IMPACTS OF STORAGE, SEASON, DURATION AND STEAMING ON PHYSICAL PROPERTIES AND EXTRACTIVE CONTENT OF ASPEN CHIPS

by

Kevin G. Shaw ©

A Graduate Thesis Submitted in Partial
Fulfilment of the Requirements for the Degree of
Master of Science in Forestry

April 12, 1995

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ISBN 0-612-09239-9

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ABSTRACT

Shaw, K.G. 1995. Impacts of storage, season, duration and steaming on physical properties and extractive content of aspen chips. M.Sc. Forestry Thesis, Lakehead University, Thunder Bay. 106 p.

Key Words: aspen, extractives, kraft, losses, moisture content, seasoning, wood chips

An investigation was made into the rates of moisture content, extractives and basic wood density losses that occur when trembling aspen (*Populus tremuloides* Michx.) wood chips are stored during different seasons of the year, in a chip pile. Seasoning is required for the pulping of trembling aspen in order to reduce the extractive content to a level that will minimize the occurrences of pitch build-up and problems in the kraft pulping process. The hypothesis for this study was that there are significant differences in losses of extractives, moisture content and basic density within a chip pile and that the extractives losses in the pile centre would match extractives losses in roundwood in a much shorter time. The change of extractives can be achieved in normal outside chip storage periods by using chips from the interior of a chip pile or in shorter periods through steaming chips under controlled conditions. Time was a very significant factor during the storage. The study found that the closer to the middle of the pile, the hotter the temperatures became. The largest rate of heat build-up was found in the first 12 days. The middle of the pile showed the lowest average moisture content after 4 months (i.e., 38.2%), whereas the top of the pile showed the highest moisture content after 4 months (i.e., 45.2%). The absolute rate of extractives loss was, on average, 0.3%/month for the 4 month study period. However, for the first month, the absolute rate was 1.0%/month (i.e., 31% of the original) and for the second month it dropped down to 0.6%/month (i.e., 39% of the original). There were no significant changes in the extractive content from month 2 to month 4. The bottom middle of the chip pile resulted in the largest loss of extractives (i.e., final extractive content of 1.8% after 4 months). No comparison was made between the aspen chip pile built in the summer with the one built in the winter since the winter pile remained frozen for the duration of the study. It was calculated that the extractive content after 1.5 weeks in the summer pile would equal the extractive content (i.e., 2.5%) of the aged roundwood (i.e., for 1 year). A minimum of emphasis should be placed on this since the sample size used to calculate the extractive content of the aged roundwood was small. The rate of basic density loss in the summer chip pile was 2.4%/month for the 4 month study period. The average moisture content of the fresh arriving chips was 47.9% as compared with the moisture content of the digester chips (i.e., 40.2%). The extractive content of the fresh arriving chips was 3.6%, as compared with 2.9% for the digester chips. There were no significant differences between the average basic densities for the fresh arriving chips and the digester chips (i.e., 0.41 g/cm^3). The study showed that steaming does not accelerate the loss of extractives in aspen chips. A method of chip reclaim is presented that collects, with the aid of an auger travelling underneath the chip pile, only the bottom middle chips. As these chips are collected, chips from the other regions of the pile fill in the gaps. A continuous chip seasoning period of 2 weeks is recommended to allow for the maximum amount of seasoning in the shortest time period. A chip pile inventory of 42 308 m^3 would be sufficient to allow for a 2 week inventory, however, a chip inventory of 3 weeks is recommended (i.e., 63 462 m^3 to allow for an extra weeks buffer). It is also recommended that Avenor consider continuous monitoring of the digester extractive contents to know that they are sufficiently low, as well as to try and correlate any operating problems in the mill with the extractive content of the chips that had entered.

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ACKNOWLEDGEMENT

I would like to thank all the people who helped me coordinate this study and keep it running smoothly. Without their help, this work could not have been completed. Special thanks to the University Research Incentive Fund (U.R.I.F.) and Avenor for their generous monetary contributions to this project. Many thanks to everyone at Avenor, specifically Gary Bloomfield, Ted Broadhurst, Steve Jones, Ken Sherrard and all the mill staff. You were all a great help. Special thanks to Larry Allen and Bruce Sitholé at PAPRICAN for your much needed expertise on this topic as well as the use of your labs for some of the analysis work. Thanks to everyone at Lakehead University, specifically Pat Cybulski and Steve Elliott from the Faculty of Forestry, Ted Garver and Peter Levis from the Dept. of Chem. Eng., and Walter Trutenko from the physical plant. Thanks to Tom Hazenberg for dealing with my statistical questions and to Allan Mackenzie at the Instrumentation Lab for analyzing some of my resin samples. Most importantly, I would like to thank my graduate committee members (K-C Yang and Allan Gilbert) for giving me direction during this study and helping me to answer some of the many questions that arose as a result of the work. Last but not least, I would like to thank my primary reader and advisor, Reino Pulkki for keeping me motivated throughout these long, last two years. I had lots of questions for you Reino, and you always seemed to get the answers when I needed them. Thanks a lot.

1 INTRODUCTION

Trembling aspen (*Populus tremuloides Michx.)* is one of the most abundant hardwood tree species in Canada, comprising more than 57% of the total hardwood standing volume and the third highest standing volume of all the tree species in Canada (3,199 million m^3)(Forestry Canada 1990). Until recently it was not used to any great extent as a source for pulp material. However, industrial use of aspen has grown substantially and it can now be considered as an important source of raw material. This can be illustrated by the 200% increase in the volume of aspen utilized between 1968 and 1985 in Quebec (Koran 1989). In the Thunder Bay region, the volume of aspen produced by field chippers rose from 140,000 m^3 in 1993 to 260,000 m^3 in 1994. It is expected that this volume will be raised to 400,000 m^3 in 1995. The annual harvest of aspen by Avenor (Thunder Bay region) also rose from 590,000 m^3 in 1990 to 950,000 m^3 in 1993.

1.1 STUDY PROBLEM

It is well known that utilizing fresh aspen roundwood in a kraft pulp mill can cause serious pitch problems (Lange 1958, Minard 1960, Affleck and Ryan 1969, Branch 1971, Allen and Lapointe 1987, Allen 1988, Ball and Forster 1990, Allen *et al.* 1991, Allen and Kowalski 1992), leading to considerable monetary losses. Allen (1988) states that it is not uncommon to lose up to $50,000/day (1988 Canadian funds) due to lost production. The production of substandard pulp can in turn lead to a loss in customer confidence. An 800 t/day fully bleached kraft mill using softwoods can spend or lose up to $1 million/yr as a result of pitch problems (Allen 1988). This figure can probably be doubled for hardwoods (Allen 1988). Components of the cost taken into consideration here are the increased use of additives and the sale of off-grade pulp which would probably have a selling price discounted by about 40%. Pitch problems arise when the wood extractives are not properly removed from the system during the pulping process. Ball and Forster (1990) also show how correcting pitch problems can lead to a cost savings of $515,000/year (1990 Canadian funds).

The Avenor mill in Thunder Bay region uses in excess of 1.1 million m^3 of aspen annually to produce bleached aspen kraft pulp. Previously, Avenor air seasoned its aspen in roundwood form for about 1 year in order to get good debarking quality and to reduce the pitch problems in the mill. With the recent shift to in-woods chipping, fresh chips are now delivered to the mill. Since Avenor is planning to have all aspen delivered as fresh chips, more information is required on the seasoning of the aspen chips to minimize the pitch problems.

The purpose of this research was to study the rate of extractives loss during chip storage of aspen to determine the best chip storage period and the conditions required. This study also examined the development of temperatures in the chip piles, chip moisture content and chip decay. The study analyzed the rate of extractives loss at various locations of a chip pile and under controlled conditions in the laboratory. These data were related to actual chip extractive contents and mill pulping results.

The hypothesis for this study was that there are significant differences in losses of extractives, moisture content and basic density within a chip pile and that the extractives losses in the pile centre would match extractives losses in roundwood in a much shorter time. The change of extractives can be achieved in normal outside chip storage periods by using chips from the interior of a chip pile or in shorter periods through steaming chips under controlled conditions.

1.2 STUDY OBJECTIVES

The objectives of this research were :

1. To determine the rate of moisture content, extractives and basic density loss in aspen chips located at different locations in a chip pile (i.e. under different temperatures and pressures) ;
2. To determine the rate of extractives loss under controlled steaming of chips in a vessel ;
3. To study the temperature development in a chip pile built in the summer and in the winter ;
4. To determine if seasoning differs between a chip pile built in the summer and in the winter ;
5. To obtain data on the quality of the fresh wood chips arriving at the mill and of the chips entering the mill process ; and
6. To correlate operating problems in kraft pulping with the occurrence of extractives in aspen chips to allow determination of the length of storage or steaming required to achieve a sufficient extractives loss.

To achieve these objectives, the following experiments were conducted :

1. A comparison between a chip pile built in the summer with one built in the winter;
2. A small chip pile to study the effect of time and maximum aeration exclusively (i.e., no compaction, heating) ;

3. Aged roundwood sampling (~ 1 yr) for extractive content and moisture content to illustrate the effect of wood handling that Avenor has used in the past (i.e., roundwood only) ;

4. Sampling of newly arriving fresh chips on a daily basis to determine the quality of the fresh wood arriving at the mill ;

5. Sampling of chips entering the digester to help illustrate the method of wood handling that Avenor is currently using (i.e., a 50:50 mixture of fresh and aged chips, and aged roundwood) ;

6. Temperature sampling over time at nine different locations in piles built in the summer and winter to determine pile temperature build-up and temperature isotherms within the piles ; and

7. A comparison of chips that have been steamed over time to determine whether steaming can shorten seasoning times.

2 LITERATURE REVIEW

2.1 HARDWOOD CHARACTERISTICS (STRUCTURE)

To obtain a better understanding of the wood resins and the part which they play in the pitch problems of kraft mills, one has to look closely at the cell structure of wood. In general, hardwoods have a more complex system of cells than softwoods. There are three main cell types that are of importance in hardwood pulping (Parham 1983). These are vessel elements, fibres and parenchyma.

