containing the material to be dehydrated are placed inside the vacuum chamber. Drying takes place at lower temperature (as vacuum reduces the boiling point of water) and hence this method of drying is useful in dehydrating food products which are heat sensitive (Ramaswamy and Marcotte, 2005).

The present study involves dehydration of coconut gratings which is essential for production of VCO (via dry processing method). Different dryers, namely, tray dryer and vacuum shelf dryer are employed for this purpose. The optimum moisture content of coconut gratings was determined for expelling of coconut oil of good quality and for higher yields. Drying kinetics of dehydration of coconut gratings was also studied.

2A.2. Materials and methods

2A.2.1. Materials

Fresh mature coconuts (10-12 months) were purchased from local market. Chemicals such as potassium iodide and sodium thiosulfate of analytical grade were purchased from Merck chemicals, Mumbai, India. Phenolphthalein (1% (w/v) indicator solution in 95% ethanol) was purchased from S D Fine-Chem Ltd., Mumbai, India.

2A.2.2. Dehydration of coconut gratings

Coconuts were deshelled, pared and disintegrated using rotary wedge cutter fitted with mess size of 3 mm (Krauss Maffei, Germany). 200 g of fresh gratings were spread in trays and the trays were placed in tray dryer (Armstrong and Smith Pvt. Ltd., India) and vacuum shelf dryer (model: VTD 12, Grovers, India) and dehydrated for 1 h at 50°C and 60°C in case of tray
dryer and 44°C and 50°C for vacuum shelf dryer (with circulating hot water temperature of 50°C and 60°C, respectively).

2A.2.3. Expelling of oil of VCO

VCO was expelled from coconut gratings, after dehydration, using a hydraulic press (B Sen Barry and CO., New Delhi) at 1000 kg/cm².

2A.2.4. Moisture content

The moisture content (MC) of fresh and dehydrated gratings samples was determined according to the AOAC (2007) method. A sample of 5-6 g was oven dried at 100 - 105°C until constant weight was recorded. The difference in weight of sample before and after drying was recorded and MC [% w/w wet basis (w.b.) / dry basis (d.b.)] was determined as per the following equation:

\[
\text{Moisture content (wet basis \%)} = \left(\frac{\text{wt. of wet sample in g} - \text{wt. of dry sample in g}}{\text{wt. of wet sample in g}}\right) \times 100 \quad (2A.1)
\]

\[
\text{Moisture content (dry basis \%)} = \left(\frac{\text{wt. of wet sample in g} - \text{wt. of dry sample in g}}{\text{wt. of dry sample in g}}\right) \times 100 \quad (2A.2)
\]

2A.2.5. Measurement of VCO quality parameters

The quality parameters of VCO such as MC, free fatty acid content and peroxide value were determined by AOAC methods (AOAC, 2007) with minor modifications. MC of oil was estimated by drying 2 to 3 g of oil at 105°C in a hot air oven for 3h. The MC was expressed as % and calculated using equation 2A.1.

For estimation of Free Fatty Acid (FFA) content, 30 g oil was weighed into 250 ml flask. 50 ml of ethanol was added, which was previously neutralized by
adding 0.1N NaOH and 1 ml of 1% phenolphthalein solution. The samples were titrated against 0.1N NaOH until faint pink colour appeared. The free fat acids are expressed as % of lauric acid as indicated in the following equation:

\[
\text{Free fatty acids (as % lauric acid)} = \frac{V \times N \times 20}{W} \]  

(2A.3)

where, ‘V’ is the volume of NaOH solution, ‘N’ the normality of NaOH, ‘W’ is the weight of the oil and ‘20’ is the equivalence factor of lauric acid.

Peroxide Value (PV) was determined by first weighing 2 g oil in an Erlenmeyer flask. 25 ml of chloroform-acetic acid mixture (2:3) was added and mixed thoroughly. Saturated potassium iodide solution (1 ml) was added and left in the dark for exactly 5 min. 30 ml of distilled water and 1 ml (0.5%) starch indicator were added. The mixture was titrated against sodium thiosulphate (0.01 N) until blue colour disappears. Blank was determined by titration following the above procedure without oil. The peroxide value is expressed as miliequivalents available oxygen/kg (m eqO₂/kg) of sample and calculated from the following formula:

\[
\text{Peroxide value} = \frac{(V_t - V_o) \times N \times 1000}{W} \]  

(2A.4)

where, \(V_t\) and \(V_o\) are the volumes (ml) of sodium thiosulphate solution used to titrate the test sample and blank, respectively, N the normality of sodium thiosulphate solution and W is the weight of oil sample (g).
2A.3. Results and discussion

