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OSMOTIC DEHYDRATION OF SELECTED COMMERCIAL CROPS

A thesis
submitted in fulfilment
of the requirements for the degree of
Master of Engineering
In Materials and Process Engineering
at
The University of Waikato

by
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2013
ABSTRACT

Osmotic dehydration can partially reduce the moisture content of most fruits and vegetables. This method was applied to New Zealand chestnuts and Philippine cassava. The effect of osmotic dehydration on mass transfer kinetics (water loss, solids gain and normalized moisture content) and quality of osmosed products were investigated. Shelled (i.e. shell removed) and unshelled (i.e. shell intact) chestnuts in bulk conditions (1kg samples) were immersed in sodium chloride, sucrose, glucose and calcium chloride solutions of 15, 22; 50, 60; 56.5 and 50 % (w/w), respectively for 2, 4, 6, 8, 10 up to 24 hours. Cassava slices with 15 mm thickness in bulk condition (1kg samples) were immersed in sodium chloride and sucrose solutions of 22 and 25; 60% (w/w), respectively for 30, 60, 90, 120, 150, 180 and 210 minutes. The solution to product ratio was kept above 10 to minimise the concentration change of the osmotic solutions during the dehydration experiments. Experiments were conducted at ambient conditions.

For chestnuts, using glucose (56.5% w/w) and calcium chloride (50% w/w) solutions produced unfavourable results. The normalised moisture content for both shelled and unshelled chestnuts using 22% (w/w) sodium chloride and 60% (w/w) sucrose solutions was reduced to about 75 and 80%, respectively after 8 hours at 20.5°C. Thus, the chestnut shell was not a significant barrier during osmotic dehydration with these solutions. Water loss rates were higher compared to solids’ gain values both for shelled and unshelled chestnuts. The optimum osmotic dehydration time for shelled and unshelled chestnuts was 6-8 hours. Furthermore, using sodium chloride (22% w/w), the recovery of wholenuts after mechanical shelling (i.e. shell removal) showed a statistically significant improvement from 28.5±0.04% (unosmosed) to around 45.3±0.04% (osmosed). In addition, with a different batch of chestnuts, using sucrose (60% w/w) solution, an improvement of almost 16.5±.08% (osmosed) from the control (unosmosed) on wholenuts shelling recovery achieved. Chestnuts osmosed with 22% (w/w) sodium chloride were darker compared to the control samples while chestnuts osmosed with 60% (w/w) sucrose was no colour difference to
unosmosed chestnuts. However, chestnuts osmosed with sucrose were more brittle than unosmosed chestnuts.

For chipped cassava the optimum osmotic dehydration time was found to be 210 minutes for 25% (w/w) sodium chloride and 180 minutes for 60% (w/w) sucrose. The sodium chloride (25% w/w) solution achieved a slightly higher moisture loss compared to that of sucrose (60% w/w) solution at 24.5°C. After osmotic dehydration, air drying was done for final drying (to a moisture content of 0.13, wet basis) of the chipped cassava. The optimum drying temperature was 70°C at 1.8 m/s air velocity. Drying rate was not affected significantly by osmotic solutions and osmosed cassava was not affected by a 10°C increase in the drying temperature. Osmosed products with 25% (w/w) sodium chloride or 60% (w/w) sucrose showed no gelatinization after air drying to 70°C. Cassava osmosed with 60% (w/w) sucrose was darker compared to samples osmosed with 25% (w/w) sodium chloride and to unosmosed, air dried chipped cassava.

If a chestnut processor’s aim is to get higher wholenuts recovery after mechanical shelling, a 6 hour osmotic dehydration period using 22% (w/w) NaCl is recommended. Otherwise, if the processor prefers good shelling recovery and considers the broken nuts to be used for second stage by-products processing (e.g. crumb, chestnut flour), 8-hour osmotic dehydration time before mechanical shelling is more advantageous.

Osmotic dehydration for chipped cassava using 25% (w/w) sodium chloride and 60% (w/w) sucrose is only effective up to 210 and 180 minutes, respectively. The optimum air drying temperature for osmosed cassava chips with no gelatinization effect was 70°C.
**ACKNOWLEDGMENT**

The author would like to express his sincerest gratitude to the following persons and organizations that help him in any ways for the possible achievement of this study and his degree in particular.

Dr. James K. Carson, the author’s supervisor cum mentor for the informative suggestions and corrections which have led to the refinement and fulfilment of this study. His unwavering support and unpaid technical knowledge imparted to the author were highly appreciated and treasured. An appreciation also was expressed, to Dr. James Agbebavi, visiting professor from Quebec, Canada, who guided the author during the conceptualization of this study. Dr. Romualdo C. Martinez and Engr. Reynaldo Gregorio, division chief and section chief, respectively of Agricultural Machinery Division of PHILMech, for their kindness and support in allowing the author to conduct the cassava experiments in the laboratory despite of heights on their projects involving the use of vacuum oven and infrared moisture analyzer.

Moreover, deepest gratitude was extended to Mr. Godfrey Larsen, chestnut grower from Gordonton City, New Zealand and Clarita Pineda of Pampanga, Philippines, cassava consolidator for providing the samples that were used for all the trials conducted. Mr. Indar Singh and Chris Wang engineering laboratory technicians, for assisting the author in purchasing the solutions and for lending some of the apparatus that were used in the experiments, respectively. A sincerest thanks was also extended to Cheryl Ward, engineering
librarian for the technical assistance in Endnote and Microsoft applications during the report writing stage of this study.

The technical information regarding the chestnuts’ industry in New Zealand and references for the literature review provided by Dr. David Klinac and Mr. John Marguetts was also appreciated. The assistance of Dr. Klinac for the use of mechanical shelling machine was significantly treasured. The Philippine Center for Postharvest Development and Mechanization (PHilMech) management for allowing the author to take an official study leave and the New Zealand ASEAN Scholarship Program (NZAS) for the financial grant during the entire duration of his master’s degree.

The author’s friend from the PG students’ office, namely ken, nab, kimi and rashid, for the camaraderie and pieces of jokes while having a ‘tea-break’ at the engineering tea room, for without them it was never been appreciated. The author’s immediate family: Mary Grace, his wife for all the understanding, love and sacrifice while the authors’ away from home and to his son, Gian Klyde and daughter, Meagan Kyle, for giving an inspiration for the author while doing this study. To Tatay Abog and Nanay Myrna and the authors’ siblings kuya Agner, kuya Rany and Edwin for the reminders and encouragement while the author is fighting homesickness and solitude.

Above all, to the Almighty God, the great provider of wisdom and knowledge, this piece of work is dedicated.
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1 INTRODUCTION

1.1. New Zealand Chestnuts

Chestnut (*Castanea sativa*) is the nut inside the chestnut tree fruit burr (Klinac, 2012) which is used for a variety of purposes. Chestnuts have been for centuries a remarkable source of carbohydrates for the population living in many areas of Asia, South Europe, North Africa and most countries bordering the Mediterranean Sea (Roy et al. 2008). New Zealand chestnuts are presently grown predominantly in the North Island as an export crop, and throughout New Zealand different postharvest practices are employed (NZCC 2000). According to the New Zealand Chestnut Council (2000), the New Zealand industry is competing successfully in world markets because of their focus on producing top quality products, whether it is processed or fresh chestnuts. The most commonly planted and harvested varieties in New Zealand are called ‘1015RK’, ‘1005’, ‘1002RK’ and ‘1011PR’ (Klinac 2012). These varieties are relatively large, by comparison with European or Asian chestnuts, and processing them whole (e.g. shelling and drying) is slow and tedious (NZCC 2000; Larsen 2012; Marguetts and Klinac 2012).

Moreover, chestnuts in New Zealand are characterised by high moisture content which is prone to spoilage and diseases after harvest (Roy et al. 2008). Biju Cletus (2007) recorded moisture contents of 0.47– 0.54 (wet basis) for fresh New Zealand chestnuts, which is similar to other varieties such as the Italian variety (Koyuncu et al. 2004), Spanish variety (Chenlo et al. 2007), French variety (Breisch 1995) and some Asian varieties (Dai et al. 2001; FAO 2002; Gao et al. 2003).
Thus, efficient methods of drying such as air drying (Baker 1997) or freeze drying (Caparino 2012) are necessary. However, to facilitate drying and other secondary processing for this product, removing the external hard shell efficiently is often necessary (Klinac 2012).

Shelling freshly harvested chestnuts is a bottle-neck for growers and processors in New Zealand (Klinac 2012; Larsen 2012). Hence, Barry Stevenson of Langdon Engineering Limited designed a mechanical shelling machine which was produced by HortResearch, Ruakura, specifically for use by the Kiwi Chestnut Cooperative Company Limited (Biju Cletus 2007). However, problems with whole-nuts shelling recovery and quality of the nuts have been continuously recorded (Klinac 2012). Biju Cletus recommended that chestnuts should be pre-dried to 0.40 (wet basis) moisture content to improve and increase the shelling recovery (Biju Cletus 2007; Biju Cletus and Carson 2008; Klinac 2012; Marguetts and Klinac 2012) during mechanical shelling.

Different methods of removing water from chestnuts have been studied during the last few years (Attanasio et al. 2004; Koyuncu et al. 2004; Moreira et al. 2005; Guine and Fernandes 2006; Moreira et al. 2007; Moreira et al. 2011). It was revealed in the earlier studies (Biju Cletus and Carson 2008) that chestnut varieties in New Zealand are characterised by high temperature sensitivity and shrinkage during drying. Drying them above 30°C will result in browning and unfavourable conditions. There are several large-scale drying facilities around New Zealand for commercial food crops; however, these are not suitable for chestnut growers as they are expensive, far away from most growers and not
always available during chestnut harvest time (NZCC 2000; Klinac 2012; Larsen 2012; Marguetts and Klinac 2012).

Osmotic dehydration employing sodium chloride (NaCl) (Chenlo et al. 2006a), glucose (Chenlo et al. 2006b) and sucrose (Chenlo et al. 2007) were found to be technically feasible for fresh chestnuts. Nothing in the literature was found on the application of the combined method of osmotic dehydration followed by shelling on this product, specifically New Zealand chestnut varieties. Therefore, there was an incentive to study the effectiveness of osmotic dehydration as a pre-drying technique of New Zealand chestnuts before mechanical shelling, since it may improve shellability while also reducing drying and storage costs.

1.2. Philippine Cassava

Cassava serves as a staple food throughout the tropical countries (Perera 2010; Osundahunsi et al. 2011). For some countries, cassava is an economic crop because it is sold as animal feed and cassava starch, which has a wide range of industrial applications (Eksittikul et al. 2001). In the Philippines, cassava is one of the major root crops that is traditionally used as food and starch (Pontawe et al. 2012), but in recent years, commercial production is rapidly increasing in view of its increasing use as a major ingredient for animal feed (Martinez 2010; Osundahunsi et al. 2011). One big processor of feedstocks in the Philippines provides guaranteed market and a premium price for granulated (e.g. chopped to pieces of around 15 mm in thickness) unpeeled cassava and dried to 0.13 (wet basis) moisture content to its feed mills nationwide (Martinez 2010). The study of Pontawe et al. (2012) revealed that freshly harvested cassava in the
Philippines has an initial moisture content of 0.60 – 0.70 (wet basis), which is similar to other cassava varieties in tropical countries (Ratanawaraha 2001; Toure et al. 2004; Flach 1979; Aviara et al. 2010).

The high water activity of fresh cassava (Andres 2004) demands the application of dehydration to preserve the characteristics and reduce chemical, enzymatic and microbial activities that are responsible for its deterioration. Worldwide, sun-drying remains the most common method of drying cassava (Touré and Kibangu-Nkembo 2004; Pontawe et al. 2012). Fresh cassava is typically dried to 0.13-0.14 (wet basis) final moisture content before sale (SMC 2011). Sun drying usually takes 2-3 days during good weather conditions (Ratanawaraha 2001; Pontawe et al. 2012); however, if sun-drying is not possible, granulated cassavas are temporarily stored piled in sacks. Fermentation and other biochemical reactions can occur resulting in mass losses and quality degradation (Martinez 2010). Fresh cassava has around 38% recovery (e.g. % of initial weight remaining after drying) if it is granulated and dried immediately; however, delayed processing (e.g. 3 days) will result in 5% recovery loss and a mass loss of around 13% (Martinez 2010). In addition, Ratanawaraha et al. (2001) found that the starch content of cassava decreased from 24% to 11% for the delayed processing.

Hence, the Philippine Center for Postharvest Development and Mechanization (PHilMech) developed a prototype belt conveyor dryer for granulated cassava under the Cassava Program of the Philippine Department of Agriculture. This served as an avenue for year-round production and harvesting for cassava farmers even during inclement weather conditions. However,
problems like high drying costs due to long drying time, non-uniformity of drying, and pulverisation of cassava granules due to subsequent handling and feeding to the dryer were encountered (Pontawe et al. 2012).

Osmotic dehydration of cassava before further processing offers several advantages according to Andres (2004); Betoret (2004); Perera (2010); Siritunga and Sayre (2004) and Udensi et al. (2005). This study is a preliminary exploration of determining the effect of osmotic dehydration on the drying rates and quality of this product before the air drying stage.

1.3. Osmotic Dehydration

Dehydration is a well-known process for prolonging the shelf-life of fruits and vegetables. However, traditional drying methods affect the flavour, colour and nutrients of the final product decreasing its nutritive and sensorial quality (Klening 2007). Enzymatic reactions occur during the drying period unless certain pre-treatments have been employed (Klening 2007) of which osmotic dehydration is an example (Chavan and Amarowicz 2012). Osmotic dehydration is a relatively gentle process, often keeping the limpidness of the final product. It has received considerable attention in recent years because of its low temperature and energy requirements (Panagiotou et al. 1999; Madamba and Lopez 2002; Falade and Igbeka 2007). In general, osmotic dehydration processes are carried out at a constant low temperature (e.g. 20-40°C); therefore, the mass transfer in osmotic dehydration is considered isothermal (Yao and Le Maguer 1996).

In the published literature the most commonly used osmotic agents (Jokic et al. 2007) are sucrose (Valdez-Frugosoet al. 1998; Panagiotouet al. 1999; Khoyi
and Hesari 2007) and NaCl (Panagiotou et al. 1999; Chenlo et al. 2006a; Maftoonazad 2010). The osmotic dehydration process has been used to reduce the water content of mostly commercial crops like potato (Rahman et al. 2001), pineapple (Saputra 2001), mango (Madamba and Lopez 2002), carrots (Matusik and Meresz 2002), tomato (Lewicki et al. 2002), pear (Park et al. 2002), apple (Mavroudis et al. 1998) and apricot (Khoyi and Hesari 2007) by up to fifty percent depending on the osmotic dehydration processes (Pan et al. 2003).

1.4. Objectives of the Study

Generally, the objective of this study is to determine the effectiveness of osmotic dehydration as a pre-drying technique for two crops: New Zealand chestnuts and Philippines cassava. The study includes the following specific objectives:

1. Determine the osmotic drying kinetics of peeled and unpeeled New Zealand chestnuts using NaCl, sucrose, and other osmotic media in bulk conditions;

2. Evaluate the mechanical shellability of New Zealand chestnuts after osmotic dehydration;

3. Determine the drying kinetics of granulated cassava using sucrose and NaCl osmotic solutions;

4. Establish moisture reduction rates related to drying of granulated cassava to laboratory-scale belt conveyor drying after osmotic dehydration; and

5. Evaluate the quality of osmosed, air-dried cassava.
2 REVIEW OF LITERATURE

2.1. Overview

This chapter comprises three sections. First is the description and discussion of the importance of New Zealand chestnuts and Philippines cassava as commercial crops. The factors that affect the dehydration of these two commercial crops are also reviewed. Second is the detailed investigation of the findings of various studies concerning theories of osmosis, osmotic dehydration, factors that influence the osmotic dehydration process as well as relevant effects of osmotic dehydration for fruits and vegetables. Lastly, is the investigation on the previous studies of osmotic dehydration for chestnuts and cassava.