The parenchyma cells (ray cells) are the most important of the three cell types from a pitch deposition point of view. The parenchyma cells serve as storage tissue for the tree. Elements that are stored in the ray cells are extractives, oils, resins, latex, tannins, starches and other raw materials (Parham 1983). The ray cells are usually responsible for the high number of fines that are produced when the wood is pulped.

Some general characteristics and properties found in aspen are listed in Table 2.1.

2.2 KRAFT PULPING

Paavila (1960) stated that kraft pulp is any pulp that has been produced by an alkaline cooking liquor in which hydroxide and sulphide anions are the effective constituents. Jimenez *et al.* (1989) talked about the differences between kraft pulps and sulphite pulps.

Table 2.1 Some general properties and qualities of aspen in regard to kraft pulping (Mitchell 1957, Thomas 1987).

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1. The tension wood of aspen gives pulps of lowered strength.
2. The tension wood in aspen increases the pulp yield.
3. Fast growing aspens compare with spruce as raw material.
4. The wood from different regions of the same tree give different yields (decreases from butt to crown).
5. The chemical requirement for aspen pulping is lower than softwoods.
6. The pulp yield of aspen is high when compared to softwoods.
7. Aspen bleaches quite readily.

The objective of kraft pulping is to dissolve the lignin from the middle lamella and cell walls of the fibres so that the fibres can be separated and still retain their beneficial physical properties (Stone and Green 1958). The criteria, according to MacLeod (1987) for kraft pulps are: 1) good physical strength, 2) high brightness, and 3) low dirt content. In the kraft pulping process, the penetration of the liquor into the wood chip while being cooked in the digester is very important. Uniform liquor penetration is important so that all chips can be cooked to the same degree, thus minimizing the amount of over-cooked and under-cooked chips which will result in rejects. Complete penetration, according to Gustafson *et al.* (1988) results in: 1) reduced cooking times, 2) increased pulp uniformity, and 3) elimination of undesirable lignin side reactions. Stone and Green (1958), on the other hand, showed that poor penetration and diffusion can be recognized by: 1) visual inspection (examination) of the chips, 2) the percentage of screening rejects, 3) the pulp quality, and 4) the chemical composition of the chip. Ross and Potter (1945) found that fresh aspen or aspen which had been soaked for several days in running water facilitated liquor penetration. Mitchell (1957) stated that if aspen is used as the sole species in the pulp, problems will arise because the aspen kraft is difficult to wash. He suggested adding a furnish of (20%-25%) softwood to the digester to eliminate this problem. Hunt (1981) found that aspen had a fast delignification rate (as compared to other hardwoods) while in the digester. Kubes (1984) showed how aspen had a higher activation energy of spent liquor when compared to other hardwoods. He attributed this higher activation energy to the presence of a large volume of extractives in the wood.

2.3 WOOD QUALITY

The literature available on the quality of the wood as it relates to the pulp and paper industry is quite extensive (e.g., Hatton *et al.* 1968, Hunt 1981, Lonnberg 1982, Chinn 1985, Horng 1986, Schmidt 1990). The quality of the raw material is of utmost importance because it has a direct effect on the finished product. Chinn (1985) stated that the greatest detrimental factor in wood quality is the variability of the wood supply. Since kraft mill cooking dose is directly dependent on the wood properties of wood chips entering the process (e.g., moisture content, temperature and dimension), a smoother flow results when the fluctuations in the wood properties are kept to a minimum. Pulkki (1990) reported that the uniformity of the moisture content of the chip in the kraft process is more important than the moisture content itself because of digester control problems. Table 2.2 presents some wood characteristics that affect pulp yields and quality.

Table 2.2 Characteristics and qualities affecting the pulping process (Pulkki 1990).

1. relative density
2. extent of decay
3. fibre morphology
4. moisture content
5. chip size distribution
6. branch and knot wood
7. inner and outer bark
8. sapwood and heartwood
9. mature and juvenile wood
10. chemical composition of the wood
11. springwood and summerwood
12. amount of sand and other impurities

2.4 CHIP CHARACTERISTICS

There have been many studies done to determine the optimal chip thickness that should be used in the kraft process. Hatton (1975) discussed the effective chip thickness versus regular chip thickness and how it plays an important role in the penetration of liquor. He describes effective chip thickness as a meaningful measure of the extent to which the wood chips have been fissured (i.e., developed cracks which are parallel to the grain). He goes on to say that effective chip thickness is always smaller than the regular chip thickness. Nieuwenhuizen *et al.* (1985), Bryce and Lowe (1987) and Horng (1986) also recognized chip thickness as being the most critical of all chip dimensions. Specially designed rollers have been developed (Bryce and Lowe 1987) to compress the wood chips before they enter the digester. It was found that the liquor penetration was improved due to a thinner, more uniform chip thickness. The literature provides a fairly good consensus as to the optimal chip sizes that should be used. It is well documented that chip sizes should be in the thickness range of 2 to 6 mm (Olson *et al.* 1980, Hunt 1981, Nieuwenhuizen *et al.* 1985, Christie 1986, Pulkki 1990). Researchers have found that when the chip's thickness exceeds 6 mm, there are substantially more shives, screenings and knotter rejects as a result of undercooking.

2.5 CHIP PILE CONSTRUCTION AND RECLAIM

The process of storing incoming wood in outside chip piles began in the late to mid 1940's in the western United States (Blackerby 1958, Hatton 1969). Springer *et al.* (1974) stated that the advantages of stockpiling chips as compared with the traditional storage of roundwood were: 1) cost savings through mechanical ease of handling chips instead of roundwood, 2) improvements in chip quality through the systematic, uniform flow to the digester, and 3) the possibility of less wood deterioration while in chip storage. Chip pile construction has progressed from piling with front-end loaders and dump trucks to specially designed chip handling systems using belt conveyors or pneumatic means. Chip reclaim, which varies from mill to mill, is done with bulldozers and front-end loaders, or with belt conveyors or pneumatic feeders using augers that have been placed either under the chip pile or on the surface. Blackerby (1958) gave the following factors that needed to be considered before building a chip pile: 1) the amount of degradation that the chips will encounter, 2) the area preparation costs, 3) the handling costs, and 4) the length of time that the chips can be feasibly stored. In addition, Hatton (1969) listed the following criteria for chip pile construction: 1) the pile design must enable the stored chips to be reclaimed in the order they had been piled (i.e., First In First Out [FIFO] policy), 2) all stored chips must have reasonable access to the reclaim system(s), and 3) the length of the pile is inconsequential provided that dating is possible. He added that the pile width and height should also be restricted to allow sufficient heat dissipation so that fires could be avoided. Fuller (1985) gave the following prescription for an economical chip pile: 1) maintain pile height below 15m (50ft), 2) restrict tractor spreading of fresh chips to a minimum, 3) mix species of different deterioration rates only as needed, especially fast deteriorating hardwoods and full tree chips, 4) store full tree chips, which contain bark, foliage and a high proportion of living parenchyma cells (ray cells), in piles less than 8m (25ft) high and for less than 2-4 weeks, 5) avoid mixing fine particles (sawdust, shavings, chip fines and pulp mill knotter rejects) in chip piles, particularly where layering can occur, and 6) monitor the pile temperatures routinely. When the chip pile is being reclaimed, Hatton (1969) recommended that the chips from the base of the pile should be completely removed before any other chips are piled. This reduces the possibility of the spread of decay from the older wood chips to the newer, fresher ones. This method of reclaim assumes that the chips will be stored for a long time and that the pile will be completely broken down.

There was general agreement (Close 1986, Zreloff 1986) that all chip piles should be managed under a FIFO type policy. A uniform turnover in the pile helped to avoid discolouration of chips and any unnecessary decay. The majority of the literature (Hatton 1969, Hatton 1985, Close 1986, Zreloff 1986) stated that belt conveyance should be chosen over pneumatic handling of the wood chips. The pneumatic systems have been known to increase the amount of fines and dust.

Extractives loss is very important in the kraft process. When wood chips are put into outdoor chip storage (OCS) facilities, their extractives are gradually broken down. The lower the extractive content of the chips, the less likely they are to be problematic when they are pulped. The changing of wood extractives during OCS was considerably more rapid than during roundwood storage, resulting in shorter storage times (Annergren *et al.* 1965). This is probably due to the fact that chips have more surface area exposed than logs do, and the heat that is produced is a result of more respiration and reaction.

The amount of extractives, according to Dunlop-Jones *et al.* (1989), may not be as important as their composition. Nugent *et al.* (1977) gave a possible reason why fresh wood gives the mill problems. He hypothesized that the living parenchyma cells, which store the extractives, are under a great deal of pressure. As the wood is aged, the pressures in these cells are gradually reduced to a point where they are less likely to rupture and cause problems while being pulped. Rogers *et al.* (1971) gave the following explanation as to the possible mechanisms of the resin breakdown. He attributes this loss to the total metabolism of the fatty acids which yield volatile products, as well as, heat. The fatty acid content of the wood actually increases at first, but then gradually decreases as it starts to be consumed by the ray parenchyma cells. After the wood is chipped, the ray parenchyma cells continue to respirate and in so doing, consume the fatty acids and produce heat, water and carbon dioxide. This explanation accounts for the rapid heat increase that piles go through when they are first built. The fatty acid content gradually depletes to a point where it can no longer support the ray parenchyma cells. At this point the temperature should start to decrease, however, subsequent actions by mesophyllic and thermophilic fungi take over and keep the temperature relatively constant. During OCS, the resin acids and the unsaponifiables are not affected, to any degree (Rydholm 1967, Foran 1984). These along with the fatty acids are the most troublesome materials during pulping (Rydholm 1967). Levitin (1967) found that in chip piles, the fatty acids were destroyed at a faster rate than the resin acids, whereas during roundwood storage, the resin acids were oxidized at a faster rate.