2A.3.1. Dehydration of coconut gratings

Coconut gratings were dehydrated at 50°C and 60°C using tray dryer and at 44°C and 50°C using vacuum shelf dryer. It can be observed (Figure 2A.2.) that ~14% (w/w w.b.) and ~3% (w/w w.b.) MC was present in coconut gratings when dehydrated using vacuum shelf dryer at 44°C and 50°C, respectively. The MC was of the coconut gratings dehydrated using tray dryer at 50°C and 60°C found to be ~5% (w/w) and ~2% (w/w, w.b.), respectively. The yield of VCO obtained after expelling the dehydrated gratings and corresponding MC of VCO is presented in Figure 2A.3. It was observed that there was not much difference in VCO yield (~ 47%) and the MC of VCO was found to be less than 0.2%. The gratings obtained after dehydration at 44°C in vacuum shelf dryer were not subjected to hydraulic pressing to obtain VCO as the MC of the gratings was higher than the recommended MC of 5% (w/w). These results indicate that tray dryer was more suitable in removal of moisture from coconut gratings when compared to vacuum shelf dryer at lower temperatures as the yield of oil was practically same and the MC of VCO obtained was less than 0.2% (w/w).

Fresh coconut gratings were dehydrated at different temperatures (50°C, 60°C and 70°C) and at different drying time (30, 60 and 90 min) using tray dryer. The dehydrated gratings were subjected to hydraulic pressing to yield the VCO. From Figure 2A.4., it can be observed that when the MC of coconut gratings is less than 2% (w/w, w.b.), the oil yield is much lower than the oil yield obtained when the MC of dehydrated gratings is more than 2% (w/w, w.b.). This can be attributed to case hardening taking place at MC less than
2% (w/w, w.b.) of coconut gratings. It was also observed that higher yield of oil was obtained when coconut gratings were dehydrated at higher temperatures for shorter period of time. The gratings obtained after dehydration at 50°C and 60°C for 30 min were not subjected to oil expelling as their MC was more than 5% (w/w, w.b.). Hence the recommended MC for expelling VCO was arrived to be the range of 2 - 5% (w/w, w.b.) in order to obtain high yield of oil.

The oil quality parameters such as moisture content, free fatty acid content and peroxide values of VCO obtained from dehydrated gratings were estimated and are given in Table 2A.1. It can be observed that the MC of VCO was less than 0.12% (w/w, w.b.), FFA was ~0.15 (% lauric acid) and PV was less than 0.9 m eqO2/kg for all the VCO samples obtained after dehydration of coconut gratings at different time and temperatures. These values are within the VCO standard limits (MC: 0.1-0.5% (w/w, w.b.), FFA: < 0.5 % lauric acid and PV: < 3 m eqO2/kg. The quality of coconut oil from copra was shown to remain unaffected when copra was dried upto 90°C (Guarte et al., 1996). These results indicate that good quality VCO can be obtained by dehydration of coconut gratings using tray dryer.

2A.3.2. Drying kinetics

Drying kinetics was studied by carrying out dehydration of coconut gratings in tray dryer at 50°C, 60°C and 70°C. The variation of MC with respect to time is shown in Figure 2A.5A. As expected, an exponential decrease in the MC with drying time was observed. The variation of drying rate with MC at different temperatures (Figure 2A.5B) shows the characteristic constant rate and falling
rate period. However, a short constant rate period was observed followed by falling rate period. In the constant rate period, the rate of diffusion of MC is equal to the rate of moisture evaporation at the surface of the gratings. The falling rate period occurs when the rate of diffusion of moisture within the coconut gratings is much lower than the rate of evaporation at the surface as the MC gets depleted in the gratings as the drying progresses. This behaviour can be easily rationalized in terms of the idea of free and bound water. The free water has the properties of pure bulk water and can move by capillarity within the solid. When the air has constant properties, the free water evaporates at a constant rate, just as pure bulk water does. When this free water is exhausted, the bound water will begin to evaporate. Because it is immobile, it must evaporate within the particle and then diffuse out of the particle and into the surrounding gas. Such bound water will move more slowly and hence, retard the drying rate (Belter et al., 1988). From Figure 2A.5B, it can be observed that most of the drying has occurred in the falling rate period.