2.2. Chestnut

Chestnut (*Castanea* spp.) belongs to the family Fagaceae (Oraguzie et al. 1997), which also includes such well-known species as oak and beech (Klinac and Lelieveld 2000). The most important species are *C. dentata*, *C. pumila* and *C. chrysophilla* in North America; *C. mollisima* and *C. crenata* in Asia; and *C. sativa* in Europe (Chenlo et al. 2010; Conedera et al. 2004; Oraguzie et al. 1997). Chestnuts should not be confused with horse chestnuts (Genus *Aesculus*) or water chestnuts (Family *Cyperacea*), which are unrelated to *Castanea*. Chestnuts are small to large deciduous trees (Tobleson, 2000) as shown in Figure 2.1 and are moderate to fast growing. The range of heights of chestnut trees from Asia and Europe was 1-30m (NZCC, 2000).
The chestnuts at immature stage are contained inside a spiny cupule, which is called a “burr” (shown in Figure 2.2) that covers the nuts. The burrs are clustered on the branch and contain 3-4 nuts depending on the variety (NZCC, 2000). The chestnuts will fall down from the tree (Figure 2.3) when it is already mature (approximately 3 months). To process (e.g. drying) the chestnuts the hard shell should be removed for the edible kernel to come out (Figure 2.4).
Chestnuts are highly nutritious and can be a dietary staple which sets them apart from other nuts. Chestnuts are rich in carbohydrates and low in fat and are susceptible to insect invasion after harvest (Roy et al. 2008). It is a fresh
nut that is stored and consumed with a relatively high percentage of water, compared to other nut varieties (McNeil and Gardner, 2000).

2.2.1. Chestnuts in New Zealand

Chestnuts were first introduced to New Zealand by some of the earliest European settlers and are now predominantly grown in New Zealand in the North Island as an export crop (NZCC, 2000). Most chestnut orchards are in the Waikato and Bay of Plenty (Biju Cletus 2007). Most New Zealand chestnuts are hybrids of Japanese and European varieties. Chestnut varieties that are commonly planted in New Zealand are known as ‘1015RK’, ‘1005’, ‘1002RK’ and ‘1011PR’ (Klinac 2012; Larsen 2012). Of these, the ‘1015RK’ variety is the dominant crop (Figure 2.5). Chestnut trees grow well across New Zealand and produce good quality nuts; however, they possess certain processing difficulties. Chestnuts in New Zealand are characterised by a high susceptibility to fungal attack and the pellicle is very hard to remove (Biju Cletus 2007).

![Figure 2.5. Production data of chestnuts in New Zealand in terms of variety (NZCC, 2000)](image-url)
Chestnut products from New Zealand in addition to the whole nut include: canned chestnut paste, puree, sandwich spread, vodka, liqueur, crumb, flour and confectionery (Cletus 2007; Klinac 2012). Of these, the liqueur is New Zealand’s most long-lived, successful commercial chestnut product. As evidence, a businessman from Taranaki is producing a chestnut liqueur annually with a standing production of 14,000 litres. The nuts are shelled then steeped in alcohol for a year before making the liqueur (Callicot 2005).

Local businesses in New Zealand use chestnut crumb to make stuffings and desserts (Callicot, 2005). In Northwest Auckland, chestnut flour is one of the main raw materials in making spaghetti. Chestnut flour is also used in different bakery products not only in New Zealand, but also overseas (Moreira et al. 2010; Saccheti and Pinnavaia 1999; Correaia et al. 2009; Lage 2003).

2.2.2. Chestnut drying

Different methods of removing water from chestnuts of different varieties have been studied in recent years (Koyuncu et al. 2004; Moreira et al. 2005; Guine and Fernandes 2006; Moreira et al. 2011) and found that the shell and pellicle affects the efficiency of drying.

In New Zealand, chestnut farmers use traditional air drying methods. Chestnuts are laid on the tray in a single-layer thickness and brought to an open area under the sun. If the intensity of the sun is high, drying is usually done for 3-5 days (Marguetts and Klinac 2012). The study of Biju Cletus and Carson (2008) confirmed that New Zealand chestnuts are characterised by high temperature sensitivity. Good quality dried chestnuts could only be achieved by drying at 30°C or an edible texture for whole nuts was observed at a drying temperature 30°C.
down to a moisture content of 0.25 (wet basis). There are several large-scale, dedicated drying facilities around New Zealand for food crops, especially for maize, grain and macadamia nuts. However, these are not suitable for chestnut growers as they are too big, expensive and far away when needed during chestnut harvest time (NZCC 2000; Klinac 2012). A portable trailer-mounted dehumidifier/drier was built by Drying Solutions Ltd. for chestnuts; however, it is too expensive for most individual growers and best suited for a central pack house location (Marguetts and Klinac 2012). Various home-made driers have been built (e.g. Figure 2.6); however, they are not generally successful and the drying settings were not suitable for chestnuts (Klinac 2012).

Figure 2.6. Home-made chestnut dryer (Klinac 2012)
2.2.3. Influence of chestnut shell and pellicle on drying

The natural covering of chestnuts (including the shell and the pellicle) protects the nuts from fungal rot but reduces the drying rates of chestnuts (Moreira et al. 2005). The pellicle has high quantities of adhesive substances which increase its resistance to water transport and allows it to remain adhered to the rough chestnut surface during the drying process (Biju Cletus 2007).

2.2.4. Problems with shelling chestnuts

New Zealand chestnuts are difficult to shell (Klinac 2012). The shell and pellicle seems to remain intact with the edible kernel and this greatly complicates all kinds of processing applications. The pellicle has a very strong astringent taste (NZCC, 2000), which seriously affects the eating quality of the nuts. Many attempts to introduce overseas, high quality chestnut cultivars that are easy to shell (especially the European and Chinese types) have been unsuccessful in New Zealand (Klinac 2012). In traditional chestnut producing countries, shelling and peeling is usually done by hand or by using steam, flame or a combination of both. However, these techniques are less effective with New Zealand’s unique chestnut cultivars (NZCC 2000; Klinac 2012). The introduction of a mechanical shelling and peeling machine has proven beneficial to the New Zealand chestnut industry but there have been no studies investigating the optimum moisture content or pre-drying technique before shelling for New Zealand chestnut varieties (Marguetts and Klinac 2012).

Biju Cletus (2007) stated that reducing the moisture content of the New Zealand chestnut cultivar to 0.40 (wet basis) or lower by means of oven drying improved the shellability by almost 90%. Gao et al. (2008) optimised the

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shellability of Chinese chestnuts by removing 4.18% moisture content using air-impingement. Vacuum shelling machine performance also improved by pre-heating the chestnuts before shelling (Yuan et al. 2010). The study of Breisch (1995) on French varieties employed the steam method to optimise the shelling operation of the Aubert-Hooven shelling machine. Hence, reduction of moisture of chestnuts from the freshly harvested state appears to be an important step in removing the shell of this commercial crop.

2.3. Cassava

Cassava (Manihot esculenta Crantz) is a perennial shrub (Figure 2.7) that belongs to the family Euphorbiaceae. It originated in South America and was subsequently distributed to the tropical and sub-tropical countries of Africa and Asia. The tuberous root of cassava is the fourth most important source of carbohydrate in the tropics after rice, maize and sugar cane (Blagbrough et al. 2010). Generally, cassava root is long and tapered with a firm flesh encased in a peelable brown rough skin. Commercial varieties can grow up to 30 cm long (FAO 2001) and the flesh can be chalk-white or yellowish when it is dried.

![Figure 2.7. Cassava root crops](image)
According to Bacusmo (2000), the main market for cassava continues to be direct consumption (44%), followed by starch products (38%), and animal feed (18%). Cassava is considered an important food especially to the countries hit hard by the global food crisis (Bacusmo 2000). In most Asian countries, cassava occupies areas unsuited to rice, the preferred food (FAO 2001). However, one problem with cassava is its long cropping duration. Cassava becomes mature 10-12 months after planting (Pontawe et al. 2012), and cash-strapped growers have difficulties in sustaining their families between planting and harvest of cassava, hence intercropping of maize and other crops is practiced (Bacusmo 2000).

2.3.1. Cassava industry in the Philippines

The cassava industry in the Philippines is relatively small compared to that of Thailand, Indonesia, and Vietnam (Bacusmo 2000). The cassava industry comprises three sectors: direct consumption, dried granules/chips for animal feed, and starch products. Direct consumption and starch processing appears to be the most significant sectors (by volume) in the industry (Bacusmo 2000). Dried cassava granule/chip processing is a relatively new industry which is gaining economic importance (Eksittikul et al. 2001; Aviara and Ajibola 2002; Pontawe et al. 2012). Cassava in the Philippines is grown in upland dry places where minimal rain occurs (Pontawe et al. 2012). Cassava is generally a low-maintenance crop; however, for the purpose of starch and dried products (e.g. granulated/chipped) more advanced cultivation practices are being employed (Bacusmo 2000). There are over a thousand varieties of cassava with different distinctive qualities (Dufour 2003), but the common varieties being planted and used for animal feed
production in the Philippines are the Lakan White and AK-50 (Pineda 2011; SMC 2011) due to its adaptability to climate conditions in the Philippines.

2.3.2. Problems with the delay in drying cassava

Freshly harvested cassava deteriorates rapidly if not immediately dried (Pontawe et al. 2012). Growers will normally delay harvesting if cassava cannot be dried within 3 days after harvest. During the rainy season in the Philippines, which usually lasts 8 months (e.g. May – December), especially in regions with high cassava production (BAS 2012), granulated cassava are temporarily stored in piled plastic sacks. According to de Luna (2011) fermentation and other biochemical reactions will occur, resulting in mass losses and quality changes. Recovery of saleable dried cassava is around 38% (e.g. % of initial weight remaining after drying) if it is dried immediately. Delay in drying can result to a mass loss of around 13% (de Luna 2011). In monetary terms, at current delivery price of Php 9.50/kg (Pontawe et al. 2012), the loss is equivalent to around Php 1.25 per kilogram.

Although cassava can be grown and harvested throughout the year, it is usually harvested during the summer months (e.g. January – April) when sun drying is most possible. If drying was possible throughout the year, growers would be incentivised to harvest cassava year round (Pontawe et al. 2012).

2.3.3. Influence of particle size and temperature on the drying rate of cassava

Generally in the Philippines, unpeeled cassava, after removing clinging soil particles, are fed into a machine called a granulator or chipper (Figure 2.8) which chops the tubers into small pieces of about 15mm in diameter before drying (Pontawe et al. 2012). In areas where granulators are still not available,
modified corn husker-shellers (Figure 2.9) are used to cut the tubers into small pieces, resulting in a wider variation of size.

Ideally, granulated cassava should have uniform size in order to have more uniform drying rates in mechanical dryer (e.g. belt and flat bed dryer) (Pontawe et al. 2012). The study of Pontawe et al. (2012) showed that cassava...
granulator (capacity of 1 tonne/hr), which is commonly used in the Philippines, has granulated size output of 6-13mm thickness (almost 50%) and >25mm (only 1%) thickness. During drying, the smaller particles tend to be over-dried and crumble during subsequent handling compared to the larger cassavas which require longer drying time. Thus, a method to pre-dry the larger cassava chips to reduce the initial moisture content (while the smaller size cassava chips could be at the equilibrium moisture content during air drying) to have a more uniform moisture content before loading to belt-conveyor dryer is necessary.

2.3.4. Cassava drying

Worldwide, sun drying remains the most common method of drying for granulated cassava (Touré and Kibangu-Nkembo 2004; Taulo 2008). As an example, in Thailand, the largest exporter of cassava for animal feed, farmers resorts to large sun drying pavements (Ratanawaraha 2001). In the Philippines, sun-drying (Figure 2.10) is the most common method of dehydrating cassava granules/chips (Pontawe et al. 2012). The sun drying procedure is very similar to that for rice paddy, involving regular raking, turning and tempering of the product. Drying usually takes 2-5 days depending on weather.

Figure 2.10. Sun-drying of granulated cassava
Several authors conducted drying experiments of cassava using different drying technologies. Alonge (2010) conducted a performance evaluation of a small-capacity (6kg) electrically operated chip dryer to investigate the effect of the quantity of drying, drying temperature (60 to 100 °C) and drying time on the rate of drying of thin (2-4mm) cassava chips. Drying rate increased as the quantity of drying material increased while it decreased with increased drying temperature and drying time.

Pontawe et al. (2012) developed a prototype dryer with 480 kg/hour capacity and conducted preliminary experiments on drying granulated cassava using a belt conveyor system (Figure 2.11) at different drying temperatures. Performance tests showed that the belt-dryer can dry granulated cassava and can be up-scaled for larger capacity (1 ton/hour).

![Figure 2.11. Prototype belt conveyor dryer system (Pontawe et al. 2012)](image)

By increasing the length of the belt with a corresponding increase in the air blower and furnace (heater) capacity, should larger drying capacity be required. However, there were a number of design and operational issues that
needed refining including non-uniformity of the moisture content of the dried product and the scope for improved drying efficiency by increasing the capacity. The non-uniformity of drying rates, meant that repeat loading of cassava granules was required.

Another locally made dryer is used by some cooperatives or cassava traders in the Philippines. The flat-bed dryer (Figure 2.12), is used to dry cassava granules/chips using a direct-fired biomass furnace as heat source and has a 6-tonne capacity. Fresh cassava chips are dried for 16 hours from 0.60 (wet basis) moisture content down to 0.13 (wet basis) moisture content. High initial moisture content and the non-uniformity (e.g. size) of granulated cassava caused problems of long drying time that hinders the utilisation of the dryer. Thus, the dryer is only used during inclement weather conditions, to prevent spoilage of cassava chips since it is costly and time-consuming on the part of the users.

Figure 2.12. A 6-tonne capacity flat-bed dryer (Pontawe et al.2012)

A typical curve on the effect of temperature on the drying time of cassava granules/chips is shown in Figure 2.13. Initial drying tests conducted using 90°C
drying temperature resulted in hardening and gelatinisation (‘case hardening’) of the outer portion of the granulated cassava (Pontawe et al. 2012). The maximum temperature that did not result in case hardening was found to be 80°C (Pontawe et al. 2012). At a bed depth of 0.1 with 0.3 m/s air velocity and drying temperature of 50°C, manually chopped cassava with initial moisture content of around 0.60(wet basis) was dried to final moisture content of around 0.14 (wet basis) in 16 hours. At drying temperature of 50°C drying time ranged from 12 to 18 hours while at 80°C drying time ranged from 5 to 7 hours. On average drying time was reduced from 16 hours to 5 hours when drying temperature was increased from 50°C to 80°C.

![Graph of moisture content vs. drying time](image.png)

Figure 2.13. Drying of manually chopped cassava at different temperatures, 0.1 m bed depth, 0.3 m/s superficial air speed (Pontawe et al. 2012)

Hence, a simple way as a pre-drying technique that could partially reduce the initial moisture content of granulated cassava with ≥15 mm (the smaller sizes (≤ 15 mm) will attain the pseudo-equilibrium moisture content during air drying
stage) to make the initial moisture content more uniform before the air drying stage and osmotic dehydration could be one way (Betoret 2004).