Springer *et al.* (1974) noted that there were two different zones in a chip pile; those being zones of compacted chips and zones of uncompacted chips. These zones are important in regard to deterioration since deterioration is much faster in the uncompacted zones (Springer *et al.* 1974, Foran 1984, Schmidt 1990). Lindgren and Eslyn (1961) gave a good comparison between chip deterioration and roundwood deterioration. Chalk (1968) and Assarsson (1969) stated that hardwoods are generally more susceptible to deterioration than softwoods in chip storages. During storage, the chips can be damaged by: 1) mechanical means (drying, dirt, handling), 2) biological effects (fungi, insects or bacteria), or 3) chemical effects like extractives reactions or tannin damage (Pulkki 1990). Table 2.3 shows the outcomes that are possible as a result of quality changes.

Table 2.3 Possible effects of chip quality changes during storage on pulping and pulp quality (Hajny 1966, Hajny *et al.* 1967, Close 1986).

1. losses in wood substance (i.e. density)
2. losses in pulp yield
3. losses in pulp brightness
4. increasing cooking requirements
5. corrosion problems in the chip reclaiming systems due to low pH of severely deteriorated chips
6. production of off-grade pulp
7. severe wood losses due to spontaneous combustion
8. increased chemical consumption in pulping

2.6 CHIP STORAGE TIMES

Most of the literature was in agreement to the lengths of time that hardwood chips should be stored for. Storage times of 1-3 months would be enough to break down the extractives in the wood to a point where minimal pulping problems would be experienced (Holekamp 1958, Erskine and Galganski 1967, Hajny *et al.* 1967, Giffin 1970, Feist *et al.* 1973, Nugent *et al.* 1977, Close 1986, Wong and Eng 1987). Hatton (1969) reported that the maximum storage time for chips should be 3-6 months. Storing chips for longer than 3 months would result in unnecessary loss of wood weight and pulp quality, as well as a longer than necessary monetary investment. For example a chip supply of 30 days inventory in a 1,000 BTPD kraft mill operating at a 45% yield and cost of $100.00/bone dry ton of wood chips represents an investment of over $6.6 million (Paavila 1960). Hajny *et al.* (1967) reported that a loss of 50% of the extractives occurred in a 6 month period and represented the biggest loss of any of the wood constituents, whereas, Assarsson *et al.* (1970) reported that 25-75% of the extractives were eliminated after only a few months of storage. On the other hand, log storage for at least 1 year would be required to get sufficient extractives breakdown (Scafer 1947, Levitin 1967, Giffin 1970, Wong and Eng 1987). Studies have shown that deterioration in OCS during the winter months in northern areas is much slower than during the warmer months since the chip piles undergo much slower chemical reactions. Wong and Eng (1987) and Erskine and Galganski (1967) reported that it would take twice as long (i.e., 6 months) to get the same amount of seasoning as it would during the summer, while Nugent *et al.* (1977) stated that very little change occurs in frozen wood. However, Allen *et al.* (1991) reported that seasoning still occurs at temperatures of -20°C.

2.7 TEMPERATURE BUILDUP (CHIP PILE)

When wood chips are piled, they almost immediately begin to accumulate heat. This heat build up is one of the factors which accounts for the faster seasoning of chips as compared with roundwood. Fuller (1985) gave a good explanation as to how the pile heats up. He explains that during the first week, the heat build up in the pile is almost entirely as a result of the living cells respirating, e.g., ray parenchyma. During the next 1-4 weeks, all events of heat build up are solely dependent on the amount of air circulation in the pile. If the pile is well compacted, then less air is allowed to flow causing temperatures to rise much more than they would in an uncompacted pile (Assarsson 1969, Assarsson *et al.* 1970, Bergman 1972). Once a temperature between 60°- 70°C is reached, a chemical reaction occurs which results in the production of acetic acid and heat. The higher the temperature in the pile, the larger the quantity of acetic acid is produced. During periods of 1 month and beyond, large amounts of deterioration can occur if the pile temperature remains at a high level. Although the acetic acid is not strong, large quantities of it can deteriorate the wood by attacking and damaging the cellulose molecules. This in turn reduces the pulp yield and strength. The increase in heat and acidity also darkens the wood. Fuller (1985) further explained that the mechanisms that can lead to temperature buildups in the range of 82°- 93°C are assumed to be exothermic chemical autoxidation reactions of the cellulose at a low pH. If the heat is not dissipated at this point, ignition may occur.

The fastest buildup of heat generally occurs in the first two weeks (Erskine and Galganski 1967, Chalk 1968, Springer *et al.* 1978). The movement of heat and air through the pile has been well documented. The air flow takes the form of a convection current (Bjorkman and Haeger 1963, Hajny 1966, Erskine and Galganski 1967, Hajny *et al.* 1967, Assarsson 1969, Assarsson *et al.* 1970, Springer *et al.* 1974). The heat buildup during the early stages of the pile development causes an upward convection flow. Oxygen (air) enters into the lower portion of the pile, is heated, rises and then moisture starts to condense as it nears the upper cooler portion of the pile. The result of this is chips with a higher moisture and acidic content in the upper portions and chips with a lower moisture and acidic content near the middle of the pile.

Temperature development in the pile depends on : 1) environmental temperature (geographical location, season and weather), 2) size and compaction of pile, and 3) fines and bark content of the chips (Bergman 1972). Spontaneous heating in a chip pile is caused by : 1) the respiration of living parenchyma cells in the sapwood, 2) metabolism of aerobic bacteria in the wood, 3) direct chemical reactions of wood constituents, and 4) respiration by fungi (Close 1986). The temperature in the centre of the pile can be raised by increasing the size of the pile while keeping its ratio of external surface area to volume at a low value (Springer and Zoch 1970) or through increasing the amount of bark and needles present (i.e., full tree chips) (Springer *et al.* 1978). Feist *et al.* (1973) noticed that heat alone, at temperatures higher than 55°C had little effect on the breakdown of extractives in aspen.

According to Close (1986), the deterioration in a chip pile can be broken down to: 1) weight loss or density loss, 2) staining, 3) extractives losses, and 4) increase in fines content. Researchers (e.g., Assarsson 1969, Assarsson *et al.* 1970) agreed that the outer portion of the chip pile forms a shell and acts as an insulator for the pile. Temperature is the most important factor for the presence of different types of microflora in different areas of the pile. Springer *et al.* (1978) found that chips treated with the chemicals formaldehyde and P-Nitrophenol kept the pile temperatures much lower than in piles of untreated chips. Chip piles that are built in the winter with frozen chips, will not be able to begin the heating process until the ambient air temperature has warmed up above 0°C (Erskine and Galganski 1967, Rydholm 1967, Assarsson 1969, Bergman 1972, Sampson and McBeath 1987). Rydholm (1967) suggested that the chips have to be awakened by steam before being blown into a pile. Bergman (1972) also noted that the outer shell temperatures of the chip pile followed the ambient air temperature. Feist *et al.* (1973) and Close (1986) showed how pulp yield (aspen, douglas fir and loblolly pine) decreased dramatically when exposed to temperatures above 65°C for periods longer than 3 months.

2.8 DETERIORATION

Tall oil and turpentine are valuable by-products of the kraft pulping process. These by-products consist of a mixture of fatty acids and resin acids (Blair-Burch *et al.* 1947). A break-even point is required when ageing wood chips since both the extractives (loss good), as well as, the by-products (loss bad) are broken down over time. Both the tall oils and turpentines are quite volatile. Springer *et al.* (1974) stated that the loss of these by-products is faster in the OCS than it is in log storage. The rates of actual loss of these by-products are quite variable.

2.9 CHEMICAL ADDITIVES

There have been a number of studies undertaken to determine the effects of preservatives in chip piles. All the studies have come up with some positive results. Springer *et al.* (1971) found that treating aspen chips with a kraft green liquor mixture (i.e., sodium sulphide and sodium carbonate) was highly effective in preventing losses in wood substance and had no adverse effects on the resulting kraft pulp quality. Springer *et al.* (1974) also found that wood substance losses could be minimized through chemical treatment of the chips. The study also found that mill green liquor, as well as, sodium N-methyldithiocarbonate had reduced the wood substance losses by 1% after 2 months of storage.

2.10 MICROORGANISMS

The two main microorganisms that have a direct role in wood deterioration during OCS are the bacteria and the fungi. Both of these organisms are effected by pile temperature, as well as the moisture content of the chips. Assarsson (1969) stated that there are no biological reactions that take place at temperatures below freezing or temperatures above 60° C. At moisture contents above 55% (wet weight) and below 23% (wet weight), fungal growth is almost stopped (Crandall 1953, Djerf and Volkman 1969). When the temperatures and moisture contents are favourable, the fungi and the bacteria will start the deterioration process. Assarsson (1969) reported that bacteria are considered harmless when compared to fungi since they decay the wood very slowly.

The fungi attack the polysaccharides and lignin in the wood, thus lowering the wood density, as well as the overall pulp yield and quality. According to Sheridan (1958), there are two types of rot caused by fungi in the chip pile: 1) white rot, and 2) corrosive rot. The white rots are usually the least harmful since the fungi mainly attack the lignin and leave the cellulose unharmed. The corrosive rots, on the other hand, attack both the lignin and the cellulose. Glennie and Schwartz (1950) found that decayed aspen lowered the yield of pulp and also caused difficulties in the bleaching process.