2A.4. Conclusion

Virgin coconut oil can be expelled from dehydrated coconut gratings at low temperatures. It is recommended to dehydrate coconut gratings to MC between 2% to 5% using tray dryer in order to increase oil yield. It was observed that most of the drying of coconut gratings in the drying temperature range of 50 - 70°C drying temperature to occur in the falling rate period.
Table 2A.1: Quality analysis of virgin coconut oil obtained from dehydrated coconut gratings

<table>
<thead>
<tr>
<th>Drying temperature (°C)</th>
<th>Drying time (min)</th>
<th>^MC (% w/w, w.b.)</th>
<th>*FFA (% lauric acid)</th>
<th>#PV (m eqO2/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>60</td>
<td>0.11 ± 0.00</td>
<td>0.15 ± 0.01</td>
<td>0.66 ± 0.03</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>0.10 ± 0.01</td>
<td>0.15 ± 0.01</td>
<td>0.76 ± 0.03</td>
</tr>
<tr>
<td>60</td>
<td>60</td>
<td>0.06 ± 0.00</td>
<td>0.14 ± 0.00</td>
<td>0.83 ± 0.03</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>0.09 ± 0.01</td>
<td>0.15 ± 0.00</td>
<td>0.83 ± 0.06</td>
</tr>
<tr>
<td>70</td>
<td>30</td>
<td>0.08 ± 0.00</td>
<td>0.14 ± 0.00</td>
<td>0.66 ± 0.03</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>0.08 ± 0.00</td>
<td>0.15 ± 0.01</td>
<td>0.76 ± 0.01</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>0.09 ± 0.01</td>
<td>0.15 ± 0.01</td>
<td>0.66 ± 0.01</td>
</tr>
</tbody>
</table>

^MC- Moisture Content  
*FFA- Free Fatty Acids  
#PV- Peroxide Value  
Values are averages ± SD form three replicate analysis
Figure 2A.1: Production of virgin coconut oil
Figure 2A.2: Dehydration of coconut gratings using vacuum shelf dryer and tray dryer.

VSD- Vacuum Shelf Drying
TD- Tray Drying
VSD- Vacuum Shelf Drying
TD- Tray Drying

Figure 2A.3: Yield of oil and moisture content of oil expelled from dehydrated coconut gratings
Figure 2A.4: Effect of drying time and drying temperature on yield of virgin coconut oil
Figure 2A.5: Drying kinetics of coconut gratings
Chapter 2B

Enzyme Assisted Extraction of Coconut Oil from Copra Gratings
2B.1. Introduction

Copra is the dried coconut endosperm (kernel) and is one of the major traditional products produced from mature coconuts. Copra can be classified as edible or milling types, where, edible grade of copra is consumed as a dry fruit while milling copra is used to expel coconut oil. Conventionally, milling copra is made by sun drying, smoke drying and kiln drying of the split coconut kernels. In order to obtain good quality oil with less than 0.2% moisture content, it is essential to dry copra below 6% (w/w) (Kurian and Peter, 2007). The optimum moisture content of copra for expelling of oil using commercial expellers is 2-3% (w/w) and the yield obtained is upto 94% (Salunkhe and Kadam, 1995). The oil expelling is usually carried out at high temperatures (~100°C) to obtain high yield of oil. The left-over coconut meal after expelling oil is either fed to animals or subjected to solvent extraction process.

‘Cold Pressing’ is the term given to the process of mechanically expelling of oil from the oil seed by application of pressure only, without the addition of heat or chemicals. Cold pressed oils offer economic advantages because of their superior quality and hence are highly priced. The processing involves simple steps with no need of refining. The cold pressed oils are a fast growing commodity in the food market driven by consumer perception of less processed foods as more nutritious and better quality (Gunstone, 2011). Minimal processing retains most of the natural antioxidants in oil which slow down spoilage due to oxidation (Fife, 2004). Hydraulic pressing is one of the techniques for obtaining cold pressed coconut oil. Under high pressure, the oil from copra expels out and is collected. The major drawback of this method of expelling of oil is the low yield.
Enzyme assisted extraction of oil has shown to increase the yield of oil from many oil seeds such as soybean, palm, peanut, coconut, rapeseed and sunflower (Ricochon and Muniglia, 2010; Rosenthal et al., 1996). The use of enzymes in industrial processes can often eliminate the use of high temperatures, organic solvents and extremes of pH, while at the same time offering increased reaction specificity, product purity and reduced environmental impact (Cherry and Fidantsef, 2003).

In this section, the effect of pretreatment of copra gratings with enzymes such as cellulase and protease has been reported. Cellulase refers to a group of enzymes which hydrolyze cellulose (a polysaccharide consisting of a linear chain of several hundred to many thousands of β linked D-glucose units). Cellulases have shown their potential application in various industries such as pulp and paper, textile, laundry, biofuel production, food and feed industry, brewing, and agriculture (Kuhad et al., 2011). Proteases are involved in digesting long protein chains into shorter fragments by splitting the peptide bonds that link amino acid residues and have wide applications in food and detergent industry, medicine and biotechnology (Salleh and Basri, 2006).