2.4. Osmotic dehydration

Due to the high demand for drying agricultural crops and the problems associated with traditional drying processes, alternative, low energy requirement methods have been studied, including osmotic dehydration (Pan et al. 2003; Falade and Igbeka 2007). Osmotic dehydration is a water removal process that involves soaking of fruits and vegetables in hypertonic solution (e.g. NaCl or sugar) to reduce the water content while increasing the soluble solid content of the product (Falade and Igbeka, 2007). The mechanism of moisture loss of mostly perishable fruits and vegetables during osmotic dehydration is due to high osmotic pressure and low water activity of the solution, which allows the water from the cell membrane of fruits and vegetables to permeate into the solution (Torreggiani and Bertolo 2001).

2.4.1. Osmosis

Osmosis is a complex physical phenomenon (Raghunathan and Aluru 2006) in which a concentration difference of solute (e.g. NaCl or sucrose) molecules across a semi-permeable membrane produces a difference in solvent density and in pressure across the membrane (Seader et al. 2011; Lion and Allen 2012). Early theories of osmosis were based on bombardment mechanisms. By their random kinetic motion, the solvent molecules bombard the membrane and exert a pressure on it. Osmosis occurred through pores which were filled with solvent vapour. The solvent would distil along these pores from the pure solvent side where its vapour pressure was higher to the solution side where its vapour...
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pressure was lower (Thain 1967). Ritter et al. (1980) suggested that osmosis was pressure-induced bulk-flow, then at osmotic equilibrium where no net flow occurs, the pressures on the solution and solvent would be equal. In more recent years, Garrick et al. (1993) and Rastogi et al. (1997) argued that osmosis occurs by diffusional mechanism or by a mechanism of bulk flow through solvent-filled pores. Osmosis has been studied extensively because of its impact in a variety of areas, including separation of ions from water (Raghunathan and Aluru 2006), examination of membrane water flux performance as a function of sucrose concentration (Garcia-Castello et al. 2009) and evaluation of composite membranes (Hoover et al. 2013). Currently, osmosis is pervasive in biological applications (e.g. waste water treatment) (Ji et al. 2011), life-saving (e.g. kidney dialysis) (Savrikov et al. 1989) and fruits and vegetables drying (Pan et al. 2003) applications.

2.4.2. Mass transfer during osmotic dehydration

Osmotic dehydration is a complex dynamic mass transfer process (Falade and Igbeka 2007). The application of osmotic dehydration to industry has been restricted due to the limited understanding on the mechanism that controls simultaneous counter-current mass flows during osmotic dehydration (Yao and Le Maguer 1996). Mass transfer during osmotic dehydration depends on operating variables such as solution concentration, solute type, temperature and osmotic time (Azoubel and Murr 2004; Khoyi and Hesari 2007). Chiralt et al. (2005) also identified that characteristics of the cell tissue such as cell size, porosity, tortuosity and cell membrane permeability affect the mass transfer in bulk conditions. There are three major mass transfer flows in the osmotic
dehydration process (Falade and Igbeka 2007; Dhingra et al. 2008; Chavan and Amarowicz 2012) (Figure 2.14):

1. Water outflow from the product;
2. Solute transfer from the solution to the product, it makes this possible to introduce the desired amount of a preservative agent, any solute or nutritional interest, or a sensory quality improvement of the product; and
3. Natural solutes such as sugars, organic acids, mineral salts, pigments etc. leach from the food into the solution, which is quantitatively minor when compared with first two types of transfer, but essential with regards to the composition of the final product (Valdez-Frugoso et al. 1998; Garcia-Martínez et al. 2002; Matusek and Meresz 2002; Falade and Igbeka 2007; Osorio et al. 2007).

Figure 2.14. The schematic diagram of mass transfer during osmosis process
2.4.3. Parameters influencing the osmotic process

The rate and extent of drying of biological materials and changes in its chemical composition depend on many variables including:

- Maturity and variety of the fruit or vegetable
- pre-treatments
- temperature
- nature and concentration of osmotic agent
- agitation
- geometry and structure of the product
- product to osmotic solution ratio
- physio-chemical properties
- presence of additives

A review of these factors is summarised in Table 2.1.

Table 2.1. Osmotic process parameters influencing osmotic dehydration kinetics

<table>
<thead>
<tr>
<th>Osmosis parameters</th>
<th>Remarks</th>
<th>Reference</th>
</tr>
</thead>
</table>
| Kind and quality of raw materials   | Variety and maturity mainly control the $S_C$ and $W_L$ of osmotic process.  
The kind of raw material has effect on dewatering process during osmotic dehydration because dewatering effect was always greater than the penetration of sugar into the plant tissue. | (Chavan and Amarowicz 2012)          |
|                                    |                                                                         | (Kowalska and Lenart 2001)          |
| Size and shape of the fruit pieces  | $W_L$ increases with the increase in the ratio of surface area to volume of product pieces. If the material is larger in size, it will dehydrate more slowly because the length of the diffusion path is greater. Smaller pieces on the other hand dehydrate more rapidly. | (Madamba and Lopez 2002)            |
|                                    |                                                                         | (Rastogi et al. 2002)               |
Table 2.1. continued. Osmotic process parameters influencing osmotic dehydration kinetics.

<table>
<thead>
<tr>
<th>Pre-treatments</th>
<th>Applying pre-treatments like blanching and other solute (e.g. CaCl₂) before osmotic process promote mass transfer acceleration and prevent fruit discoloration during drying. (Moreno et al. 2000; Lewicki et al. 2002)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersion time</td>
<td>Keeping the concentration of the solution constant, the increase of the immersion time resulted in the increase of $W_L$, but the rate of mass transfer generally decreases with time. Several studies showed that the highest mass exchange rate occurs during the first two hours of osmotic treatment. (Singh, et al. 2008; Panadés, et al. 2006; Kowalska and Lenart 2001)</td>
</tr>
<tr>
<td>Temperature of the osmotic solution</td>
<td>The temperature of the osmotic solution significantly affected the rate of osmotic dehydration. Although the rate increased with temperature, several authors claimed that it should not exceed 60°C as it will destroy the cell membranes. Also, working at ambient temperature was recommended as it resulted in an economic process. (Saputra 2001; Genina-Soto et al. 2001; Chenlo et al. 2007; Khoyi and Hesari 2007)</td>
</tr>
<tr>
<td>Concentration of osmotic solution</td>
<td>Generally, the higher the concentration of osmotic solution, the faster the rate of osmosis. (Rastogi et al. 1997; Rahman et al. 2001; Saputra 2001)</td>
</tr>
<tr>
<td>Osmotic agents</td>
<td>Table 2.2 presents the review of different osmotic agents and their effects in osmotic dehydration process. Adapted from the results of the study of (Chavan and Amarowicz 2012)</td>
</tr>
<tr>
<td>Agitation</td>
<td>The rate of dehydration increases as the level of agitation is increased. The speed of agitation has a positive effect on $W_L$ during osmotic treatment. An adequate level of agitation ensures minimisation or elimination of liquid-side mass transfer effects. (Rastogi et al. 2002)</td>
</tr>
</tbody>
</table>
Table 2.1. continued. Osmotic process parameters influencing osmotic dehydration kinetics.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Product to solution ratio</td>
<td>An increase in solution to product ratio also increases the rate of osmosis and favours higher solids’ uptake. However, careful selection of the ratio is critical, since a larger product to solution ratio has more difficulties in handling the syrup fruit mixture processing. One study recommended a ratio of 1:5 (product:solution) which has no significant difference on $S_G$ between 1:10 and 1:20, hence more advantageous on waste management and cost of osmotic process. Several authors recommended greater than 1:10 (product:solution) in order to ensure that the concentration change on the solution can always be neglected and uniform driving force can be assumed. (Dalla Rosa and Giroux 2001; García-Martínez et al. 2002) (Saputra 2001; Rastogi et al. 2002; Chenlo et al. 2007)</td>
</tr>
</tbody>
</table>

Recent technologies for the enhancements of osmotic process have also been introduced including: ultrasound, partial vacuum (Simal et al. 1998), liquid nitrogen pre-treatments (Ketata et al. 2013), and pulsed electric field and centrifugal force (Amami et al. 2007).

The selection of different parameters during osmotic dehydration process depends on the application; for instance, candying needs high solid gain, which is favoured by low molecular weight of the osmotic solute at low concentration of the solution. Therefore, it is very important to determine the balance between these process parameters so that the relative rates of the two main mass transfers suit the application at hand. Table 2.2 reviews a selection of solutes that have been used for the osmotic dehydration of foods.
Table 2.2. Different osmotic agents and their effects in osmotic dehydration process (Chavan and Amarowicz 2012)

<table>
<thead>
<tr>
<th>Osmotic agent</th>
<th>Remarks</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcium chloride</td>
<td>Increased the firmness of apple slices and preserved the texture during storage.</td>
<td>(Pointing 1973)</td>
</tr>
<tr>
<td>Ethanol</td>
<td>Decreases viscosity and freezing point of the osmotic solution in cooling and freezing process.</td>
<td>(Biswal et al. 1989)</td>
</tr>
<tr>
<td>Glucose</td>
<td>Glucose, has a more profound effect on water activity depression than disaccharides and polysaccharides (sucrose and malto-dextrins). Solids gain and water loss is higher (compared to sucrose) due to smaller size of the molecules contributing to the higher mass transfer from solution to food material.</td>
<td>(Saputra 2001; Chenlo et al. 2006b; Falade and Igbeka 2007)</td>
</tr>
<tr>
<td>Invert sugar</td>
<td>Theoretically more effective than the same concentration of sucrose because when completely inverted, it has twice as many molecules per unit volume. In practice, little difference in the rate of osmotic dehydration of fruit by sucrose or invert syrups of the same concentration and temperature.</td>
<td>(Pointing et al.1966)</td>
</tr>
<tr>
<td>Lactose</td>
<td>Much lower level of sweetness than sucrose. Low solubility in aqueous solution.</td>
<td>Hawkes and Flink 1978</td>
</tr>
<tr>
<td>Malto Dextrin</td>
<td>Can be used as an osmosis solute at higher total solids concentration, or in mixed systems. Less effective than sucrose at the same concentration.</td>
<td>(Hawkes and Flink 1978)</td>
</tr>
<tr>
<td>NaCl</td>
<td>Mostly used for vegetables as it retards oxidative and non-enzymatic browning. Hinders shrinkage at the surface of the product. Sometimes blanching effect on coloured products can be prevented using mixture of salt and sugar.</td>
<td>(Hawkes and Flink 1978; Lenart and Flink 1984)</td>
</tr>
<tr>
<td>Sucrose</td>
<td>Sugar solution reduces browning by preventing the entry of oxygen. Sweetness hinders its use in vegetable processing. Increased brittleness of the product</td>
<td>(Pointing, et al. 1966; Farkas and Lazer 1969; Flink 1975)</td>
</tr>
<tr>
<td>Starch/Corn syrup</td>
<td>Similar final water content to sucrose with minimal solids gain however, slower dehydration rates than sucrose.</td>
<td>(Flink 1975)</td>
</tr>
</tbody>
</table>
2.4.4. Extent of moisture loss during osmotic dehydration

Osmotic dehydration facilitates partial water removal to lower water activity of mostly fruits and vegetables (Falade and Igbeka, 2007). Some examples of fruits and vegetables with corresponding $W_L$ values after osmotic dehydration process were presented in Table 2.3.

Table 2.3. Extent of moisture loss during osmotic dehydration of selected crops

<table>
<thead>
<tr>
<th>Kind of crop</th>
<th>Moisture loss</th>
<th>Osmotic parameters</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Papaya cubes</td>
<td>28%</td>
<td>After 6 hours of osmotic dehydration, sucrose solution at 37°C</td>
<td>Jain et al. 2011</td>
</tr>
<tr>
<td>Apple</td>
<td>48%</td>
<td>After 180 minutes, sucrose (61.5% w/w) at 30°C</td>
<td>Kowalska, 2001</td>
</tr>
<tr>
<td>Potato (v. white spunta)</td>
<td>50%</td>
<td>After 10 hours of osmotic dehydration, 60% (w/w) sucrose at 30°C</td>
<td>Rahman, 2001</td>
</tr>
<tr>
<td>Pineapple (v. queen)</td>
<td>50%</td>
<td>After 9 hours using 50% (w/w) sucrose at 30°C</td>
<td>Saputra, 2001</td>
</tr>
<tr>
<td>Tomatoe (v. revernum)</td>
<td>50%</td>
<td>After 180 minutes using 61.5% (w/w) sucrose at 30°C</td>
<td>Lewicki et al. 2001</td>
</tr>
<tr>
<td>Chestnut</td>
<td>20%</td>
<td>After 8 hours using 22% (w/w) NaCl at 35°C</td>
<td>Chenlo et al. 2006a</td>
</tr>
</tbody>
</table>

2.4.5. Osmotic dehydration in combination with air drying

Osmotic dehydration before air drying can improved or maintained the quality (e.g. colour) however, no significant increased on the drying rates of osmoosed, air-dried products. As an example, Andres et al. (2004) conducted a osmotic dehydration of cassava slices (6mm thickness) using NaCl (20% w/w) solution for 60 minutes (osmotic time) at 30°C before air drying with 40°C drying temperature, air velocity of 1 m/s and relative humidity of 62%. Compared to
unosmosed-dried products the osmosed cassava slices reached a pseudo-equilibrium moisture content almost similar time (after 500 minutes) of air drying. In terms of the quality of the osmo-dried product (compared to unosmosed), no significant volume changes and cellular collapsed was observed in the microscope. Another example is the study of Moreira et al. (2011). In spite of the lower moisture content compared to unosmosed samples, there was no significant increased on the drying rates of osmosed Spanish variety chestnuts (shell and pellicle removed) with NaCl (22% w/w) at 25°C (solution temperature) after air drying (constant velocity (2.7 m/s) and relative humidity (30%) at varying air temperatures (45, 55 and 65°C). This could due to the higher resistance to moisture loss by the salt gained during osmotic dehydration. Also, the difference on the air drying temperature (55 and 65°C) has no significant effect on the drying rates of the osmosed products. The quality of the osmosed product after air drying revealed no enzymatic browning.

Osmo-dehydrated tomato slices (v. Revermun and v. Lima) retained their structural properties and only showed slight shrinkage and retained the original shape as compared to unosmosed samples (control) during the subsequent air drying stage (Lewicki et al. 2002). The aroma, which is another important quality factor when dehydration is applied to fruits and vegetable, was better than that of unosmosed product after further drying for some foods (Pani et al. 2008; Lewicki et al. 2002; Marconi et al. 2012).

2.4.6. Osmotic as low cost dehydration

Osmotic dehydration has proven to have minimal cost during processing of fruits and vegetables (Panagiotou et al. 1999; Madamba and Lopez 2002;
In addition, Chenlo et al. (2006a; 2006b and 2007) recommended ambient temperature (25°C) during osmotic dehydration of chestnuts as it requires no additional thermal energy and appears to have no significant difference compared to higher (e.g. 40°C) solution temperature in terms of $W_L$ and $S_G$ of the product.

Several authors have investigated the practicality and technical feasibility of recycling the osmotic solutions. Garcia-Martinez et al. (2002) showed that the water activity of the solutions and the drying kinetics of kiwifruit showed no significant change after 10 cycles during osmotic dehydration with the same solution concentration (no make-up of solution). Valdez-Frugoso et al. (1998) in their study using sucrose (60% w/w) with apple noticed no significant changes in results after up to 20 cycles of osmotic dehydration using the same osmotic solution concentration (no make-up of the solution). Marconi et al. (2012) also found that the mass transfer kinetics remained relatively constant up to 15 cycles when sucrose (no make-up of the solution) syrup was used to dehydrate peaches.