2.11 EXTRACTIVES CHARACTERISTICS (PITCH)

Pitch, or wood resin, is used to denote any material in wood or wood pulps that is insoluble in water, but soluble in neutral organic solvents (Levitin 1970, Allen 1988). Wood resin forms the major portion of the extractives removed from wood by any organic solvent. The standard method for analyzing wood resins involves fractionating them into their component parts (Mutton 1962). The major constituents of resins are 1) free acids, 2) combined acids, and 3) unsaponifiables. Unsaponifiables are those components that do not form soluble soaps during the kraft pulping process and are therefore troublesome. The acid fractions can be broken down further into resin acids and fatty acids. Sitholé *et al.* (1992) stated that aspen wood resin consists of fatty acids, resin acids, waxes, alcohols, terpenes, sterols, steryl esters and glycerides. They group these resin components down into more practical classes (i.e., saponifiables or nonsaponifiables). The saponifiables include fatty acids, resin acids, some steryl esters and glycerides. The nonsaponifiables include some steryl esters, diterpene alcohols and aldehydes, sterols, triterpene alcohols and fatty alcohols. There are usually no (or very small traces) resin acids in hardwoods (including aspen). The fatty acids account for 62-85% of the total amount of extractives in hardwoods, while the unsaponifiables account for approximately 14%

(Mutton 1962, Levitin 1970). Dunlop-Jones *et al.* (1991) stated that the bulk of acetone extractives for aspen are neutrals which is normal since the majority of the fatty acids present are esters (e.g., triglycerides). They also found that as one moves up the tree, the proportion of neutrals in the heartwood increases, whereas the proportion of the total free acids decreases. Aspen tends to have a high proportion of waxes (e.g., steryl esters and esters of higher alcohols with fatty acids) present in its extractives which are hard to saponify (Dunlop-Jones *et al.* 1991). Dunlop-Jones *et al.* (1991) also found that after saponifying the neutrals in the laboratory, sterols and triterpene alcohols (i.e., nonsaponifiables) remained. Wood resins are also a valuable source of other by-products, namely tall oil and turpentine. Allen (1988) found that the acetone extractive content of a freshly felled aspen tree was 4.5%. Sitholé *et al* (1992) showed how the extractive content of aspen decreases when it is seasoned. They found that the acetone extractive content for aspen as a per cent of freeze-dried sample decreased from about 2% for fresh wood to 1.2% for wood that was aged for 1 year. Table 2.4 shows the diethyl ether extractive contents for many of the commonly used trees in North America. The table shows that aspen has the second highest extractive content of the hardwoods (Birch has the highest extractive content). Although some of the softwoods have comparably high extractive contents or even higher (i.e., Jack pine), they do not cause problems in the kraft pulping process. This is because their extractives are composed of a large percentage of saponifiables which will form soaps when pulped. No literature could be found comparing the acetone extractives of different species.

Table 2.4 Diethyl ether extractive contents for some commonly used trees (Mutton 1962).

Species	Extractive Content (% of oven-dried wood)
Aspen	1.0 - 2.7
Spruce	0.4 - 2.1
Balsam Fir	1.0 - 1.8
Douglas Fir	0.4 - 2.0
Eastern Hemlock	0.2 - 1.2
Western Hemlock	0.3 - 1.3
Jack Pine	1.3 - 4.3
Sugar Maple	0.2 - 0.9
Beech	0.3 - 0.9
White Birch	1.5 - 3.5

2.12 PITCH PROBLEMS

Pitch problems are a serious concern in the hardwood kraft pulping industry. Allen (1988) estimated the cost of these problems at about $1 million/mill/year. The components of these costs being: 1) sale of off-grade pulp contaminated with pitch dirt, 2) premature replacement of machine clothing, 3) time lost for cleanups, and 4) cost of additives to control the problem (Allen 1988). The reports on pitch contamination in the literature are numerous (Minard 1960, Levitin 1970, Branch 1971), and areas of problem occurrence are fairly consistent. Specific areas of pitch deposition are given in Table 2.5.

Table 2.5 Specific areas of pitch deposition (Affleck and Ryan 1969, Ball and Forster 1990).

1. centrifugal cleaners - pitch forms balls and clogs reject tip of cleaners
2. consistency regulators - build up on sensing equipment
3. probes for magnetic flowmeters
4. deposits at liquid levels on tanks and machine wire pits, chest agitators and repulper screws
5. brown white water chest
6. brownstock areas
7. unbleached screenroom - on walls of pipes, surface of vats, screen plates, brownstock decker apron and repulper screw
8. points of high shear
9. points where air/water interfaces exist
10. points where there are temperature and pH changes

The main problem occurs when pitch deposits break off and wind up as specks or streaks in the final product. During the process, (in some instances) defoamers are added to help in the removal of the resin particles in the liquid. The addition of too much defoamer, however, can also lead to problems since it too will start to co-deposit with the pitch (Allen 1988). Also, the purity of the cooking liquor is important since suspended materials (i.e., calcium carbonate or carbon) tend to co-deposit with the pitch (Allen and Kowalski 1992).

In order to avoid pitch problems during the kraft pulping process, specific measures must be taken (Table 2.6).

Table 2.6 Measures to counteract pitch problems (Allen 1988, Ball and Forster 1990).

---

1. season wood before pulping
2. use sufficiently large alkali charge to avoid zero residual effective alkali
3. improve white liquor clarification
4. ensure good debarking
5. improve efficiency of brownstock washing
6. control pH of unbleached screenroom water to 6.0-6.5
7. avoid high feed rates of defoamer
8. avoid the use of silica-in-oil or wax-in-oil defoamers in the bleach plant
9. improve screening and cleaning
10. maintain pH below 6.0 on pulp machine
11. eliminate unnecessary fresh water dilution from brown white water chest to maintain the lignin concentration > 0.20 g/L
12. skim the brown white water chest to remove the foam concentrated pitch materials from the process

---

Aspen resin has been shown to be troublesome when it is pulped. Allen (1988) gave the following reasons why pulping aspen usually leads to problems: 1) it is one of the most resinous species, 2) the wood sometimes contains substantial amounts of bark and woodrot (especially in the winter when debarking is more difficult), and 3) aspen has a low saponifiables to nonsaponifiables ratio (i.e., 1:2). The saponifiables produce soaps which aid in resin removal. A ratio of 3:1 or larger (saponifiables : nonsaponifiables) will generally result in fewer pitch problems when the wood is pulped (Allen 1988, Dunlop-Jones *et al.* 1991). Resin acids act as surfactants in the process but aspen lacks these substances (Dunlop-Jones *et al.* 1991). To improve the ability to completely solubilize the aspen saponifiables, resin acids can be added to the process in the form of tall oils. Because of the importance of pitch problems to the kraft pulp and paper industry, Allen and Kowalski (1992) have developed an expert system which can be used by mill personnel. The system is used as a troubleshooting tool which is able to deduce the source of mill problems once the user has answered a series of questions relating to the problem.

2.13 ADDITIVES (DISPERSANTS)

Additives, which are composed of resin acids, are especially important in hardwood pulping since they are lacking in hardwoods (Dunlop-Jones *et al.* 1991). Affleck and Ryan (1969) stated that dispersants are the quickest way to control pitch, however, they must be used with caution since they are expensive. Dunlop-Jones *et al.* (1989) felt that using the resin acid additives will not lead to a problem since they have been found in only trace amounts in many pitch analyses. A study was undertaken to see which additives were the most effective in resin removal. The research found that the additives, in order of effectiveness were Distilled Tall Oil > Canadian Tall Oil > Gum Rosin > Abietic Acid (Dunlop-Jones *et al.* 1989). The main difference between these tall oils was their acid number. The higher the acid number, the better the deresination (Dunlop-Jones *et al.* 1989). The higher acid number tall oils, however are more expensive.

2.14 CHIP STEAMING

Studies have found that pre-steaming wood chips before they enter the digester results in more uniform cooks and pulps of higher yields (Anon 1947, Ross and Potter

1945). Wood chips are pre-steamed: 1) to evacuate the air from the chip, 2) to cause greater compaction in the digester, 3) to give the chips a uniform temperature and moisture content, and 4) to increase digester capacity (Zreloff 1986). Nugent *et al.* (1977) presented an accelerated deresination system in which hot air is pumped through wood chips for periods of up to 72 hours at temperatures of 55°C. It is felt that this reduces the resin content of the chips to levels which would be reached in normal outside chip storage periods.

3 MATERIALS/METHODS

3.1 PILE CONSTRUCTION

To study the effect of season, two chip piles were constructed (summer [unfrozen] and winter [frozen]) using freshly felled, delimbed and debarked aspen (bark content was typical of mill run chips (i.e., 0.8%-0.9%)). The piles were 12 m long, 12 m wide and 6 m tall representing a volume of approximately 500 m^3. The chips were piled using an overhead conveyor and then formed using a front end loader and a crawler tractor. The aspen chips for the summer pile (i.e., from June to September) came from the Camp 329 (Avenor) chain flail delimber debarker chipper in Ignace, Ontario, while the chips for the winter pile (i.e., from January to May) came from the Dorion Fibre-Tech ring-debarker chip plant located in Dorion, Ontario.

3.2 CHIP SAMPLING

Since sampling for the wood chips over the storage period, with only minor disruptions to the pile was a priority, a method of sampling using a soil auger and plastic PVC pipes was utilized. A total of 9 pipes were put in the pile during construction so their ends were at the correct depth for sampling. The pipes were insulated using pipe insulation pieces attached together end-on-end by a nylon rope which travelled through the middle of each piece. The pieces were pushed inside of the PVC pipes and when required, could be removed from the pipe by pulling the rope. The outside exposed ends of the pipes were capped with PVC caps. Sampling depths of 6 m (middle), 2 x 4 m (midway) and 2 x 2 m (edge) measured from the edge of the pile, were used at the bottom of the pile, 2 x 2 m (edge) and 4 m (middle) midway up the pile and 2 m (middle) at the top of the pile. The bottom sampling positions were 1 m above the ground. The nine sampling positions are illustrated in Figure 3.1 (not to scale). Temperature thermocouples were placed at the ends of the pipes (i.e., sampling location) so that temperature readings in those areas could be taken. Chip samples were taken weekly for the first month and monthly thereafter for a total of 4 months. Five bags of chips were collected for analysis from each of the pipes at the start. However, this figure was lowered after statistical sample size tests showed that the frequency could be 2 bags per pipe. Table 3.1 shows the sampling dates for each of the two piles. After sampling, the bags were immediately placed in a freezer (-16.0°C) in sealed plastic bags.

3.3 TEMPERATURE SAMPLING

For the summer pile, temperature readings at the 9 locations were taken daily as it began to heat up. Once it had reached a fairly constant temperature (i.e., pile temperatures peaked), the frequency of sampling was reduced to once every week. For the pile built in the winter, temperature readings were also taken daily at the start (i.e., for 7 days). Since the pile showed no signs of warming up, temperature readings were taken weekly thereafter. All temperature readings were taken at 4:00 pm. Weather data from Environment Canada, spanning all pile sampling days were used to help in the explanation of each pile's temperature build-up.

Table 3.1 Chip sampling dates for both the summer pile and winter pile.