2B.2. Materials and methods

2B.2.1. Materials

Copra was purchased from local market. Cellulase (from \textit{Trichoderma reesei}) and Protease (from \textit{Bacillus licheniformis}) were purchased from Sigma Aldrich, St. Louis, USA.
2B.2.2. Preprocessing of copra and expelling of oil

Cora was subjected to size reduction using rotary wedge cutter (Krauss Maffei, Germany) fitted with 3 mm sieve. The copra gratings were pretreated with enzyme solutions for 3h (at 15% moisture content w/w of copra gratings). The gratings were dehydrated to final moisture content 2 to 4% (w/w) at 60°C using cabinet tray dryer (Armstrong and Smith Pvt. Ltd., Mumbai). Copra gratings (with and without preprocessing) were subjected to hydraulic pressing (B Sen Barry and Co., New Delhi) at 1000 kg/ cm² to expel coconut oil.

2B.2.3. Moisture

Moisture content of copra gratings was estimated by the procedure already described in section 2A.2.4.

2B.2.4. Fat

Soxhlet method (AOAC, 2007) was used to estimate fat content in copra gratings with few a modifications. Sample was weighed (~5 g) and transferred into cellulose extraction thimble. The thimble was placed in a soxhlet apparatus and extraction was carried out using hexane (of analytical grade) for 16 h. The solvent was evaporated and the residual oil weight was recorded and fat content was expressed as % (w/w) using the following equation.

$$\text{Fat (\% w/w)} = \left[ \frac{\text{wt. of fat in g}}{\text{wt. of wet sample in g}} \right] \times 100$$ (2B.1)
2B.2.5. Coconut oil yield

The coconut oil yield obtained after expelling is expressed as % (w/w) and given by the following equation:

\[
\text{Oil yield (\% w/w)} = \left[ \frac{\text{wt. of oil expelled in g}}{\text{wt. of oil present in sample in g}} \right] \times 100
\]

(2B.2)

2B.3. Results and discussion

The moisture and fat contents of copra gratings were found to be 5.41 ± 0.13 and 68.78 ± 1.20 (% w/w, w.b.), respectively. From Figure 2B.1, it can be observed that when copra gratings were subjected to hydraulic pressing without dehydration, ~51% oil yield was obtained. When the moisture content of copra gratings was reduced to less than 4% (w/w), ~16.5% increase in oil yield was observed. Increased creep, and thus decreased rate of pressing, observed with increased moisture content was shown during hydraulic pressing of oil seeds (Willems et al., 2008). Copra was grated into smaller particles in order to increase the surface area of interaction with enzyme. From Figure 2B.2, it can be observed that pretreatment of copra gratings with enzymes resulted in higher oil yield compared to control. In case of protease, the yield of oil increased with enzyme concentration up to 5% (≈ 0.120 Anson units/g of copra gratings). Further increase in enzyme concentration led to a decrease in oil yield. This can be attributed to production of hydrophobic peptides that bind to oil and restrict its release from the copra matrix. Lipid bodies in oil seeds are enmeshed in a kind of cytoplasmic network composed of proteins (Wolf, 1970). Disruption of the protein network aids in expelling of oil from oilseeds. Pretreatment of copra gratings with cellulase has also
shown to increase yield of oil when used up to 5% enzyme concentration (35 units/g of copra gratings). Pretreatment of copra gratings with more than 5% cellulase has not shown significant increase in oil yield. The plant cell walls are mainly composed of cellulose, hemicellulose, lignin and pectin (Rosenthal et al., 1996). Pretreatment with cellulase breaks down the cell wall thus helps releasing of oil from cells when external pressure is applied. Pretreatment of copra gratings with protease and cellulase has shown to have a synergistic effect. Protease and cellulase treatment (both at 5% concentration) have led to increase in oil yield up to 77% (w/w) compared to control (59.5%, w/w). Pretreatment of copra with combination of protease and cellulase (both at 5% concentration) led to ~29% increase in oil yield compared to control.

2B.4. Conclusion

Copra, which is known to have the highest content of fat compared to any other oil seed, was employed to produce oil using cold expelling method. Pretreatment with enzymes such as protease and cellulase has shown to increase the yield of oil expelled using hydraulic press. The yield of oil expelled was shown to significantly increase from copra gratings when pretreated individually with 5% (w/w) protease and 5% (w/w) cellulase (74 and 71% w/w, respectively) when compared to control (59%, w/w). Enzymatic treatment of copra gratings with the combination of protease (5%, w/w) and cellulase (1%, w/w) resulted in slightly higher oil yield compared to protease (5%) alone. Combination of protease (5%, w/w) and cellulase (5%, w/w) has resulted in the highest yield of oil (77%, w/w), which was considered to be optimum combination of enzyme concentration for pretreatment of copra gratings for expelling oil.
Figure 2B.1: Effect of moisture content of copra gratings (%, w/w) on yield of oil (% w/w)
Figure 2B.2: Effect of enzyme pretreatment of copra gratings
Figure 2B.3: Effect of pretreatment of copra gratings with combination of enzymes