2.4.7. Disadvantages of Osmotic Dehydration

In spite of the numerous advantages of osmotic dehydration, there are also disadvantages (Falade and Igbeka 2007). Soluble solid leaching and extensive solids uptake are the main deficiencies of osmotic dehydration (Lazarides et al. 1995). Chiralt et al. (2005) also found that the cell turgor (which is the main factor that contributes to the mechanical properties of plant tissue), can be lost due to induced osmotic treatments. Furthermore, osmotic dehydration process is time-consuming (Rastogi et al. 2000; Torres et al. 2006).
and causes shrinkage on tissue depending on the concentration of the osmotic dehydration process (Nieto et al. 2001). Lastly, osmotic dehydration facilitates only partial removal of moisture (up to 50%), thus final drying is still needed for storage and processing (Dhingra et al. 2008).

2.5. Osmotic dehydration of chestnuts

Chenlo et al. (2006a) conducted osmotic dehydration of Spanish variety chestnuts using NaCl as osmotic solution under different experimental conditions. The study aimed to determine experimentally the osmotic drying kinetics and colour parameters in different experimental conditions. Four samples (10g each approximately) were analysed at different time intervals with initial moisture content of 0.571±2.4 (wet basis). Samples osmosed with 26.5% (w/w) NaCl solution had greater rates of solids’ uptake compared to lower NaCl solution (22% w/w). They also found-out that working at 22% (w/w) NaCl concentration and near ambient temperature (25°C) resulted in relatively high (compared to 26.5 % w/w) average ratio of $W_L/S_G$. Possibly at the highest concentration (26.5% w/w) the osmotic pressure is so high that breaks the cellular walls and the solutes can penetrate easily into the product. Also, chestnuts osmosed with 26.5% (w/w) NaCl solution had poor appearance (e.g. colour characteristics) after osmotic dehydration. A small but very dark zone on the surface of chestnuts was developed (which the authors suggested was due to chemical activated reactions for the NaCl ions has developed). The penetration of NaCl on the bulk of chestnuts after four hours (typical periods of contact) during osmotic dehydration process was only significant in the zone near the surface (less than 4mm).
Chenlo et al. (2006b) also investigated osmotic dehydration of a Spanish variety of chestnut using glucose solutions. Five samples (9g each) with 0.564±0.02 (wet basis) initial moisture content were used for each experiment. Hand-peeled, whole chestnuts (shell and pellicle removed) were processed at three different temperatures (25, 35 and 45°C) and glucose concentrations (40, 50 and 56.5% w/w). Results revealed that increased temperature and glucose concentrations showed greater rates of dehydration, reaching lower values of moisture content. $W_L$ and $S_G$ were also affected by the temperature of the osmotic solutions, but not in the same way as the normalised moisture content (there is only slight differences on NMC between 40 to 56.5% w/w). However, the two highest glucose concentrations (50 and 56.5% w/w) showed no appreciable dependence of $W_L$ and $S_G$ for each of the three on temperature. Also, they found that after about four hours, the penetration of glucose into the chestnut was less than 4 mm from the surface. The study concluded that the optimum solution concentration was 56.5% (w/w) glucose, since it offered appreciable water removal without adding too much solute to the chestnut tissue. Operating at 25°C (near ambient conditions) was considered the most economical.

Osmotic dehydration of using sucrose solution on an individual Spanish variety chestnut was also conducted by Chenlo et al. (2007). The study aimed to model the osmotic dehydration kinetics using a diffusional model taking into account the process temperature, concentration and operation time. They used a small variety of chestnut (average weight of 9g) with five whole chestnuts per sample for determining the osmotic dehydration kinetics and two chestnuts per
sample for composition profile experiments. The samples were peeled (shell and pellicle removed) and cut. They found that the mass transfer rates were proportional to the temperature and concentration of the osmotic solution. Their analysis on the profiles of moisture and sucrose content showed that the external slabs (4mm thickness from the outer surface) have the highest average contents of solute. The authors concluded that 60% (w/w) sucrose osmotic solution offers appreciable water removal without adding too much solute to the chestnuts. With regard to the temperature, although 45°C offered the best results in terms of $W_L$, for optimal economic conditions, working at ambient temperature (25°C) was recommended.

2.6. Osmotic Dehydration of Cassava

Andres et al. (2004) conducted a osmotic dehydration of cassava slices (6mm thickness) using NaCl (20% w/w) solution for 60 minutes osmotic time at nearly ambient temperature (30°C) conditions before air drying (micro-wave drying). They revealed a 3.4% $W_L$ of Spanish variety cassava chips after osmotic dehydration before air drying. In terms of the quality of the osmo-dried product (compared to unosmosed), no significant volume changes and cellular collapsed was observed in the microscope. Finally they concluded osmotic dehydration of cut cassava improved the sensorial quality of the dehydrated products due to the influence of the absorbed NaCl solution.

The study of Betoret (2004) on the drying kinetics of osmosed cassava chips revealed no substantial differences in $W_L$ and $S_G$ for higher temperatures (30 and 45°C) and higher NaCl solution concentrations (20 and 22% w/w).
Moreover, Perera (2010) claimed that controlled drying after osmotic dehydration of cassava chips significantly reduced cyanide (cyanogenic glycoside) content, a potential toxin for humans and animals (Siritunga and Sayre 2004). Similarly, Udensi et al. (2005) found that soaking cassava chips into hypertonic solution (e.g. NaCl) achieved higher reduction of the cyanide.

2.7. Summary

Chestnuts in New Zealand are regarded as an important commercial crop because of its uses for various food and wine products. Drying above 30°C of New Zealand chestnuts showed unfavourable conditions. Although drying technologies were already available, it is not yet fully adopted by the growers because of some technical problems (e.g. unmatched capacity, inappropriate heating control) which hinder the appreciation of the technology. New Zealand chestnuts need to have at least 0.40 (wet basis) moisture content for the effective removal of the shell for further postharvest processing (e.g. shelling and drying). It was also revealed that removing the shell of chestnut is a bottle-neck of chestnut processors in New Zealand and other chestnut producing countries.

Cassava is becoming an important ingredient for animal feed production. Worldwide, sun-drying is the most common method of dehydrating cassava. In the Philippines, drying of cassava during inclement weather conditions is still a problem. Investigations of drying cassava granules/chips using mechanical dryers were presented and were found to have an issue on the long drying time, non-uniformity of drying and quality of the product after air drying. Application of osmotic dehydration using different solutions and parameters (e.g. temperature)
has been found to be advantageous in improving the quality of cassava after air drying.

Osmotic dehydration showed simple, low cost, and can partially reduce the moisture content and improve the quality and shelf-life of mostly commercial crops. There are different parameters (e.g. temperature, product to solution ratio, solution concentration, etc.) that influence the mass transfer rate during osmotic dehydration. Mass transfer kinetics during osmotic dehydration can also enhance in conjunction with different pre-treatment like, blanching, pulse vacuum, etc. On the other hand, looking at the numerous advantages and remarkable effects of osmotic dehydration, defect and difficulties also occurred during osmotic dehydration. Soluble solid leaching, extensive solids uptake and time consuming process are some of the defects of osmotic dehydration.

Osmotic dehydration studies for chestnuts (other specific variety) have been presented however, there is no direct studies reviewed specifically for the New Zealand chestnut varieties for osmotic dehydration before mechanical shelling. Also, an issue on the study of the drying kinetics on the bulk (≥ 1 kg) peeled and unpeeled chestnuts at lower ambient conditions (≥18°C temperature) were overlooked. Thus, the application of osmotic dehydration on the bulk unpeeled chestnuts (specifically chestnuts ≥15g each) sample in conjunction with mechanical shelling was the main issue in this study.

Osmotic dehydration of sliced cassava in conjunction to other drying techniques was also presented however; drying rate, influence of temperature and related technical conditions and the quality of dried material after air drying trials on bulk conditions was not thoroughly explored.
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3 OSMO-DEHYDRATION OF NEW ZEALAND CHESTNUT

3.1. Introduction

The determination of kinetics of osmotic dehydration for the shelled and unshelled New Zealand chestnuts in sodium chloride (NaCl), sucrose, glucose and calcium chloride (CaCl₂) solutions is described in this chapter. The measurements were performed under bulk conditions under the optimum operating conditions as found by previous researchers (Saputra 2001; Chenlo et al. 2006a; Chenlo et al. 2006b; Chenlo et al. 2007; Khoyi and Hesari 2007).

3.2. Materials and Methods

3.2.1. Overview of the experiments

Chestnut samples were collected from Gordonton, Hamilton City, Waikato region. New Zealand chestnut variety known by the number “1015” and colour of the tree ‘red’ (Larsen, 2012), was harvested during the month of April (Southern Hemisphere fall) and stored in ventilated plastic bags (Figure 3.1) at a temperature of 2°C. The mature chestnuts (Figure 3.2) naturally fall to the ground and were manually harvested and removed from the burr prior to storage. The variety of ‘1015’, which is considered a commercial variety in Waikato region (NZCC, 2000) was used for all osmotic dehydration and shelling recovery trials. The flow-sheet for osmotic dehydration kinetics experiments is shown in Figure 3.3. The osmotic dehydration trials were performed at (on average) 20.95 °C and 59% relative humidity (refer to Appendix Figure A. 1).
Figure 3.1. Chestnut samples stored in ventilated chiller in plastic bags

Figure 3.2. Mature chestnuts on the ground
3.2.2. Raw material grading method

Grading of freshly harvested chestnuts was initially performed during harvesting where the good nuts were chosen out from the chestnut burr. The chestnuts used in the study were manually graded for size (approximately 15g
at the field before storage. At the laboratory flotation grading, which has proven to be a useful technique to grade out rotten nuts (worm-eaten, dry or undeveloped) and is successfully employed in New Zealand chestnut industry (Klinac 2012), was used to select the chestnuts for this study. Chestnuts samples that had been in storage (approximately 2 months) were dropped into a tank of water (Figure 3.4). The chestnuts that float (“floaters”) in water are generally not suitable for sale, while chestnuts that sink in water (“sinkers”) are more likely to be saleable.

All chestnuts used for the osmotic dehydration trials and shelling trials after osmotic dehydration in this study were ‘sinkers’. Chestnuts of similar size (approximately 15g each) were manually selected to eliminate, as much as possible, the effect of size on experimental results.
3.2.3. Determination of moisture content

The understanding of moisture content of food materials is a pre-requisite in determining their behaviour in subjecting to a more intense process such as drying (Baker 1997). Also, moisture content is a critical variable for characterising a drying process, and hence for designs of postharvest processing machineries (Sajeev et al. 2010). Therefore, determination of moisture content at the different times was an important component of this research.

3.2.3.1. Standard procedure for moisture content determination

1. The initial mass \( M \) of samples (either individually or collectively) was recorded using a digital balance (precision: 0.0001g).

2. The samples were then placed into oven at temperature of 105°C.

3. The mass of the samples were monitored (about 48 hours) until they reached an equilibrium value (moisture-free).

4. The samples were then placed inside a desiccator for 10 minutes (to cool down), before recording the final mass \( m \).

5. The recorded \( M \) and \( m \) will be converted into moisture content (wet basis and dry basis) which was determined as per equation (3.1) & (3.2):

\[
W = \frac{\text{mass of water}}{\text{mass of solids} + \text{mass of water}}
\]

i.e. \[ W = \frac{(M-m)}{M} \] \hspace{1cm} (3.1)

\[
X = \frac{\text{mass of water}}{\text{mass of dry solids}}
\]

i.e. \[ X = \frac{(M-m)}{m} \] \hspace{1cm} (3.2)
The moisture content (dry basis) may have values greater than 100%, since the amount of water present in a sample may be greater than the amount of dry solids present. A dry basis is often used to evaluate the moisture content since the moisture-free material, if inert, does not lose mass on drying. The bone-dry matter thus provides a mass-balance tie over a drying process (Baker 1997). Nevertheless, for this study the solid contents of the samples could not be used as mass-balance tie because of solute diffusion from the solution into the product, thus wet basis moisture content is more convenient to use for osmotic dehydration studies.

Three replicates for each moisture content determination was employed with approximately 100 g for each replicate (e.g. 4-5 nuts). The moisture content of the samples was determined prior to the start of the osmotic dehydration and after 2 hour intervals up to 24 hours during osmotic dehydration trials.

3.2.4. Osmotic dehydration trials

The osmotic-dehydration trials were conducted in bulk condition, with (≥1 kg) whole chestnuts was used for each trial. Two types of samples were used: ‘shelled’ chestnuts (14-15g each) as shown in Figure 3.5, refers to chestnuts without external hard shell but with the episperm still attached and unshelled chestnuts (18-20g each) as shown in Figure 3.6, refers to chestnuts with external skin (hard-shell) intact. The average osmotic solution temperature throughout the experiments was 20.95°C.
The osmotic solutions used were prepared from commercial NaCl, sucrose, glucose or CaCl$_2$ and distilled water of varying concentrations. The osmotic concentrations were 15 and 22% (w/w) for NaCl; 50 and 60% (w/w) for sucrose; 40 and 56.5% (w/w) for glucose and 50% (w/w) for CaCl$_2$. The latter

Figure 3.5. Shelled chestnut samples (episperm remaining)

Figure 3.6. Unshelled chestnut samples (with external shell intact)
concentrations for NaCl (22%), sucrose (60%) and glucose (56.5%) correspond to the optimum concentration at 20 °C according to Chenlo et al. (2006a), Chenlo et al. (2006b) and Chenlo et al. (2007), respectively. The trials involving sucrose involved the two osmotic concentrations recommended by previous researchers (Chenlo et al. 2007; Yadav et al. 2012), which resulted in higher water loss with minimal sucrose impregnation into the food material when working at ambient conditions. The solution to chestnuts ratio was keep above 10 (Chenlo et al. 2006a) in order that the concentration change on the osmotic solution could be considered negligible. No agitation was employed in any of the experiments. Covered plastic buckets (Figure 3.7) were used for the osmotic dehydration experiments.

![Buckets used during the osmotic dehydration experiments](image)

Figure 3.7 Buckets used during the osmotic dehydration experiments

3.2.4.1. Experimental procedure

The experimental procedure in this study was similar to the earlier studies of Chenlo et al. (2006a; 2006b; 2007), who investigated osmotic dehydration of European chestnuts. However, in this study, bulk samples (1 kg, approximately 60 - 70 nuts) were employed compared to the previous studies
which only considered samples comprising 3-5 chestnuts. This was done to more closely simulate the conditions of a commercial osmotic dehydration operation. Samples, once weighed were submerged in the aqueous solutions within the plastic bucket. To determine the drying kinetics during osmotic dehydration, sub-samples (e.g. 5 chestnuts) were randomly selected and labelled. After 2 hours interval, sub-samples were removed from the solution and blotted in order to remove the excess of osmotic solution over the surface of the chestnuts. The osmotic dehydration process (2 hours interval) was repeated for the remaining sub-samples (up to 24 hours).