SUMMER Sample	Date
start	June 5, 1993
week 1	June 14, 1993
week 2	June 19, 1993
week 3	June 26, 1993
month 1	July 3, 1993
month 2	July 31, 1993
month 3	August 28, 1993
month 4	September 28, 1993
WINTER Sample	Date
start	January 23, 1994
week 1	January 30, 1994
week 2	February 6, 1994
week 3	February 13, 1994
month 1	February 20, 1994
month 2	March 20, 1994
month 3	April 17, 1994
month 4	May 17,1994

A small experiment was conducted to test the accuracy among the temperature sensors. The sensors were placed together on a table at room temperature and temperature readings were taken at 10 minute intervals for one hour. An analysis of variance was performed on the resulting data.

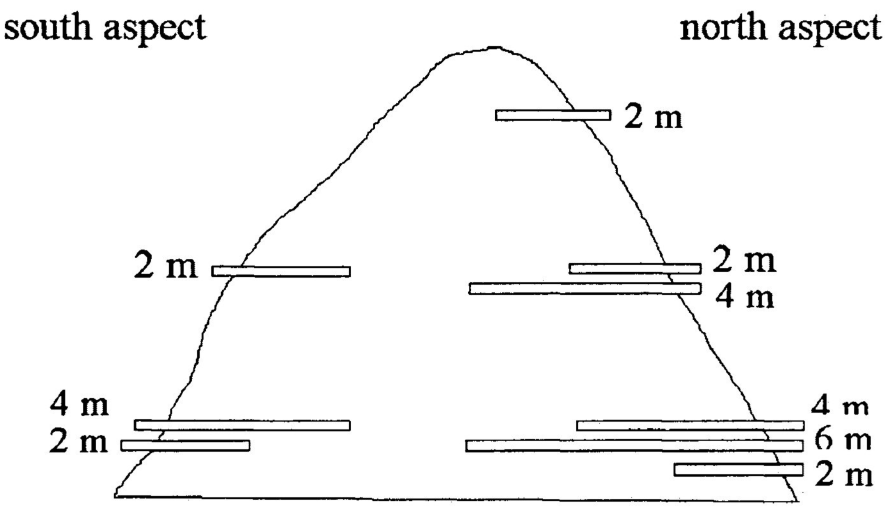

Figure 3.1 Diagram illustrating the sampling positions of the nine pipes.

3.4 LABORATORY

3.4.1 MOISTURE CONTENT

Wood chips were ground to a powder in a Wiley mill so that the moisture content variation between the samples would be reduced. This resulted in smaller sample sizes to be measured. At least 3 g of the powder were transferred to tared aluminum dishes and weighed. They were then dried in an oven at 100°C for 24 hours. The dried wood was then weighed again. The wet and dry weights of the wood were calculated by subtracting the weight of the empty dishes from the weights of the wood + dishes before and after drying in the oven. The moisture content based on green weight was calculated using the following formula :

$$\text{Moisture Content (\%)} = \frac{\text{green weight (g) - oven-dry weight (g)}}{\text{green weight (g)}} \times 100$$

3.4.2 EXTRACTIONS

3.4.2.1 FRESH CHIPS VERSUS AIR-DRIED CHIPS

The CPPA standard G.31P states that wood chips must be air-dried (~12% moisture content based on a dry weight basis) before they are extracted. An experiment was conducted to test the effects of moisture content on the total extractive content of the wood. A large sample of fresh wood chips were ground up using the Wiley mill and then dried down to a moisture content of approximately 12%. The sample was then subdivided into smaller samples. One sample of the dried wood was put aside for extraction and differing amounts of distilled water were added to the other samples to increase their moisture contents. A regression line was calculated on the resulting extractive contents to illustrate the relationship between moisture content and extractive content.

3.4.2.2 EXTRACTION PROCEDURE

The wood chips were prepared for Soxhlet extraction as per CPPA standard G.31P (with the exception that the wood chips were not air dried to a 12% moisture content). This involved grinding the fresh wood chips to a powder in a Wiley mill.

If the powder was not used immediately, it was put into an airtight bag and put in the freezer. At least 5 g of fresh wood powder was used in each thimble and 125 ml of acetone was used in each flask. The aspen resin was extracted from the ground up wood chips using CPPA standards G.13 and G.20. A minimum of 4 cycles through the condenser were required every fifteen minutes. The extractions ran for at least 7 hours in order to ensure that the maximum amount of resin possible was obtained. After completion of the extraction, the thimbles were removed from the condensers and the acetone was boiled from the flasks until approximately 20 ml of solution remained. The acetone that was collected as a distillate in the collection tube was transferred back into its original container for reuse. The remaining solution (resin and acetone) was carefully transferred from the flask to a tared evaporating dish and placed on a hot plate to remove the remaining volume of acetone. The dishes with the extract were then placed in an oven (100°C) and dried for one hour. The percentage of extractives in the wood was calculated as :

$$\text{Extractives (\%)} = \frac{A}{W} \times 100$$

Where A = oven-dry weight of the wood extractive (g)
W = oven-dry weight of the test specimen (g)

The extractives percentage is expressed to the nearest 0.1% based on the oven-dry weight of the wood. Sample size calculations were performed continuously at the start to determine the required sample sizes for each portion of the procedure (i.e., moisture content, extractive contents). The required sample sizes for both the moisture content and extractive content were 6.

The solvent used for the extractions was Acetone (pesticide grade). The extraction heating unit used for this procedure was a Lab-Line 5000 model which had 6 hot plates. The extracting apparatus was a KIMAX brand with Allihn condenser and the thimbles used were single thickness cellulose thimbles (33 mm x 80 mm). The thimbles were continuously reused and only replaced every few weeks. Figure 3.2 shows the extraction apparatus setup.

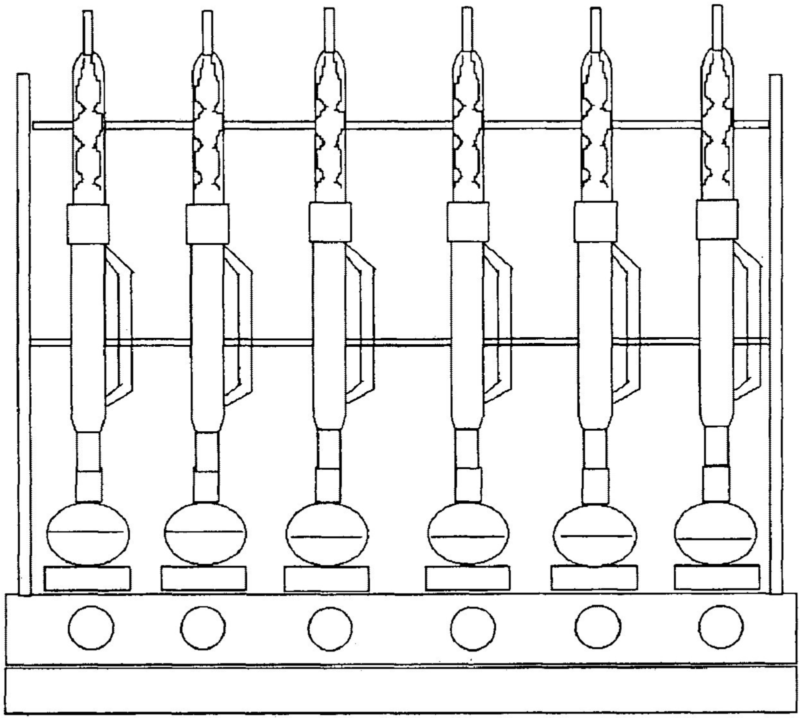

Figure 3.2 Diagram illustrating the setup of the extraction apparatus.

3.4.3 BASIC DENSITY

Ten individual chips, later reduced to 5, from each of the samples were used to determine the basic density of the sample. Sample size calculations showed that a sample of 5 wood chips (confidence level of 90%) was sufficient in determination of the basic density. Volume (green) of the chips was determined by the water immersion method. The chips were oven dried for 24 hours at a temperature of 100°C and then weighed. The basic density was calculated using the following formula:

$$\text{Basic density (g/ml)} = \frac{\text{oven dry weight (g)}}{\text{green volume (ml)*}}$$

* green volume at a moisture of 30% or higher

3.5 ROUNDWOOD

Ten randomly chosen poplar bolts that had been aged for 1 year, were taken and broken down into 5 samples. Each sample consisted of 2 bolts. These roundwood samples represent the method of wood handling and preparation that Avenor has used in the past (i.e., only roundwood, no chips). Each sample was analyzed for moisture content and extractive content in the lab. Basic densities were not taken with the roundwood because they were not going to be a part of the comparison. These results were used for comparison with the chip analysis results from this study.

3.6 DIGESTER AND FRESH ARRIVING CHIPS

One of the objectives of this study was to obtain moisture content, extractive content and basic density data on the quality of the fresh wood chips arriving at the mill and of the chips entering the mill process. Fresh chips arriving at the mill, as well as chips bagged just before entering the digester were taken for laboratory analysis. The fresh arriving chips were sampled for a period of 15 days and the digester chips were sampled for a period of 10 days. The digester chips consisted of a 50:50 mixture of aged roundwood chips (~1 year), and fresh and aged chips. Approximately 25% of the digester chip mixture had been aged for periods of 4-8 weeks and the other 25% was fresh from the field. The moisture contents, extractive contents and basic densities of the digester chips represent the current method of wood handling and preparation found at Avenor. The results were then compared to the past method of wood handling and preparation, as well as to the data from the two experimental piles (i.e., future method) so that conclusions as to the best method of handling and preparation could be made.

3.7 MAXIMUM EXPOSURE PILE

A small pile (conical shaped) consisting of 5 bags of fresh aspen chips (approximately 0.1 m^3) was used to help explain what happens to the wood chips when the effects of the sun, wind and compaction (i.e., heating) are eliminated. The important factor that this pile tests is time. The maximum exposure pile was spread out at room temperature (21 °C) for 25 days. Chip were sampled daily for the pile duration and moisture contents and extractive contents were measured. The resulting data illustrate how the moisture contents and extractive contents decrease over time.

3.8 STEAMING

Fresh wood chips were steamed (at temperatures of 100 °C) in a closed vessel under normal atmospheric pressure for periods up to 24 hours. Random samples were taken periodically throughout the process for further analysis in the laboratory. Wood chips were also left exposed at room temperature as a control (i.e., unsteamed chips). The control chip samples were randomly taken at the same interval as the steamed chips. The extractive contents of the steamed chips were compared to the extractive contents from the outdoor chip piles. No basic densities were taken since it was hypothesized that no significant amount of density loss would occur in the short time span of the steaming experiment.

3.9 ANALYSES

3.9.1 MODEL DEVELOPMENT

A model was developed to help explain the response variables moisture content, extractive content and basic density within the summer pile and to act as an aid in forecasting values. Four continuous variables were measured separately in the summer chip pile experiment. These variables were moisture content (MC), extractive content (Ext), basic density (Den) and pile temperature (Temp). For each variable a separate univariate analysis was carried out. Since each experiment had differing levels of replication, no multivariate analysis was performed. The factors for this experiment included time as the covariate, and aspect, height and depth of sample location. The model used to describe the experimental design of the summer chip pile is as follows :

$$Y_{ijkl} = \mu + A_i + H_j + D_k + ß_1 (X_1 - \bar{X1}) + ß_2 (X_2 - \bar{X2}) + \epsilon_{ijkl}$$

where : Y_{ijkl}= response variable : MC
Ext
Den
Temp

μ = the overall mean of the population when all factors and covariates are accounted for
A_i = aspect (north, south, centre)
H_j = height in pile (top, middle, bottom)
D_k = depth in pile (2 m, 4 m, 6 m)
ß = regression coefficients of the covariates time and time^2
X_1 = time
X_2 = time^2
ϵ_{ijkl} = random effect of the ith aspect with the jth height with the kth depth with the lth repetition
i =1,2,3
j =1,2,3
k =1,2,3
l =1,...,8 (for MC, Ext, and Den) and l = 1,...,39 (for Temp)

The interaction terms have been omitted from the model because they were of no practical importance and were not significant. The model used to explain the variables (steaming experiment), moisture content and extractive content, is as follows:

$$Y_{ij} = \mu + T_i + \epsilon_{ij}$$

where : Y_{ij} = response variable : MC
Ext

μ = the overall mean of the population when all factors and covariates are accounted for
T_i = time (0,3,6 hours)
ϵ_{ij} = the random effect of the i^{th} time with the j^{th} repetition

$i = 1,2,3$
$j = 1,\ldots,60$

All calculations and analyses were carried out with MINITAB Release 9.1. The general linear model procedure was used by MINITAB since the data sets had different numbers of replications, as well as some missing values.

4 RESULTS

4.1 FRESH CHIPS VERSUS AIR-DRIED CHIPS

Figure 4.1 shows the data points and the regression line that compares the effects of differing moisture content levels on the total extractive contents of the aspen wood chips down to the air-dried moisture content of 12%. The regression equation calculated for the data points is as follows:

$$\text{Extractive content (\%)} = 0.011 \text{ moisture content} + 2.90$$

The r^2 for this regression equation is 0.953. Due to the large number of extractions necessary for this study and the time required to air-dry the wood chips before the extraction process (as specified in the CPPA G.31P standard), it was decided that fresh chips would be used and the resulting extractive contents transformed to a base line air-dried extractive content (i.e., MC = 12%). This method is valid since the degree of fit for this regression equation is high.

4.2 SUMMER CHIP PILE

4.2.1 MOISTURE CONTENT

Figure 4.2 shows the moisture contents over time for each of the sampling positions, as well as the average value for each pile location. All locations exhibit the same general loss trend until month 3. At this time, all the moisture contents

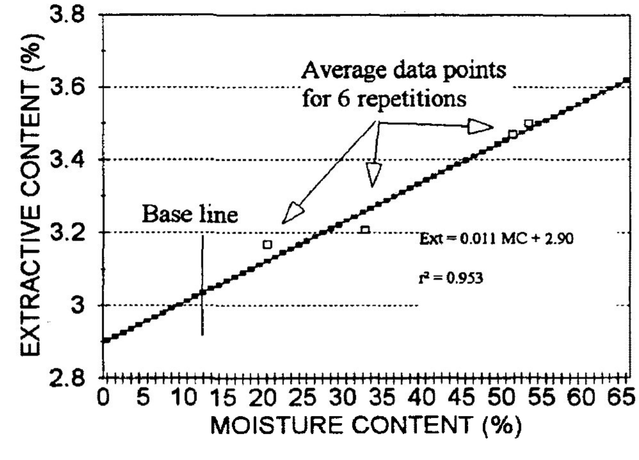

Figure 4.1 Regression line illustrating the relationship between moisture content (green weight) and extractive content, for aspen (*Populus tremuloides* Michx.).

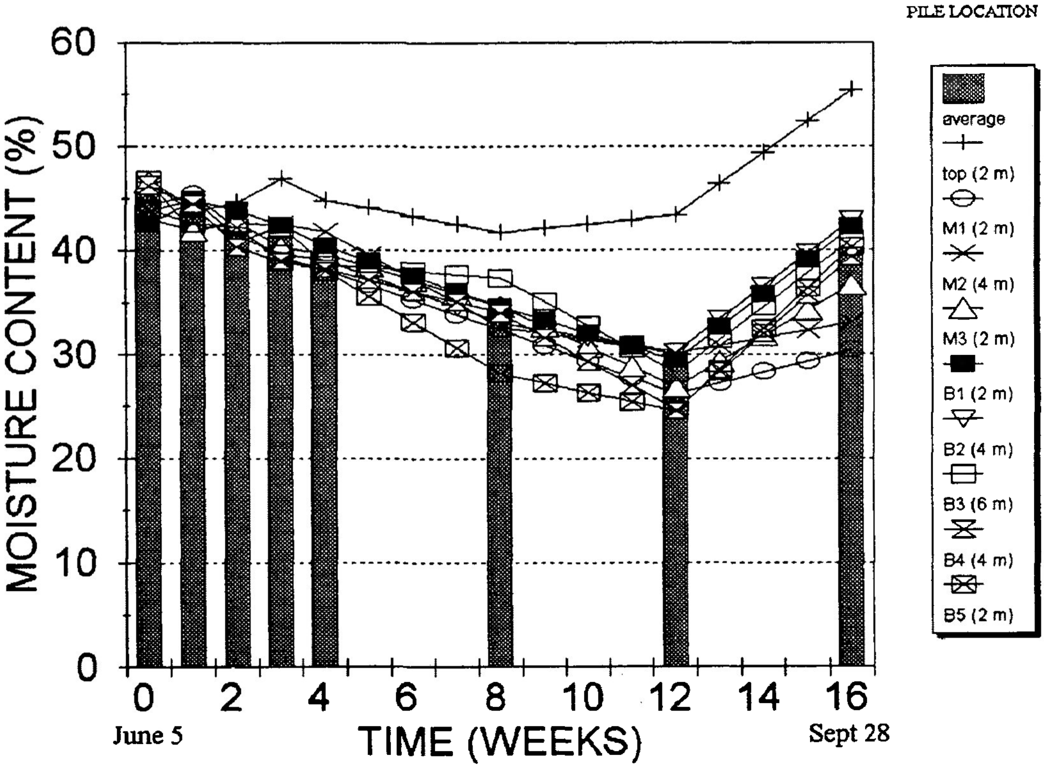

Figure 4.2 Average moisture contents based on green weight at the 9 sampling locations (summer pile) vs time.

increased. The top position shows the largest of the increases. The rise in moisture content after the third month (i.e., August) is probably due to a change in chemical reactions in the pile. Rogers *et al.* (1971) state that as the ray parenchyma cells continue to respirate, they consume the fatty acids and produce heat, water and carbon dioxide. Precipitation would not explain the increase in moisture content since the total precipitation decreased from 88.1 mm in month 3 to 45.0 mm in month 4 (Environment Canada 1993). Erskine and Galganski (1967) stated that moisture content losses due to evaporation were about equal to gains due to precipitation except during unusually rainy periods.

The average pile temperatures dropped slightly in the fourth month and the mean air temperatures decreased from 18.2°C in month 3 to 9.8°C in month 4 (Environment Canada 1993). This decrease in temperature could also be responsible for the increased moisture contents since there would be more condensation of evaporating water within the pile. The ANCOVA table for the moisture content data after 4 months storage is shown in Table 4.1.

Table 4.1 ANCOVA table for moisture content (summer pile).

SOURCE	DF	Seq SS	Adj SS	Adj MS	F	P(F)
time	1	14256.4	20571.7	20571.7	1534.99	0.000
time^2	1	13713.7	13686.8	13686.8	1021.26	0.000
aspect	2	2718.2	442.9	221.5	16.52	0.000
height	2	4329.1	3101.0	1550.5	115.69	0.000
depth	2	14.7	14.7	7.4	0.55	0.577
Error	1283	17194.6	17194.6	13.4		
Total	1291	52226.4				

Table 4.1 shows the covariates (time and time^2) and the factors aspect and height to be highly significant, and depth in the pile not significant at all. The adjusted moisture content means and their standard errors for each of the factors is shown in Table 4.2. These values are the overall averages for the experiment. They have been adjusted to eliminate any remaining effect of the covariate (i.e., time).

Table 4.2 Overall moisture content data adjusted means and standard errors for the 4 month storage period (summer pile).

aspect	Mean	Standard error
N	40.0	0.4668
S	41.3	0.4670
Centre	41.5	0.1768
height		
Top	45.2	0.6762
Middle	38.2	0.2877
Bottom	39.3	0.1439
depth		
2 m	41.0	0.1441
4 m	40.8	0.2880
6 m	41.1	0.6757

The middle of the pile showed the lowest moisture content after 4 months (i.e., 38.2%), whereas the top of the pile showed the highest moisture content (i.e., 45.2%). Multiple comparisons of the means were performed using Tukey's *w* procedure (Steel and Torrie 1980). This procedure was used because of the conservative results that it produces and because it only requires a single value for judging the significance of all differences. Tukey's critical value is calculated using the following formula :

$$w = q_{\alpha}(p\ ,\ f_e\ )\ s_y$$

Where w = Tukey's critical value
p = # of treatments
f_e = DF (error)
s_y = standard error

The critical value using the aspect means and a 99% confidence level is 0.74. Table 4.3 shows the comparison of the aspect mean differences with the critical value.