Normalized moisture content ($NMC$), weight reduction ($W_R$), water loss ($W_L$) and solids gain ($S_G$) were calculated at different time intervals during osmotic dehydration (2, 4, 6, 8, 10, 12 up to 24h) according to the relationships previously identified by Panagiotou et al. (1999); Moreira et al. (2005) and Chenlo et al. (2006a; 2006b; 2007).

\[
NMC = \frac{(O_f - S_f)}{S_f} / \frac{(O_i - S_i)}{S_i} \tag{3.3}
\]

\[
W_R = \frac{(O_i - O_f)}{(O_i)} \tag{3.4}
\]

\[
S_G = \frac{(S_f - S_i)}{O_i} \tag{3.5}
\]

\[
W_L = W_R + S_G \tag{3.6}
\]

Where $O_i$ represents the initial mass of the sample before osmotic dehydration, $O_f$ is the mass of the sample after osmotic dehydration, $S_f$ is the mass of the solids contained in that sample (osmosed sample) and $S_i$ is the initial mass of the solids contained in that sample before osmotic dehydration.
Drying rate $D_R$ for a given time ($t$) was calculated following the expressions:

$$D_R = \frac{dW}{dt} \tag{3.7}$$

Uncertainty ($\Omega$) (e.g. variation in the measured values due to instrument conditions, ambient condition factors, etc.) was calculated following the equations identified by Bell (2001).

$$
\Omega = \frac{1}{2}(value_{max} - value_{min}) \tag{3.8}
$$

### 3.3. Results and Discussion

#### 3.3.1. Moisture content of sinkers and floaters

Figure 3.8 shows the moisture contents of floaters and sinkers. The results agreed with the previous study of Biju Cletus (2007) which stated that ‘sinkers’ possessed significantly higher moisture content than floaters.

![Figure 3.8. Moisture content for sinkers and floaters chestnut samples after 2 months storage](image)

The samples used for this experiment were previously stored for 2 months at $2^\circ$C. On average the difference in moisture content between sinkers and floaters is $0.294\pm0.13$ (wet basis), meaning that the initial moisture content
of sinkers is almost 50% higher than the floaters in this study. Hence, only sinkers were used in the succeeding experiments.

3.3.2. Kinetics of osmotic dehydration

3.3.2.1. Drying rates in sodium chloride solutions

Figure 3.9 shows the $NMC$ of shelled and unshelled chestnuts osmosed with 15% (w/w) NaCl solution. Results suggest that osmotic dehydration using 15% (w/w) NaCl solutions did not have significant mass transfer rates due to low driving force (Chenlo et al. 2006a) caused by low osmotic concentration.

![Figure 3.9](image)

Figure 3.9. Normalized moisture content (wet basis) shelled and unshelled chestnuts osmosed with 15% (w/w) NaCl (error bars±0.03)

In fact, it is also confirmed that variations of negative values of $S_G$ and $W_L$ were generated up to 8 hours period (e.g. increased moisture content). For this reason 15% (w/w) NaCl solutions were not considered further.

Figure 3.10 presents the normalized moisture content (wet basis) of shelled and unshelled chestnuts osmo-dehydrated with 22% (w/w) NaCl solution.
It is important to highlight from the results that unshelled chestnuts showed a comparable moisture reduction to shelled chestnuts samples. This indicates that the shell and pellicle of the New Zealand chestnuts is not a significant mass transfer resistance during osmotic dehydration with 22% (w/w) NaCl solutions at ambient temperature. It is also confirmed by this study that 22% (w/w) NaCl solution is effective for larger New Zealand chestnut variety (15-20 grams each) and comparable to the $NMC$ (wet basis) values obtained by Chenlo et al. (2006a) on smaller chestnut variety (9 grams each) working at ambient temperature. For shelled and unshelled chestnuts in bulk condition, a dehydration time of 6 - 8 hours was required to reach a pseudo-equilibrium value.

Results for $S_G$ using 22% (w/w) NaCl osmotic solution over time is presented in Figure 3.11. The $S_G$ values generated from this study displayed a significant increase for the first 6 hours for shelled $(0.0385 \pm 0.004 \text{ kg/kg sample})$ and unshelled $(0.057 \pm 0.007 \text{ kg/kg sample})$ chestnuts.
Figure 3.11. Solids gain of shelled and unshelled chestnuts at 22% (w/w) NaCl at ambient conditions

This suggests that osmotic pressure at the beginning of the process is so high that it breaks the cellular wall of the product and the solutes penetrates easily into the product (Sacchetti et al. 2001; Jain et al. 2011). It is also interesting to note that the results generated for shelled samples have a lower $S_G$ values than unshelled sample. This might be explained by the larger solute absorption capacity by the samples with shell because of the presence of the shell tissue (Moreira et al. 2005).

Figure 3.12 shows the drying curves of shelled and unshelled chestnuts during osmotic dehydration with 22% (w/w) NaCl solutions.
Figure 3.12. Comparison of drying rates of shelled and unshelled chestnuts using 22% (w/w) NaCl solution at ambient temperature (error bars ±0.2)

The drying rates between the two samples (shelled and unshelled) are comparable. As expected, drying rate decreased as moisture content of the chestnut decreased. The moisture loss rapidly reduces (2 to 4 hours) and then a pseudo-equilibrium moisture loss was observed after 6-8 hours up to 24 hours. These drying rate results can be compared to the study of Chenlo et al. (2006a), however the values obtained from this study are lower. This may be explained by the fact that these experiments were performed in bulk samples (≥ 1 kg) and larger nuts (approximately 15-20 g each) than those used on the study of Chenlo et al. (2006a).

The results implied that osmotic dehydration using 22% (w/w) NaCl solution can reduce the moisture content of shelled and unshelled chestnuts in bulk conditions at 20.95°C effectively. The optimum osmotic dehydration time was recorded to be 6-8 hours, hence working above this time (8 hours) is not beneficial for either shelled or unshelled chestnuts. The experiments also
showed the shell is not a significant hindrance during osmotic dehydration with 22% (w/w) NaCl solutions. However, due to the presence of shell during osmotic dehydration, the unshelled (with shell tissue) chestnuts generated a higher $S_G$ compared to shelled chestnuts samples.

### 3.3.2.2. Drying rates in sucrose solution

Figure 3.13 shows the NMC (wet basis) of osmosed shelled and unshelled chestnuts in 50% (w/w) sucrose solution. The results show that the chestnut samples (shelled and unshelled) after 2 hours of osmotic dehydration started to increase the normalised moisture content.

![Normalized moisture content of shelled and unshelled chestnuts at 50% (w/w) sucrose solution at ambient conditions (error bars ±0.02)](image)

With these results, it can be concluded that shelled and unshelled bulk chestnuts will have a moisture loss up to 2 hours only during osmotic dehydration with 50% (w/w) sucrose solution. Thus, continuing the experiment more than 2 hours is not beneficial in terms of moisture loss. Hence 50% (w/w) sucrose solution was not used for further experiments.
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The $NMC$ (wet basis) after osmotic dehydration for shelled and unshelled chestnuts using 60% (w/w) sucrose solution at ambient temperature is shown in Figure 3.14. A pseudo-equilibrium value did not appear to be reached in any of the drying trials. The lowest moisture reduction was recorded after 8 hours of dehydration time. Although, the shelled samples had higher initial moisture content ($0.54\pm0.13$, wet basis) compared to the unshelled ($0.50\pm0.13$ wet basis) samples prior to the experiments, it is interesting to note that unshelled chestnut samples exhibited similar moisture reduction.

![Figure 3.14. Normalized moisture content of shelled and unshelled chestnuts at 60% (w/w) sucrose at ambient conditions (error bars ±0.025)](image)

A reduction of 20% from the initial moisture content was recorded both for shelled and unshelled samples up to 8 hours period. After 8 hours the $NMC$ of the samples (shelled and unshelled) did not reduce any further. Thus, the results suggest that osmotic dehydration should not be employed for periods longer than 8 hours using 60% (w/w), which coincides with the recommendation of Chenlo et al. (2007).
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The $S_G$ values obtained during osmotic dehydration of shelled and unshelled chestnuts with 60% (w/w) sucrose solutions are presented in Figure 3.15. With regards to the $S_G$ of shelled and unshelled chestnuts, a pseudo-equilibrium value did not appear to be reached in any of the drying trials.

![Figure 3.15. Variations of solids' gain of shelled and unshelled chestnuts in 60% (w/w) sucrose at ambient conditions](image)

An interesting observation has been plotted that the $S_G$ values of shelled samples is higher compared to unshelled chestnuts after 2 hours. However, it was reversed (lower) after 4 hours. This could be explained by the fact that the solute impregnation during osmotic dehydration is more effective at shorter times with samples without cell wall (e.g. shell for chestnuts) in which the solution impregnates the surface of food material quickly (Moreira et al. 2005; Guine and Fernandes 2006). Nevertheless, unshelled chestnuts has higher $S_G$ (from 4-6 hours) due to the presence of the shell and pellicle. In general, the results obtained with respect to the maximum solids' gain value (after 8 hours) for shelled and unshelled samples was lower compared to the data of Chenlo et al. (2007) using 60% (w/w) sucrose osmotic media at 25°C.
Drying rates of shelled and unshelled chestnuts during osmotic dehydration with 60% (w/w) sucrose solution is presented in Figure 3.16. The moisture loss rapidly reduces for the first 4 hours and then a pseudo-equilibrium moisture loss was observed after 8 hours up to 10 hours.

![Figure 3.16. Drying rates of shelled and unshelled chestnuts using 60% (w/w) sucrose solution (error bars ±0.08)](image)

There is no significant difference between shelled and unshelled samples in terms of the drying rates. The result also indicates that the shell is not a significant barrier during osmotic dehydration of bulk chestnuts using 60% (w/w) sucrose solutions at 20.95°C. The results on the drying rates obtained from this study (with larger size, 15-20 grams each) are comparable to the results obtained by Chenlo et al. (2007) with smaller (9 grams each) size working on 60% (w/w) sucrose solutions at near ambient (25°C) conditions.

The study allow to conclude that 60% (w/w) sucrose solutions can reduce the moisture content of shelled and unshelled chestnuts. The optimum osmotic time with maximum moisture loss of the product was 8 hours. The unshelled
New Zealand chestnut variety had comparable drying rates to shelled chestnut samples with 60% (w/w) sucrose solutions in bulk condition at 20.95°C.

3.3.2.3. Drying rates in glucose solutions

Figure 3.17 shows the NMC (wet basis) results of using 56.5% (w/w) glucose solutions during osmotic dehydration of shelled and unshelled chestnuts. Shelled samples showed an increased NMC after 2 hours up to 8 hours during osmotic dehydration process. While the unshelled samples showed an 85% NMC after 2 hours of osmotic dehydration, however an increasing trend was plotted from 4 to 10 hours of osmotic dehydration.

![Figure 3.17](image-url)

**Figure 3.17. Normalized moisture content of shelled and unshelled chestnuts at 56.5% (w/w) glucose at ambient conditions (error bars ±0.02)**

These results were not consistent with the results of the study of Chenlo et al. (2006b) on the Spanish variety shelled and cut chestnuts who found that the use of 56.5% (w/w) glucose osmotic solutions at 25°C showed appreciable water removal without adding too much solutes (glucose) to the chestnuts. One reason could be due to the harder (Ferrini 1997) and thicker external shell (Klinac 2012) of New Zealand variety chestnuts that greatly provides adhesive
substances in the shell during osmotic dehydration as also mentioned by Biju Cletus and Carson (2008). These results suggested that glucose solution (56.5% w/w) was not worth investigating further as a solution for osmotic dehydration for shelled and unshelled New Zealand chestnuts.

3.3.2.4. Drying rates in calcium chloride solutions

Figure 3.18 shows the NMC (wet basis) of shelled and unshelled chestnuts using a 50% (w/w) CaCl$_2$ solution. A pseudo-equilibrium moisture reduction was reached after 6 hours of dehydration for shelled samples, while there’s no pseudo-equilibrium moisture reduction was plotted for the unshelled samples. Shelled and unshelled samples exhibited a comparable moisture reduction every 2 hours. Samples showed average moisture reduction of 0.0305±0.06 (wet basis) and 0.0299±0.02 (wet basis) per hour for shelled and unshelled chestnuts, respectively.

![Normalized moisture content of shelled and unshelled chestnuts at 50% (w/w) CaCl$_2$ at ambient conditions.](image)

Figure 3.18. Normalized moisture content of shelled and unshelled chestnuts at 50% (w/w) CaCl$_2$ at ambient conditions.
These results were in agreement in terms of the NMC values with the previous investigation conducted by Lewicki et al. (2002) and Chavan and Amarowicz (2012).

Although the drying rates in CaCl$_2$ (50% w/w) was higher compared to NaCl (22% w/w) and sucrose (60% w/w) solutions, an observation was made that the chestnut samples (shelled and unshelled) turned dark and soft when osmosed with high amount of CaCl$_2$ (e.g. 50% w/w). Therefore, the use of CaCl$_2$ osmotic solutions was not investigated further for shelled and unshelled New Zealand chestnuts.

3.3.3. Water loss over solids gain for unshelled chestnut

The ratio of $W_L$ and $S_G$ for unshelled samples with respect to time for 22% (w/w) NaCl and 60% (w/w) sucrose at 20.95°C was analyzed. This was undertaken to identify the possible osmotic time that would give the highest moisture loss with minimal solids’ gain in unshelled chestnut samples.

Table 3.1 shows the ratio of $W_L/S_G$ recorded for NaCl and sucrose after osmotic dehydration. The data for 22% (w/w) NaCl solution shows that the maximum $W_L$ with negligible $S_G$ on the samples occurred after 8 hours of osmotic dehydration. However, at the start of the dehydration (up to 2 hours), 60% (w/w) sucrose showed higher $W_L/S_G$ values compared to NaCl (22% w/w).
Table 3.1. Experimental $W_L/S_G$ values over time of osmotic dehydration of chestnut with NaCl and sucrose solutions at ambient conditions (error±0.05)

<table>
<thead>
<tr>
<th>Time</th>
<th>22% NaCl</th>
<th>60% sucrose</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>1.238</td>
<td>1.586</td>
</tr>
<tr>
<td>4</td>
<td>1.288</td>
<td>1.231</td>
</tr>
<tr>
<td>6</td>
<td>1.260</td>
<td>0.989</td>
</tr>
<tr>
<td>8</td>
<td>1.509</td>
<td>1.300</td>
</tr>
</tbody>
</table>

The tabulated results show no significant difference between the two solutions (at different concentrations) in terms of $W_L/S_G$. Using NaCl (22% w/w) solution, 8 hours osmotic time shows significant moisture loss (compared to 2-6 hours) than NaCl gained. As for sucrose (60% w/w) solution, although the highest $W_L/S_G$ value was recorded after 2 hours, 8 hours osmotic dehydration was still the optimum time, since it produces greater moisture loss compared to 2-6 hours as discussed in Section 3.2.2.2. The ratio of $W_L/S_G$, however, on the final hour (after 8 hours) are low compared to the previous works of Chenlo et al. (2006a) and Chenlo et al. (2007), for NaCl (22% w/w) and sucrose (60% w/w) at 25°C solution temperature, respectively.

This indicates that 22% (w/w) NaCl or 60% (w/w) sucrose solutions will have an optimum moisture loss with minimal solids’ gain after 8 hours of osmotic dehydration for fresh unshelled New Zealand chestnuts. Chapter 4 discusses the impact of the osmotic dehydration process on chestnut processing and quality.
3.4. Summary

The initial moisture content of the chestnut samples used in the entire experiments ranged from 0.54±0.04 to 0.63±0.04 (wet basis). Low NaCl (15% w/w) and sucrose (50% w/w) solutions trialled for shelled and unshelled chestnuts showed unfavourable results for \( \text{NMC}, S_G \) and \( W_L \). Glucose with 56.5 (w/w) solutions showed an increased \( \text{NMC} \) values for the unshelled chestnuts after 2 hours. Using CaCl\(_2\) (50% w/w) as osmotic solution produced unfavourable colour and texture changes on both shelled and unshelled chestnuts.