Table 4.3 Comparison of the aspect mean differences with Tukey's critical value (moisture content).

		N 40.0	S 41.3	Centre 41.5
Centre	41.5	1.53**	0.17ns	-
S	41.3	1.36**	-	-
N	40.0	-	-	-

Table 4.3 shows that the means for the southern aspect and the centre of the pile are not statistically different. The means for the northern aspect are, however, statistically lower than those of the southern aspect and the middle of the pile. Table 4.4 shows the comparison of the mean differences for height with Tukey's critical value.

Table 4.4 Comparison of the height mean differences with Tukey's critical value (moisture content).

		Bottom 38.2	Middle 39.3	Top 45.2
Top	45.2	6.97**	5.88**	-
Middle	39.3	1.09**	-	-
Bottom	38.2	-	-	-

Table 4.4 shows that the top, middle and bottom moisture content means for height are all significantly different from each other.

A regression analysis of the moisture content data yielded the following equation along with each of the standard errors of the coefficients in brackets:

$$\text{Moisture Content (\%)} = 47.4 - \underset{(0.08866)}{2.93}\ \text{time (week)} + \underset{(0.005471)}{0.148}\ \text{time}^2\ \text{(week)}$$

The r^2 for this regression equation is 0.536. The ANCOVA table for the moisture content equation (Table 4.5) shows that the regression equation is highly significant.

Table 4.5 ANCOVA table for moisture content regression analysis (summer pile).

SOURCE	DF	SS	MS	F	P(F)
Regression	2	27970	13985	743.16	0.000
Error	1289	24257	19		
Total	1291	52226			

Figure 4.3 shows the plotted regression line for the moisture content data. A minimum is reached on the regression line at approximately week 10 (i.e., 9.90 weeks). The regression moisture content decreases from 47.4% at week 0 to 32.9% at week 10. This is an absolute decrease of 14.5% (31% of the original moisture content).

4.2.2 EXTRACTIVES

Figure 4.4 shows the extractive content over time for each of the sampling locations, as well as the average value for each sampling period. All the sampling locations exhibit the same general trend (i.e., quick loss at the start then a levelling off); however, the top sampling location exhibited a steady rise in extractives after the fourth week instead of levelling off after 6 weeks (as was the case with the other locations). This rise in extractives at the top of the pile has been well documented for aspen and other species (Bjorkman and Haeger 1963, Hajny 1966, Erskine and Galganski 1967, Hajny et al. 1967, Assarsson 1969, Assarsson et al. 1970, Springer et al. 1974). The rise in extractives at the top of the pile has been attributed to the upward convection flow of moist air as it travels from the warmer lower regions of the pile to the cooler upper regions. As the moist air rises, it cools and condenses on the chips. Table 4.6 shows the resulting ANCOVA table for the analysis of the extractive content data. Again, all the factors were highly significant.

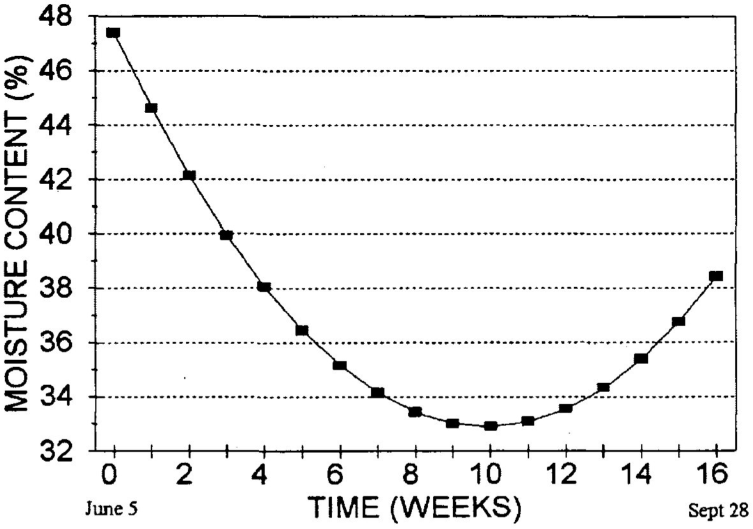

Figure 4.3 Estimated regression line for the summer pile moisture content vs time.

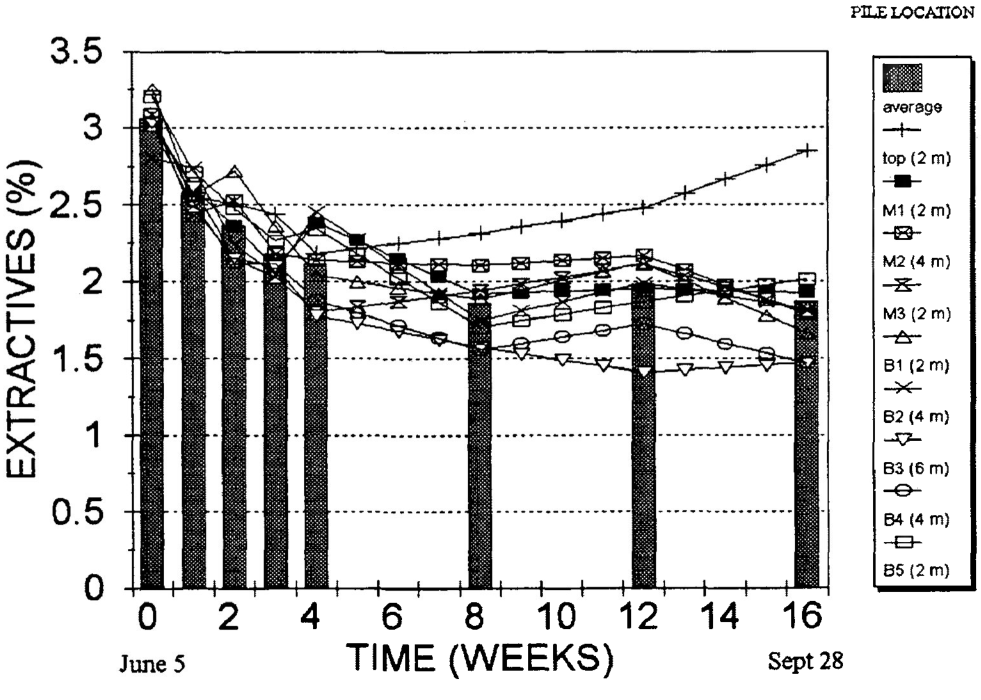

Figure 4.4 Average extractive contents at each of the 9 sampling locations (summer pile) vs time.

Table 4.6 ANCOVA table for the extractives data (summer pile).

SOURCE	DF	Seq SS	Adj SS	Adj MS	F	P(F)
time	1	52.001	41.296	41.296	549.23	0.000
time^2	1	22.398	22.500	22.500	299.25	0.000
aspect	2	1.373	2.988	1.494	19.87	0.000
height	2	6.747	1.140	0.570	7.58	0.001
depth	2	4.753	4.753	2.377	31.61	0.000
Error	632	47.520	47.520	0.075		
Total	640	134.792				

The adjusted extractive content means and their standard errors are shown in Table 4.7. These overall means (i.e., overall averages for the experiment) have been adjusted for time thereby eliminating any remaining effect of the covariate (time).

Table 4.7 Overall adjusted means and standard errors for the extractive content (%) (summer pile).

Aspect	Mean	Standard Error
N	2.0	0.04958
S	2.0	0.04963
Centre	2.3	0.01883
Height		
Top	2.1	0.07182
Middle	2.1	0.03062
Bottom	2.2	0.01537
Depth		
2 m	2.4	0.01531
4 m	2.2	0.03059
6 m	1.8	0.07192

The 6 m depth (i.e. bottom, middle of the pile) showed the lowest overall extractive content (1.8%), whereas the 2 m depths showed the highest overall extractive contents (2.4%). This would be expected since the extractives broke down faster in the middle of the pile then they did on the outside of the pile since there was less heat and chemical reactions nearer the surface of the pile.

Tukey's critical value was calculated as 0.078 at the 99% confidence level for the extractive means. A comparison of this critical value with the mean differences for aspect is shown in Table 4.8.

Table 4.8 Comparison of the aspect mean differences with Tukey's critical value (extractive content).

		N	S	Centre
		2.0	2.0	2.3
Centre	2.3	0.324**	0.244**	-
S	2.0	0.080**	-	-
N	2.0	-	-	-

Table 4.8 shows that all the aspect means are significantly different from one another. Table 4.9 shows a comparison of the height mean differences of the extractive data with Tukey's critical value.

Table 4.9 Comparison of the height mean differences with Tukey's critical value (extractive content).

		Middle 2.1	Top 2.1	Bottom 2.2
Bottom	2.2	0.112**	0.071ns	-
Top	2.1	0.041ns	-	-
Middle	2.1	-	-	-

The means for the top and the bottom sampling areas as well as the means for the top and middle sampling areas, according to Table 4.9, are not significantly different from one another. The middle sampling location means are, however, significantly lower than the bottom means. The comparison of the depth mean differences with Tukey's critical value is shown in Table 4.10. The table shows that all the depth means are significantly different from one another.

Table 4.10 Comparison of the depth mean differences with Tukey's critical value (extractive content).

		6 m 1.8	4 m 2.2	2 m 2.4
2 m	2.4	0.577**	0.183**	-
4 m	2.2	0.394**	-	-
6 m	1.8	-	-	-

The regression equation for the extractive content data as shown in Figure 4.5, along with the standard errors of the coefficients (in brackets) is as follows :

Extractive Content (%)= 2.79 - 0.186 time (weeks)+0.00848 time^2 (weeks)
(0.008935) (0.0005514)

The r^2 for this equation is 0.552. The ANCOVA table for the regression equation (Table 4.11) shows the equation to be highly significant.