The results of using 22% (w/w) NaCl and 60% (w/w) sucrose solutions highlighted unshelled chestnuts performed a similar moisture reduction to shelled chestnuts samples. This suggested that the shell of the New Zealand chestnuts is not a significant barrier to osmotic dehydration with 22% (w/w) NaCl or 60% (w/w) sucrose solutions at 20.95\(^\circ\)C. It is also interesting to note that the results generated for shelled samples have a lower \( S_G \) values than unshelled sample (both for NaCl and sucrose solutions).

The optimum osmotic dehydration time of using NaCl (22% w/w) and sucrose (60% w/w) solutions for shelled and unshelled chestnuts was 6-8 hours. This indicates that osmotic dehydration beyond 8 hours is not beneficial for shelled and unshelled chestnuts in bulk conditions at 20.95\(^\circ\)C. The results also showed that the water loss rates up to this time (6-8 hours) were still higher compared to solids’ gain. Thus, the results implied that the optimum shelling recovery of New Zealand chestnuts could be attained after 6-8 hours of osmotic dehydration using either 22% (w/w) NaCl or 60% (w/w) sucrose. The shellability study was discussed in Chapter 4.
4 SHELLABILITY OF OSMOSED NEW ZEALAND CHESTNUT

4.1. Introduction

The shelling recovery of New Zealand chestnuts after osmotic dehydration and a comparison of shelling recovery to unosmosed samples (control) is presented in this chapter. Quality evaluation in terms of the colour and texture of the final product is also described.

4.2. Materials and Methods

4.2.1. Overview of the experiments

Chestnuts after osmotic dehydration with 22% (w/w) NaCl and 60% (w/w) sucrose solutions at different time intervals were subjected to shelling experiments. This was done to evaluate the effect of the osmotic dehydration on the shellability of New Zealand chestnuts (variety ‘1015’). The process flowsheet for the shellability trials of the osmosed chestnuts is presented in Figure 4.1.

4.2.2. Shelling experiments for chestnuts

A mechanical shelling machine was used to determine the effect of osmotic dehydration on the mechanical shelling recovery of New Zealand chestnuts. A control sample comprised of unosmosed chestnuts from the same variety as the osmosed samples was used for comparison. Three different osmotic dehydration times (4, 6 and 8 hours) were used as treatments both for sucrose (60% w/w) and NaCl (22% w/w) solutions. There was approximately 1 kg of chestnut for every treatment. While waiting for the availability of the mechanical shelling machine, the chestnuts osmosed with sucrose and NaCl were
temporarily stored for 24 hours at (2\degree C) (to maintain the moisture of the samples) together with the unosmosed control samples prior to the shelling experiments.

![Diagram](image)

**Figure 4.1. Process flow sheet of chestnut shellability experiments**
The mechanical shelling machine (Figure 4.2) was designed by Barry Stevenson of Langdon Engineering Limited and produced by HortResearch, Ruakura, specifically for use by Kiwi Chestnut Cooperative Company Limited (KCCCL) (Klinac 2012). When the term “shelling” is used in this study, this means the removal of the outer skin (shell) of the chestnut, but not the episperm.

The shelling recovery trials proceeded as follows:

1. The initial moisture content (before osmotic dehydration) of the chestnuts at the time of each trial was determined as described in section 3.2.3.1.

2. Then 1 kg chestnut sample was osmo-dehydrated with sucrose (22% w/w) NaCl or sucrose (60% w/w) solutions for 4, 6 or 8 hours.

3. The final moisture content after the osmotic dehydration process was determined using equation 3.1 for each osmosed sample.
4. After the osmotic dehydration the nuts were passed through the mechanical shelling machine.

5. Then the nuts were graded and counted, and recoveries (as defined by equation 4.1) were determined.

The term ‘wholenuts’ (Figure 4.3(a)) corresponds to the chestnuts for which the shell was fully removed but with episperm remaining; the term ‘unshelled’ (Figure 4.3(b)) refers to those whole chestnuts with some shell and episperm remained and the term ‘broken’ refers to chestnuts (Figure 4.3(c)) which were broken during shelling experiments. The number of wholenuts, unshelled and broken chestnuts was counted after each shelling trials.

The shelling recovery was determined by recording

a. The total number of chestnuts subjected to shelling for individual trials

b. The number of ‘wholenuts’, ‘unshelled’ and ‘broken’.

c. The shelling recovery was determined from Equation (4.1):

\[
\text{Recovery} = \frac{\text{number of whole nuts}}{\text{Total number of osmosed nuts}} \times 100\% \tag{4.1}
\]

Figure 4.3. Chestnuts termed as (a) shelled; (b) unshelled and (c) broken nuts after shelling trials
4.2.3. Quality evaluation

Colour is an important parameter for consumer acceptability of a food product (Singhet al. 2008) and is often assessed by visual or instrumental evaluation (Osorio et al. 2007). The colour of osmo-dehydrated chestnuts compared to the unosmosed samples was manually evaluated using Nikon camera DLSR 3100.

4.2.4. Data analysis

Analysis of variance (ANOVA) was carried out to see the influence of osmotic solution type and osmotic dehydration time on the shellability of New Zealand chestnuts. A single-factored ANOVA was conducted using the Microsoft Excel Program with two variables (osmotic time and solution). A single-tailed T-test was conducted to compare with unosmosed chestnuts the effect of osmotic dehydration on the effectiveness of mechanical shelling of New Zealand chestnuts.

4.3. Results and discussion

4.3.1. Shelling recovery of chestnuts after osmotic dehydration

4.3.1.1. Influence of NaCl solution on shelling recovery of chestnuts

Figure 4.4 displays the results generated from the shellability experiments after osmotic dehydration of using 22% (w/w) NaCl solution. Osmo-dehydrated chestnuts had higher wholenuts recovery values compared to the unosmosed (control) samples. The initial moisture content of the samples before osmotic dehydration was 0.51±0.04 (wet basis). The final moisture content after
each time intervals of 4, 6 and 8 hours was recorded in wet basis as follows:

0.42±0.04, 0.39±0.04 and 0.37±0.04, respectively.

![Graph showing recovery of wholenut, unshelled, and broken nuts during different osmotic dehydration times.](image)

Figure 4.4. Shellability of chestnuts after osmotic dehydration to 22% (w/w) sodium chloride with respect to time.

The result (after 8 hours osmotic dehydration) in wholenut recovery during mechanical shelling relative to the unsmososed (control) was improved from 28.5±0.04% to around 45.3±0.04%. Samples osmosed for 4 and 6 hours also improved the percentage of wholenut recovery compared to unosmosed (control) samples with values 35.18±0.042% and 43.26±0.042%, respectively.

According to the single-tailed T-test ($\alpha = 0.05$) presented in Table 4.1 after shelling the osmosed chestnuts with NaCl (22% w/w) solution, the value of $P$ ($5.49\times10^{-7}$) for the wholenuts recovery was far below $\alpha = 0.05$. This suggest that the apparent increase in the percentage wholenuts recovery of chestnuts due to osmotic dehydration with 22% (w/w) NaCl was statistically significant. Also, the fraction of unshelled nuts decreased significantly compared to unosmosed samples.
Table 4.1. T-test analyses among the percentage values of chestnuts processed with 22% (w/w) sodium chloride before shelling.

<table>
<thead>
<tr>
<th>Processed Variable</th>
<th>Control</th>
<th>4-hours (OD)</th>
<th>6-hours (OD)</th>
<th>8-hours (OD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>whole nuts</td>
<td>64.29</td>
<td>67.29&lt;sup&gt;s&lt;/sup&gt;</td>
<td>72.63&lt;sup&gt;s&lt;/sup&gt;</td>
<td>71.72&lt;sup&gt;s&lt;/sup&gt;</td>
</tr>
<tr>
<td>Unshelled</td>
<td>21.43</td>
<td>12.96&lt;sup&gt;s&lt;/sup&gt;</td>
<td>7.37&lt;sup&gt;s&lt;/sup&gt;</td>
<td>2.02&lt;sup&gt;s&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>s</sup> – significant at 95% level of confidence

With regards to the recovery of the broken nuts, it coincides with the results of Chenlo et al. (2006a) that chestnuts subjected to NaCl (22% w/w) become harder and more brittle as the time of immersion increased. The brittleness makes the mechanical shelling process easier, however increases the number of broken nuts.

If a processors aim is to get higher wholenuts for chestnut products (e.g. for export), a 6 hour osmotic dehydration period using 22% (w/w) NaCl is recommended before shelling. Otherwise, if the processor prefers good shelling recovery and considers the broken nuts to be used for second stage by-products processing (e.g. crumb, chestnut flour), 8-hour osmotic dehydration time before mechanical shelling is more advantageous.

4.3.1.2. Influence of sucrose solution on shelling recovery of chestnuts

The shellability of New Zealand chestnuts osmo-dehydrated with 60% (w/w) sucrose is presented in Figure 4.5. An improvement of around 16.5±0.08% was recorded on the wholenuts recovery of chestnut osmosed with 60% (w/w) sucrose after 8 hours of osmotic dehydration relative to the unosmosed (control) bulk chestnut samples.
Figure 4.5. Shellability of chestnuts after osmotic dehydration to 60% (w/w) sucrose with respect to time

The samples used for this experiment were taken from different batch (e.g. lastly harvested chestnuts) with same variety ('1015) as those samples used for osmo-dehydration using NaCl solutions before shelling. The samples used for this experiment has higher initial moisture content (0.54±0.04, wet basis) compared to samples used with 22% (w/w) NaCl solution with 0.51±0.04 (wet basis).

The mean results using single-tailed T-test (α = 0.05) of wholenuts recovery has statistically increased (compared to unosmosed samples) at 8 hours osmotic dehydration time (Table 4.2); however, 4 and 6 hours showed no statistically significant increase (compared to control). The quantity of unshelled chestnuts decreased significantly (compared to unosmosed samples) over time.
Table 4.2. T-test analyses among the percentage mean values of wholenuts and unshelled chestnuts processed with 60% (w/w) sucrose osmotic dehydration before shelling

<table>
<thead>
<tr>
<th>Processed Variables</th>
<th>Control</th>
<th>OD (4h)</th>
<th>OD (6h)</th>
<th>OD (8h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wholenuts</td>
<td>40.575</td>
<td>50.00 ns</td>
<td>48.53 ns</td>
<td>58.21 s</td>
</tr>
<tr>
<td>Unshelled</td>
<td>55.39</td>
<td>39.89 s</td>
<td>45.59 s</td>
<td>37.31 s</td>
</tr>
</tbody>
</table>

s - significant at 95% level of confidence  
ns – not significant at 95% level of confidence

It is also important to note that chestnuts osmo-dehydrated with high concentration of sucrose (i.e. 60% w/w) increased the texture characteristics of chestnuts over time, as indicated by the broken nut recovery values generated from the experiments. When the time of immersion increased the broken nuts values decreased. This is consistent with results from other studies (Telis et al. 2005; Chenlo et al. 2007) using other chestnut varieties.

Based on these results, wholenuts recovery from mechanical shelling can be improved (compared to unosmosed samples) after 8 hours of osmotic dehydration using 60% (w/w) sucrose solution with significant increase in the wholenuts recovery during shelling.

4.3.1.3. Influence of solution on shelling recovery of chestnuts

The quality (efficiency) of using different solutions (NaCl or sucrose) relative to the unosmosed (control) samples during osmotic dehydration prior to mechanical shelling was determine by the ratio of shelled/unshelled chestnuts. Osmotic dehydration for 6 and 8-hour period using NaCl (22% w/w) solution statistically showed significant differences compared to the unosmosed chestnuts (see Table 4.3).
Table 4.3. Experimental shelled/unshelled values over time of osmotic dehydration of chestnut with NaCl and sucrose solutions at ambient conditions

<table>
<thead>
<tr>
<th>Time</th>
<th>Osmotic solutions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>22% NaCl</td>
</tr>
<tr>
<td>control</td>
<td>3</td>
</tr>
<tr>
<td>6</td>
<td>9.857(^s)</td>
</tr>
<tr>
<td>8</td>
<td>35.500(^\ddagger)</td>
</tr>
</tbody>
</table>

\(s\) – significant at 95% level of confidence

In the same way, using sucrose solution (60% w/w), osmosed chestnuts with 6 and 8 hours showed significant differences relative to the unosmosed (control) samples.

Chestnuts osmo-dehydrated with NaCl gives the highest value of shelled/unshelled recovery of 35.5 relative to the control with only 3 after 8 hours osmotic dehydration time. While shelled/unshelled recovery for sucrose was 1.56 compared to only 0.82 for the unosmosed (control) samples. This could be due to the roughly half-molecular concentration of sucrose solutions and significantly lower osmotic pressure compared to NaCl (Panagiotou et al. 1999). The highest value shelled/unshelled for NaCl (22% w/w) and sucrose (60% w/w) confirmed that 8 hours was the optimum osmotic dehydration time for the unshelled (with shell intact) prior to mechanical shelling.

Therefore, the study allow to conclude that chestnuts osmosed with NaCl (22% w/w) or sucrose (60% w/w) solutions can produce a higher wholenut recovery during shelling compared to unosmosed chestnuts after 8 hours of osmotic dehydration.
4.3.2. Quality analysis of osmosed chestnuts

During osmotic dehydration, the change of colour is one of the most significant changes observed (Osorio et al. 2007). Analysis of the quality of osmosed samples was undertaken for chestnuts osmosed with 22% (w/w) NaCl and 60% (w/w) sucrose for 8 hours (optimum wholenuts recovery) to qualify any adverse effects of osmotic dehydration on shelled and unshelled chestnuts. Unshelled chestnuts osmosed with 22% (w/w) NaCl solutions (as shown in Figure 4.6) displayed minimal effects on the physical structure of the samples. A slight change in colour (darker) was noticed on the samples after osmotic dehydration.

Looking at the episperm of the chestnuts, using 22% (w/w) NaCl solution during osmotic dehydration showed a change in colour (e.g. darker) on the shelled samples (Figure 4.7).
These observations were in agreement with the study of Moreira et al. (2011) on shelled and cut chestnuts after 8 hours of osmotic dehydration prior to convective air drying. This could be the penetration of salt ions that increased the enzymatic browning reactions on the chestnuts.

Figure 4.8a shows the unshelled chestnuts processed with 60% (w/w) sucrose after 8 hours of dehydration. Visual assessment with the aid of a high definition camera, showed no significant effect on the texture of the samples compared to unosmosed samples (Figure 4.8b). Furthermore, when the shell was removed, the samples immersed with 60% (w/w) sucrose for 8 hours showed no colour difference as compared to unosmosed (control) chestnuts (Figure 4.9).
4.4. Summary

Unshelled chestnuts after osmotic dehydration with either 22% (w/w) NaCl or 60% (w/w) sucrose solutions showed statistically significant increase in the wholenuts recovery and reduction in the number of unshelled nuts after mechanical shelling. Also, the results revealed that osmotic dehydration of bulk chestnut samples using 22% (w/w) NaCl or with sucrose (60% w/w) had greater wholenut recovery compared to unmososed (control) chestnuts. The osmotic

Figure 4.8. Physical structure of unshelled (a) osmosed (b) unosmosed chestnuts after 8 hours osmotic dehydration with 60% (w/w) sucrose

Figure 4.9. Physical structure of shelled (a) osmosed (b) unosmosed chestnuts after 8 hours osmotic dehydration with 60% (w/w) sucrose
dehydration time significantly affects the shellability of chestnuts. Optimum recorded wholenuts recovery for both NaCl and sucrose solutions were after 8 hours of osmotic dehydration prior to mechanical shelling.

In terms of the quality of the osmosed product, a slight change in colour (darker) was noticed on the samples osmosed with 22% (w/w) NaCl both for shelled and unshelled samples. Shelled and unshelled chestnuts osmosed with 60% (w/w) sucrose showed no significant differences on the texture and colour as to the unosmosed chestnuts.
Chapter 5: Philippine cassava

5 OSMO-DEHYDRATION OF PHILIPPINE CASSAVA

5.1. Introduction

Osmotic dehydration experiments using NaCl or sucrose solutions were performed to determine the dehydration kinetics of 15mm cassava chips ambient conditions. The $NMC, S_g$ and $W_L$ were analyzed over a period of 210 minutes at 30 minute intervals.

5.2. Materials and Methods

5.2.1. Overview of the experiments

Generally, in Philippines cassava is harvested manually (Figure 5.1) when it is mature (after 10 months). The samples for this study were collected from Porac, Pampanga, Philippines. The samples collected came from a single field, were of one variety (Lakan White) and were gathered within a one week interval.

Figure 5.1. Harvesting of cassava tubers in the Philippines
The process flow diagram for the cassava drying kinetics experiments is presented in Figure 5.2. Freshly harvested cassava tubers were cleaned to remove the clinging soil and dirt before cutting into chips that were used for osmotic dehydration trials. The relative humidity and osmotic solution temperature were recorded during the trials.

![Process Flow Diagram](image)

Figure 5.2. Process flow diagram for cassava experiments

5.2.2. Raw materials grading method

To retain the freshness of cassava after harvest, the collected cassava tubers remained attached to the stem until used. Collection and selection of cassava samples were done by ‘feel and roll’ method. In the ‘feel and roll’ method, the samples are touched and pressed softly by hand: if the sample is
still hard to press, then it is still fresh; however, if the sample is rotten it would feel spongy. The cassava tubers were carefully selected according to its diameter to maintain the uniformity of the samples. After selection, the samples were manually chopped (Figure 5.3) into 15 mm thick slices for all the moisture content and osmotic dehydration trials. The thickness and diameter were measured using a vernier calliper.

![Figure 5.3. Manual chopping of cassava tubers during the experiments](image-url)

5.2.3. Moisture content determination of samples

Moisture content of chipped cassava was an important factor in determining its quality and expected market price (SMC 2011). Cassava with final moisture content of higher than 0.14 (wet basis) is not acceptable for retail (de Luna, 2011). Moisture content analyses for cassava experiments were executed in a similar manner as for chestnuts, following the steps outlined in Section 3.2.3.1.
The moisture content of cassava chip samples was determined with three replicates from each sample. Each replicate had a total weight of 10 grams with approximately the same thickness (15mm) and diameter (30 mm).

5.2.4. Osmotic dehydration trials

The solutions used during the osmotic dehydration trials were commercial NaCl or sucrose in distilled water. To determine the osmotic dehydration kinetics, different osmotic solution ratios were used. For NaCl, 22 and 25% (w/w) were used and 60% (w/w) for sucrose solutions. Although, 22% (w/w) NaCl solution was earlier (Section 3.3.2.1) identified and also found by Chenlo et al. (2006a) as the optimum level, this study found that 25% (w/w) NaCl solution had slight increase in the moisture loss and $S_G$ in cassava chips. The chipped cassava samples were in bulk (≥1 kg) conditions and were unpeeled, since unpeeled cassavas are acceptable for retail. The average osmotic solution temperature throughout the experiments was 24.5°C. The cassava to osmotic solution ratio was kept above 10 in order to minimise the concentration change on the osmotic media (Khoyi and Hesari 2007). No agitation was employed in any of the experiments. A bucket covered with plywood lid (Figure 5.4) was used for the osmotic dehydration experiment.

![Figure 5.4. Plastic bucket used during the osmotic dehydration experiments](image)
5.2.4.1. Experimental procedures

The experimental procedure was carried out with only a single trial for each solution due to limited time (less than 2 months) available and constraints on the availability of the PHilMech oven which was used for conducting the experiments in the Philippines. Cut cassava samples were weighed (≥1 kg) into a digital scale (precision 0.001 g). Samples once weighed were submerged in the plastic bucket (as shown in Figure 5.5) in the osmotic solutions.

Figure 5.5. Chipped cassava samples submerged into solutions

Sub-samples (approximately 20g) were removed from the container at regular intervals so that moisture content and solids’ gain could be determined over the course of the experiments. Initially, measurement intervals were scheduled for 2, 4, 6, 8, 10 up to 24 hours, however, after the first trial (data not shown) it was found that after 210 minutes of osmotic dehydration, the sample’s weight actually began to increase. Thus, the osmotic time was reduced to 210 minutes with 30 minute composition sampling intervals. After each interval, the samples were blotted to remove excess surface osmotic solution and then
weighed. The dry matter content of the osmosed samples (moisture-free) were determined following the detailed equation of measurement in Section 3.2.3.1 however, oven drying was conducted for 72 hours (Pontawe et al. 2012), rather than 48 hours as was the case with chestnuts. This allowed the masses recorded during the trials to be used in the analysis of $NMC, W_L$ and $S_G$ as determined using the equations presented in Section 3.2.4.1. The uncertainties were calculated as per equation (3.9) presented in section 3.2.4.1.

5.3. Results and Discussion

5.3.1. Moisture content

Figure 5.6 shows the initial moisture content of cassava samples used in this study. The highest value recorded was 0.61±0.01 (wet basis) and 0.55±0.01 (wet basis) for the lowest initial moisture content with an average of 0.57±0.01 (wet basis).

Figure 5.6. Initial moisture content of cassava samples used in the experiments
The experiments were conducted within the month of November (considered part of the dry season in the Philippines). Normally, the initial moisture content of cassava is lower (Pontawe et al. 2012) during dry season (November-April) than in wet season (May – September). The initial moisture content of fresh Philippine cassava ranged from 0.60-0.70 (wet basis) during wet season period, as determined in a previous study (Pontawe et al. 2012).

5.3.2. Kinetics of osmotic dehydration

5.3.2.1. NaCl solutions

Figure 5.7 shows the \( NMC \) (wet basis) of chipped cassava osmosed with 22 and 25% NaCl. The two NaCl solution concentrations (22 & 25%) show significant mass transfer rates during the early stage of dehydration, 30 minutes for 22% (w/w) and 60 minutes for 25% (w/w) NaCl, respectively and later a progressive reduction in transfer rates were observed due to decreasing of the driving forces as the samples were getting closer to apparent equilibrium (after about 90 minutes).

![Normalized moisture content from the osmotic dehydration of cassava with 22 and 25% (w/w) NaCl](image)

**Figure 5.7.** Normalized moisture content from the osmotic dehydration of cassava with 22 and 25% (w/w) NaCl
The results on moisture reduction of using 25% (w/w) NaCl suggested a slight increase in $W_L$ rates compared to using 22% (w/w) NaCl solutions. This would be due to the driving force of less concentrated solution (22% w/w) is smaller compared to 25% (w/w) NaCl solution. Contrary to the recommendation of Chenlo et al. (2006a) on their study of Spanish variety chestnuts, 22% (w/w) was recommended compared to 26.5% (w/w) due to significant difference on $S_G$ (higher concentration NaCl (26.5% w/w) recorded higher solid uptake) and almost similar $W_L$.

The $S_G$ results from the two different salt solutions (22 & 25% w/w) are presented in Figure 5.8. On the average, the $S_G$ of the 25% (w/w) NaCl solution was comparable to the 22% (w/w) NaCl solution.

![Figure 5.8. Variations of solid gain of cassava chips in 22 & 25% (w/w) NaCl solutions at ambient conditions.](image)

The results suggest that 25% (w/w) NaCl is more advantageous compared to 22% (w/w) NaCl due to its slightly higher moisture content reduction while there is no significant difference between the $S_G$ values. Hence,
for chipped cassava, a 25% (w/w) NaCl solution was used for further investigations.

Figure 5.9 displays the variations of $W_L$ and $S_G$ for 25% (w/w) NaCl solutions over time. The $W_L$ and $S_G$ values achieved a pseudo-equilibrium values from the early stage (after 30 minutes) during the experiments.

![Figure 5.9. Variations of water loss and solids’ gain of cassava chips in 25% (w/w) NaCl solutions at ambient conditions](image)

Again, when the osmotic concentration and processing time increased consequently, the $W_L$ and $S_G$ values increased. Significantly, the results show that $W_L$ values were higher compared to $S_G$ values.

5.3.2.2. Sucrose solution

Figure 5.10 displays the $NMC$ (wet basis) of cassava chips using 60% (w/w) solutions over a period of 180 minutes (with 30 sampling minutes interval) at $24.5^\circ$C. It is important to highlight that the reduction in $NMC$ of cassava osmosed with 60% (w/w) sucrose was similar to NaCl (22 or 25% w/w) osmosed samples. Also an interesting pseudo-equilibrium for this experiment appeared to be reached after 90 minutes. Hence, the optimum osmotic time for chipped cassava
in 60% (w/w) sucrose solution was attained after 90 minutes of osmotic dehydration.

![Normalized moisture content from the osmotic dehydration of cassava with 60% (w/w) sucrose](image1)

**Figure 5.10.** Normalized moisture content from the osmotic dehydration of cassava with 60% (w/w) sucrose

Figure 5.11 displays the $W_L$ and $S_G$ during the osmotic dehydration of cassava chips osmosed with 60% (w/w) sucrose solution at 24.5°C. It confirmed that after 180 minutes of dehydration time the $W_L$ decreased, while the $S_G$ continued to increase.

![Variations of solids’ gain and water loss of cassava chips in 60% (w/w) sucrose solution at ambient conditions](image2)

**Figure 5.11.** Variations of solids’ gain and water loss of cassava chips in 60% (w/w) sucrose solution at ambient conditions
5.4. Summary

Generally, increase in osmotic concentration and time also increased the $W_L$ and $S_G$ of chipped cassava osmosed with NaCl and sucrose solutions. The $W_L$ rates for both 25% (w/w) NaCl and 60% (w/w) sucrose solutions have greater rates compared to the $S_G$ values.

The study found that the 25% (w/w) NaCl solutions (up to 210 minutes immersion time) produced slightly greater $W_L$ rates compared to the 22% (w/w) NaCl solution, without significant difference between the $S_G$ values. The moisture loss in terms of NMC of cassava osmosed with 22 or 25% (w/w) NaCl gave a higher results compared to samples osmosed with 60% (w/w) sucrose. The $S_G$ rates using 60% (w/w) sucrose was lower than for 25% (w/w) NaCl.

Also, partial moisture reduction for chipped cassava can be achieved using 60% (w/w) sucrose solutions during osmotic dehydration at ambient conditions for up to 180 minutes without much sugar penetrating the cell tissue of the product. Therefore, both 25% (w/w) NaCl and 60% (w/w) sucrose solutions can be applied before the air drying trials described in Chapter 6.
6 AIR DRYING OF OSMOSED PHILIPPINE CASSAVA

6.1. Introduction

The air drying characteristics of chipped cassava after osmotic dehydration of using 25% (w/w) NaCl and 60% (w/w) sucrose solutions and unosmosed samples (control) are presented in this chapter. Quality evaluation of air dried (osmosed) final product in terms of the colour and hardness is described and compared to unosmosed air dried product.

6.2. Materials and Methods

6.2.1. Overview of the experiments

The process flow sheet for the air drying trials after osmotic dehydration is presented in Figure 6.1.

![Figure 6.1. Process flow sheet of cassava air drying experiments](image-url)
6.2.2. Laboratory test rig drying experiments

To evaluate the effect of the osmotic dehydration on the cassava chips, control samples (unmosed cassava chips) were also dried. Drying trials were performed at 70 and 80°C, which was previously identified by Pontawe et al. (2012) as being the optimum temperature range. The first temperature (70°C) was considered the most economic option, while the latter (80°C) was the upper temperature limit for cassava chips without gelatinization effect on the dried material. The moisture content prior to drying (after osmotic dehydration) was recorded for each trial. A laboratory drying test rig (Figure 6.2) was developed to simulate the operation of the 1-ton/hour capacity belt dryer described in Section 2.3.2. The set-up was installed at the enclosed laboratory room of PHiLMech, Science City of Munoz, Nueva Ecija, Philippines.

![Laboratory drying test rig](image)

Figure 6.2. Laboratory drying test rig used in the study

The schematic diagram of the laboratory drying test rig is shown in Figure 6.3. The drying bin (as shown in Figure 6.3) was made of metal sheet with inside
dimension of 0.2 m in length, 0.2 m in width and 0.15 m in height. The bin had open top, while the bottom was made of perforated metal sheet, with 2.4 mm diameter holes 3 mm apart (square pitch). The side walls were double walled with ceramic fibre insulation in between walls. The bin was fitted above plenum section (as shown in Figure 6.3) of the dryer.

![Figure 6.3. Schematic diagram of the laboratory drying test rig.](image)

During drying, ambient air was drawn in by a 0.23 m diameter axial fan through a long duct with cone damper at intake to regulate air flow rate to the required sample superficial air velocity of 1.8 m/s (velocity of air in the actual belt conveyor dryer). The air passed through three 1.5 KW U-shaped electric heaters controlled by a thermostat. The heated air flowed to the plenum of the drying bin, upward through the sample bed and exhausted in the open air.

6.2.2.1. Laboratory drying test rig procedure

Cassava chips that had been osmosed with 25% (w/w) NaCl solution for 210 minutes or 60% (w/w) sucrose solution for 180 minutes were loaded to the laboratory dryer test rig as shown in Figure 6.4. The laboratory drying trials started when the drying bin loaded with 1 kg of cut cassava at 10.16 cm depth (maximum depth for belt conveyor dryer) was exposed to hot air in upward direction. The belt dryer being simulated has 6 levels with 27 minutes residence time for each level (before the sample falls down to the next level), hence, when the simulated sample residence time was reached (27 minutes) for the first level
of the belt dryer, the drying bin was immediately removed from the dryer and weighed using a digital scale, so that moisture content profiles could be determined.

A 10 g sub-sample randomly selected was loaded to the AD-50 Infrared Moisture Analyzer (Figure 6.5) to get the moisture content (wet basis) at the end of each level. The cassava sample was carefully turned (individually), which simulates the turning effect inside the belt dryer when the cassava pieces fall to the level below, and immediately returned to the drying chamber. Given that there are 6 levels present on the actual belt dryer, 6 cycles of this procedure were performed in each drying experiment until the sample reached 0.13 (wet basis) moisture content, which was the equilibrium moisture content (Baker, 1997). The final moisture content was determined using the procedure described in section 3.2.3.1. The $D_R$ and uncertainties were calculated as per equations (3.8) and (3.9) presented in section 3.2.4.1.
6.2.3. Quality evaluation

Colour is one of the most important parameters for market acceptability of dried food products (Singh et al. 2008). After each trial, visual evaluation (photo analyses using Nikon camera DLSR 3100) was undertaken to record the colour changes after air drying of osmosed, and the control cassava. In addition, cassava (osmosed with NaCl and sucrose) after air drying was subjected to mechanical hardness tester (KutaSelgunsho LTD) as shown in Figure 6.6. The brittleness of the osmo-dried product was compared to unosmosed air dried cassava samples, since cassava when it is dried should not be brittle to minimize the pulverization when it is being used as extenders for animal feeds production (SMC 2011).
6.3. Results and Discussion

6.3.1. Effect of drying temperature on drying rates of osmosed cassava

The drying rate of unosmosed cassava air dried in the belt conveyor increased when the drying temperature increased (Pontawe et al. 2012). It is surprising for these experiments that the temperature change (10°C increase) had no significant increase in the drying rates of osmosed cassava with 25% (w/w) NaCl (Figure 6.7) and 60% (w/w) sucrose solutions (Figure 6.8).

Figure 6.6. Hardness tester used to evaluate osmosed, air dried cassava samples

Figure 6.7. Drying curve for cassava osmosed with 25% (w/w) NaCl solution at 70 and 80°C drying temperature
The analysis of the drying curved shows two falling rate periods (no constant rate period) for both cassava osmosed with 25% (w/w) NaCl and 60% (w/w) sucrose solutions dried for 70 and 80°C, respectively. The first falling rate periods (27- 81minutes) is mainly dependent on the surrounding conditions (e.g. air movement) which affects the mass transfer coefficient (Biju Cletus and Carson, 2008) and the second falling rate period (108- 162minutes) was affected by the nature of the product (Koyuncu et al. 2004) and postulated to depend on internal mass transfer resistance (Nieto et al. 1998).

Thus, it can be drawn from the initial results of this study that increasing the drying temperature (from 70 – 80°C) of laboratory drying test rig (simulation of belt-conveyor dryer) for cassava chips have no benefits in terms of drying rate after osmotic dehydration with 25% (w/w) NaCl and 60% (w/w) sucrose solutions. Hence, 70°C drying temperature is the optimum considering the energy savings of using lower (70°C) temperature compared to drying osmosed cassava at 80°C. Therefore, further investigation on the effects of osmotic
dehydration before air drying in comparison to the fresh cassava (unosmosed) samples in terms of drying rates was limited to 70°C drying temperature.

6.3.2. Effect of osmotic dehydration on the drying rates of cassava

The moisture contents of cassava measured using the infrared moisture analyzer for drying at 70°C is presented in Table 6.1. The initial moisture contents of the cassava used for the control (unosmosed) samples, and samples osmosed with NaCl or sucrose solution were similar; 0.56, 0.55 and 0.55 (wet basis) respectively. As shown in Table 6.1, samples osmosed with 25% (w/w) NaCl solution reached a moisture content of 0.493 (wet basis) after osmotic dehydration and the samples osmosed with 60% (w/w) sucrose solution recorded a similar moisture content of 0.487 (wet basis) prior to air drying trials.

Table 6.1. Variations of moisture content during laboratory drying test rig experiments at 70°C.

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Moisture Content (MC), % (wet basis)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial MC</td>
<td>Control 55.55</td>
</tr>
<tr>
<td>0 - 271</td>
<td>55.55</td>
</tr>
<tr>
<td>27 - 542</td>
<td>38.35</td>
</tr>
<tr>
<td>54 - 813</td>
<td>30.35</td>
</tr>
<tr>
<td>81 - 1084</td>
<td>22.35</td>
</tr>
<tr>
<td>108 - 1355</td>
<td>16.45</td>
</tr>
<tr>
<td>135 – discharge6</td>
<td>12.04</td>
</tr>
</tbody>
</table>

1 - 6 represents the number of layers of simulated belt conveyor dryer

*moisture content after osmotic dehydration

**pseudo-equilibrium values

In addition to the energy savings (due to no thermal energy required) from reducing the moisture content (approximately 11%) of cassava applying osmotic dehydration before air drying, the osmosed samples reached the
acceptable limit of final moisture content 33.33% faster than unmosmed (control) cassava (after 108 minutes). However, if the osmotic dehydration time was included in the whole process, a 43% increase in the total drying time will be added when applying the process of osmotic dehydration before air drying of chipped cassava.

The effect of osmotic dehydration on drying rates of air dried chipped cassava at 70°C drying temperature is shown in Figure 6.9.

![Figure 6.9. Effect of osmotic dehydration on drying rates of cassava at 70°C drying temperature (error bars±0.46)](image)

The drying rates of the osmosed (NaCl or sucrose) cassava was comparable to the control in spite of the lower initial moisture content of osmosed cassava samples due to the osmotic dehydration. Probably, this could be due to the higher resistance (compared to unmosmed samples) to water removal by the solutes (NaCl or sucrose) gained during osmotic dehydration and agrees with other results found for other food products (Singh and Gupta, 2007; Moreira et al. 2006; Moreira et al. 2011). Interestingly, cassava samples osmosed with sucrose solutions had comparable drying rates to cassava osmosed in NaCl solution.
The drying time comparison between osmosed (NaCl or sucrose) and unosmosed samples was presented in Figure 6.10. Due to reduced moisture content (0.493 and 0.487 (wet basis) for NaCl and sucrose, respectively) of the osmosed samples, the time to reach a pseudo-equilibrium (acceptable final moisture content 0.12, wet basis) of the osmosed cassava (control) was only 108 minutes (1.8 hours) while it was 162 minutes (2.7 hours) for unosmosed cassava (0.13, wet basis) samples.

![Figure 6.10. Drying time comparison of osmosed and unosmosed cassava at 70°C temperature](image)

The osmosed samples reached a pseudo-equilibrium moisture content earlier compared to unosmosed (control) cassava because the osmosed samples started at lower (0.487, wet basis) while the unosmosed products’ moisture content was 0.55 (wet basis).

The results of the experiment allow to conclude that the application of osmotic dehydration applying 25% (w/w) NaCl or 60% (w/w) sucrose solutions did not have significant increased on the drying rates, however because of the reduced moisture content and energy saved applying osmotic dehydration the
drying time to reach an equilibrium moisture content of fresh chipped cassava was reduced. This conclusion is in agreement with the general observations of previous works (Lenart 1996; Lewicki et al. 2002) applying osmotic dehydration before air drying of mostly fruits and vegetables.

6.3.3. Quality of osmo-dehydrated cassava

The analysis of quality of the dried cassava chips was done only for those samples dried with 70°C drying temperature since 70°C is concluded to be the optimal drying temperature. Gelatinization of cassava chips during air drying was the adverse effect of using high (e.g. greater than 80°C) temperature during belt-conveyor drying (Pontawe et al. 2012). Gelatinization is the yellowing of the inside cell membrane of cassava when it is exposed to high drying temperature. This is not acceptable at the processors’ level, since gelatinized dried cassava is hard to process due to its brittleness and easily breaks during the process of animal feed production (SMC, 2011).
6.3.3.1. Physical changes

Figure 6.11 shows the cassava chips dried in the laboratory dryer test rig using 70°C drying temperature after being osmosed with 25% (w/w) NaCl.

![Figure 6.11. Physical comparison of osmosed (25% w/w NaCl) and unosmosed cassava after drying to belt dryer test rig at 70°C drying temperature](image)

For the osmosed, air dried samples there were no significant changes on the surface structures and colour as compared to unosmosed air dried cassava samples. This result was in agreement with the study of Andres et al. (2004) who worked with the same product.

In the case of cassava osmosed with 60% (w/w) sucrose, the outer part was darker (e.g. dark-brown) as compared to unosmosed cassava samples (Figure 6.12) after drying in the laboratory dryer test rig. This can be due to the caramelization effect of the sucrose on the surface of the samples. Caramelization is the browning of the retained sugar on the surface of the samples due to hot air exposure during drying. Nevertheless, it is important to
highlight that either sucrose (60% w/w) or NaCl (25% w/w) osmosed cassava samples showed no gelatinization after air drying at 70°C.

Figure 6.12. Physical comparison of osmosed (60% w/w sucrose) and unosmosed cassava after drying to belt dryer test rig at 70°C drying temperature

6.3.3.2. Hardness results

Figure 6.13 displays the results of the hardness test of the osmosed, air dried products. A slight increase in hardness values was recorded for samples osmosed with 60% (w/w) sucrose compared to the NaCl (25% w/w) and unosmosed (control) product.

Figure 6.13. Variations of hardness for osmosed (60% sucrose or 25% (w/w) NaCl) and unosmosed cassava after air drying
This can be explained by the fact that during air drying, movement of water from the interior of the product osmosed with sucrose to its surface causes migration of solutes and their concentration in outer layers increases, so that they become rigid and often acquire considerable mechanical strength. This observation on the brittleness of osmosed product using sucrose solution (60% w/w) was consistent with the results on the New Zealand chestnut study (Section 4.3.1.2). This phenomenon of crust formation and case hardening which caused brittleness is common with foods that contain dissolved sugars in high concentration (Sosa et al. 2012).

**6.4. Summary**

The effect of osmotic dehydration before air drying to the laboratory dryer test rig (simulated belt conveyor dryer) did not improved the drying rates of chipped cassava osmosed with 25% (w/w) NaCl or 60% (w/w) sucrose solutions. However, in addition to the energy savings from reducing the moisture content of cassava applying osmotic dehydration before air drying, the drying time was faster in reaching the equilibrium moisture content of osmo-dried cassava than the unosmosed (control) samples because they started with lower moisture content. Nevertheless, if the osmotic dehydration time was included in the whole process, a 43% increase in drying time will be added when applying osmotic dehydration before air drying.

The effect of increasing the drying temperature (e.g. 70 to 80°C) on the osmosed product during the laboratory dryer test rig experiments showed no significant increase in terms of the drying rates. Also, the two osmotic solutions
(NaCl or sucrose) at different concentrations and molecular weights showed no significant differences on the drying rates of osmo-dried chipped cassava.

Cassava chips osmosed with 60% (w/w) sucrose was darker (compared to unosmosed cassava) and samples osmosed with 25% (w/w) NaCl showed no colour changed compared to unosmosed cassava samples after air drying at 70°C temperature. Osmo-dried (NaCl or sucrose) samples showed no gelatinization using 70°C air drying temperature. The hardness tests showed a slight increased on the hardness of samples osmosed with 60% (w/w) sucrose than 25% (w/w) NaCl or unosmosed cassava chips after air drying experiments.


7 CONCLUSIONS AND RECOMMENDATIONS

7.1. Conclusions

7.1.1. New Zealand chestnut

From the results obtained on working with New Zealand chestnuts the following conclusions were drawn:

1. Osmotic dehydration using either 22% (w/w) NaCl or 60% (w/w) sucrose solutions can reduce the moisture content up to 25% after 8 hours for shelled and unshelled chestnuts in bulk conditions at 20.5°C.

2. The unshelled chestnuts had comparable osmotic dehydration rates to shelled chestnuts with either 22% (w/w) NaCl or 60% (w/w) sucrose solutions. Thus, the shell is not a significant mass transfer barrier during osmotic dehydration. However, unshelled (e.g. with shell intact) chestnuts recorded a higher solids gain compared to shelled chestnuts samples using NaCl (22% w/w) or sucrose (60% w/w).

3. The optimum osmotic dehydration time of using either 22% (w/w) NaCl or 60% (w/w) sucrose solutions was 6-8 hours for either shelled or unshelled chestnuts.

4. The drying rates of shelled and unshelled chestnuts during osmotic dehydration using NaCl (22% w/w) solution over sucrose (60% w/w) solution was higher than solids’ gain into the chestnut samples.

5. Osmotic dehydration using lower concentration for NaCl (<22% w/w) and sucrose (<60%) and other osmotic media like glucose (56.5% w/w) and
calcium chloride (50%) was not successful for bulk shelled and unshelled chestnuts.

6. Using sodium chloride (22% w/w), the recovery of wholenuts after mechanical shelling (i.e. shell removal) showed a statistically significant improvement from 28.5±0.04% (unosmosed) to around 45.3±0.04% (osmosed).

7. Using a different batch of chestnuts with sucrose (60% w/w) solution, an improvement of almost 16.5±.08% (osmosed) from the control (unosmosed) on wholenuts shelling recovery achieved.

8. Osmosed chestnuts with 22% (w/w) NaCl solution was slightly darker compared to the samples osmosed with 60% (w/w) sucrose and to the unosmosed (control) samples. Chestnuts osmosed with 60% (w/w) sucrose are more brittle compared to the samples osmosed with 22% (w/w) NaCl and to the unosmosed (control) samples.

7.1.2. Philippine cassava

The following conclusions were generated from the results obtained in experimenting cassava chips in the Philippines:

1. Generally, increase in osmotic concentration and time also increased the $W_L$ and $S_G$ of chipped cassava osmosed with NaCl and sucrose solutions. The $W_L$ rates for both 25% (w/w) NaCl and 60% (w/w) sucrose solutions have greater rates compared to the $S_G$ values.

2. The normalised moisture content of cassava osmosed with 22 or 25% (w/w) was reduced to 80 and 75%, respectively, which were higher results compared to samples osmosed with 60% (w/w) sucrose with 83%.
The study found that the 25% (w/w) NaCl solutions (up to 210 minutes immersion time) produced slightly greater $W_L$ rates compared to the 22% (w/w) NaCl solution, without significant difference between the $S_G$ values.

3. Increasing the drying temperature (from 70 – 80°C) of laboratory drying test rig (simulation of belt-conveyor dryer) for cassava chips had no benefits in terms of drying rate after osmotic dehydration with 25% (w/w) NaCl and 60% (w/w) sucrose solutions. Hence, 70°C drying temperature is the optimum considering the energy savings.

4. The drying rates of the osmosed (NaCl or sucrose) cassava were comparable to the drying rates of the unosmosed control.

5. Cassava chips osmosed with 60% (w/w) sucrose were darker (compared to unosmosed cassava) and samples osmosed with 25% (w/w) NaCl showed no colour change compared to unosmosed cassava samples after air drying at 70°C temperature.

6. Osmosed, air-dried (NaCl or sucrose) samples showed no gelatinization using 70°C air drying temperature. Samples osmosed with 60% (w/w) sucrose showed a slight increased on the hardness than 25% (w/w) NaCl or unosmosed cassava chips after air drying experiments.
Chapter 7: Conclusions and Recommendations

7.2. Recommendations

7.2.1. New Zealand chestnut

The application of osmotic dehydration for a large-scale before mechanical shelling of unshelled New Zealand chestnuts variety is technically feasible employing sodium chloride (22% w/w) or sucrose (60% w/w) as osmotic solutions at 20.5°C.

The study did not examine the final taste of the osmosed product. It would be good information for the targeted consumers of the osmotic dehydration technology to undertake taste experiments after osmotic dehydration following mechanical shelling experiments.

The penetration of the osmotic media into the cell tissue of food materials can be predicted more and fitted into different models if the movement of the solutes overtime could be seen and analyzed. Hence, the study using Scanning Electron Microscopy (SEM) for osmotically dehydrated food materials may prove valuable.

Another recommendation from this study is to examine the storage life of New Zealand chestnuts having undergone osmotic dehydration using NaCl, sucrose and glucose osmotic solutions under different storage conditions. A benefit of this study would be for farmers who are waiting for all chestnuts to be harvested for bulk selling, since chestnuts are harvested within 2 months period.

7.2.2. Philippine cassava

The data generated from this study has potential for use in a large-scale application of osmotic dehydration of cassava chips before belt-conveyor drying.
However, further optimization of osmotic dehydration considering different solution concentrations, drying temperature and thickness of the samples loaded into belt dryer should be done for a more efficient application of osmotic dehydration before air drying of fresh cassava for animal feed production.

The penetration of the osmotic media into the cell tissue of food materials can be more predicted and fitted into different models if the movement of the solutes overtime could be seen and analyzed. Hence, the study using Scanning Electron Microscopy (SEM) for osmotically dehydrated food materials may prove beneficial.
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Appendix Figure A. 1. Solution temperature and relative humidity for New Zealand chestnut study

Appendix Figure A. 2. Moisture content (wet basis) comparison between 48 and 72 hours of oven drying experiment