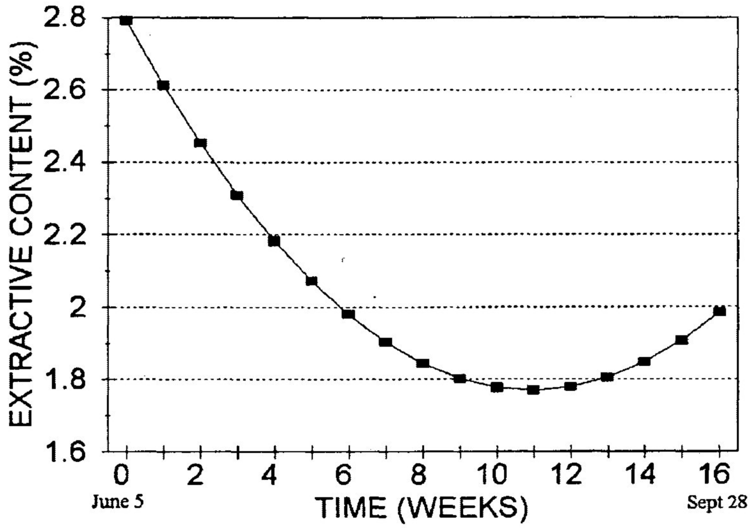

Figure 4.5 Estimated regression line for the summer pile extractive content vs time.

Table 4.11 ANCOVA table for extractive content regression analysis (summer pile).

SOURCE	DF	SS	MS	F	P(F)
Regression	2	74.399	37.200	392.98	0.000
Error	638	60.393	0.095		
Total	640	134.792			

The plotted regression equation (Figure 4.5) shows a minimum extractive content at approximately week 11. The regression extractive content decreases from 2.8% at week 0 to 1.8% at week 11. This is an absolute decrease of 1.0% (36% of the original starting extractive content). This regression line fits the data quite well for the first 12 weeks (where it reaches its minimum), but then starts to rise for the 4 weeks. This rise could be falsely interpreted since the top sampling area increased dramatically from 2.5% at week 12 to 2.9% at week 16. The average of the remaining areas in the pile decreased from 1.9% at week 12 to 1.7% at week 16. This regression equation can still be used in this study since most aspen chip piles would not be left to age for more than 3 months. For periods under three months, this equation is a satisfactory estimator for this study. If the chips are to be stored for more than 3 months, then the minimum value of the regression equation should be used.

4.2.3 BASIC DENSITY

The resulting ANCOVA table for basic density data obtained from the summer pile is shown in Table 4.12.

Table 4.12 ANCOVA table for the basic density data (summer pile).

SOURCE	DF	Seq SS	Adj SS	Adj MS	F	P(F)
time	1	0.323687	0.000009	0.000009	0.01	0.933
$time^2$	1	0.021453	0.021482	0.021482	17.44	0.000
aspect	2	0.001343	0.002028	0.001014	0.82	0.439
height	2	0.007274	0.007153	0.003576	2.90	0.055
depth	2	0.000752	0.000752	0.000376	0.31	0.737
Error	2143	2.640298	2.640298	0.001232		
Total	2151	2.994807				

The only significant factor present is $time^2$, which is highly significant. All other factors are not significant. The overall adjusted means and their standard errors are shown in Table 4.13. Again, all means have been adjusted for time.

Table 4.13 Overall adjusted means and standard errors for the basic density data (summer pile).

	Mean	Standard Error
Aspect		
N	0.43	0.003467
S	0.43	0.003468
Centre	0.43	0.001309
Height		
Top	0.43	0.005019
Middle	0.43	0.002142
Bottom	0.43	0.001069
Depth		
2 m	0.43	0.001071
4 m	0.43	0.002140
6 m	0.43	0.005018

The regression equation describing the basic density value and its standard error (in brackets) is :

$$\text{Basic Density (g/cm}^3\text{)} = 0.440 - \underset{(0.00000872)}{0.000146}\ \text{time}^2\ \text{(weeks)}$$

The time covariate was left out because it was not significant. The r^2 value for the equation is 0.115. The r^2 value for the basic density data is small because the data had such a large spread, therefore a satisfactory fit was hard to obtain. The regression equation's ANCOVA table is shown in Table 4.14. The table shows the equation to be highly significant.

Table 4.14 ANCOVA table for the estimated basic density regression equation (summer pile).

SOURCE	DF	SS	MS	F	P(F)
Regression	1	0.34513	0.34513	280.05	0.000
Error	2150	2.64968	0.00123		
Total	2151	2.99481			

The average basic densities, as well as the regression line are shown in Figure 4.6. Due to the nature of the basic density data, no maximum or minimum value can be calculated from the regression equation. The regression line shows a steady drop from start to finish. On average, the basic density dropped 2.4%/month during the summer, or a total of 9.6% over the 4 month storage period.

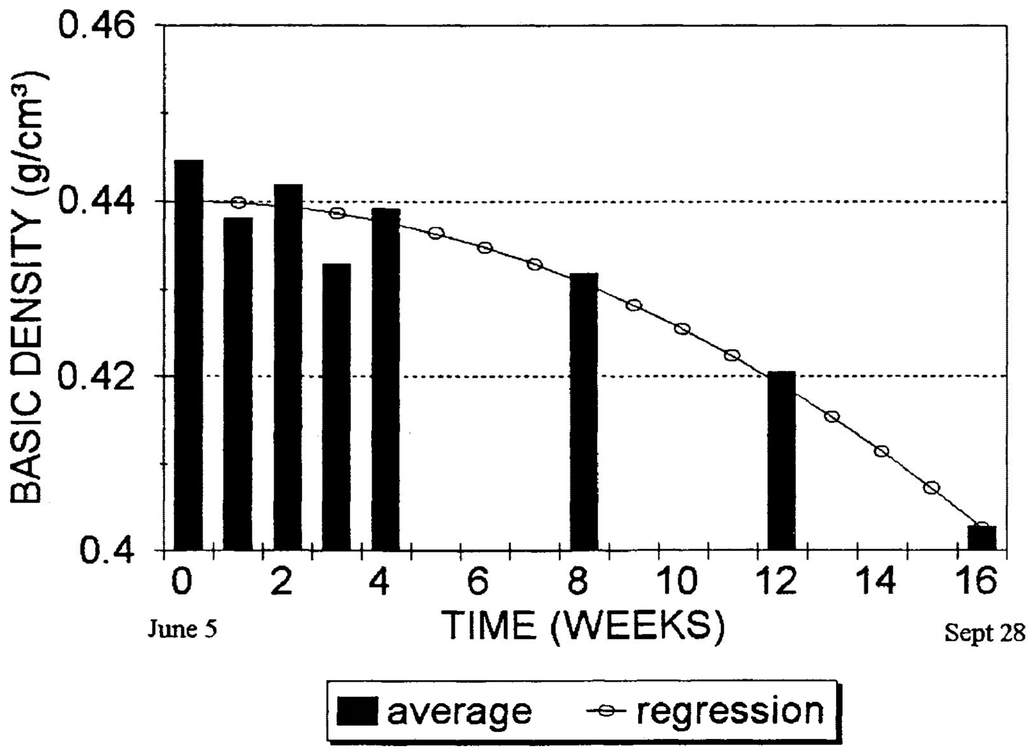

Figure 4.6 Average basic density and regression line (summer pile) for basic density and time.

4.2.4 TEMPERATURE

The analysis of variance for the temperature sensors calibration data showed that there were no statistical differences between the readings that each sensor gave.

All factors for the summer temperature data, as shown by the ANCOVA table in Table 4.15, are highly significant.

Table 4.15 ANCOVA table for the temperature data (summer pile).

SOURCE	DF	Seq SS	Adj SS	Adj MS	F	P(F)
time	1	6704.5	8030.0	8030.0	228.66	0.000
$time^2$	1	4736.4	4778.9	4778.9	136.08	0.000
aspect	2	5476.2	610.8	305.4	8.70	0.000
height	2	3079.1	3041.9	1520.9	43.31	0.000
depth	2	1634.8	1634.8	817.4	23.28	0.000
Error	339	11904.8	11904.8	35.1		
Total	347	33535.7				

The overall adjusted means for the temperature value, as well as their standard errors are shown in Table 4.16. All means have been adjusted for time.

Table 4.16 Overall adjusted means and standard errors for the temperature data (summer pile).

	Mean	Standard Error
Aspect		
N	28.5	1.4606
S	30.7	1.4523
Centre	34.3	0.5479
Height		
Top	30.9	2.1047
Middle	35.0	0.8987
Bottom	27.5	0.4488
Depth		
2 m	26.7	0.4481
4 m	33.0	0.9018
6 m	33.7	2.1036

Tukey's critical value was calculated as 0.55 at the 99% confidence level for the temperature means. A comparison of this critical value with the mean differences for aspect is shown in Table 4.17.

Table 4.17 Comparison of the aspect mean differences with Tukey's critical value (temperature).

		N 28.5	S 30.7	Centre 34.3
Centre	34.3	5.78**	3.59**	-
S	30.7	2.19ns	-	-
N	28.5	-	-	-

Table 4.17 shows that the north aspect temperature means are not significantly different from the south aspect temperature means. The centre temperature means, however, are significantly different from the north aspect and south aspect means. Table 4.18 shows a comparison of the height mean differences of the temperature data with Tukey's critical value.

Table 4.18 Comparison of the height mean differences with Tukey's critical value (temperature).

		Bottom 27.5	Top 30.9	Middle 35.0
Middle	35.0	7.51**	4.05**	-
Top	30.9	3.46**	-	-
Bottom	27.5	-	-	-

Table 4.18 shows that all the height means are significantly different from one another. The comparison of the depth mean differences with Tukey's critical value is shown in Table 4.19.

Table 4.19 Comparison of the depth mean differences with Tukey's critical value (temperature).

		2 m	4 m	6 m
		26.7	33.0	33.7
6 m	33.7	6.97**	0.63ns	-
4 m	33.0	6.34**	-	-
2 m	26.7	-	-	-

Table 4.19 shows that the mean temperatures at the 4 m depths are not significantly different from the mean temperatures at the 6 m depths. The 2 m temperature means, however, are significantly different from both the 4 m and 6 m temperature means.

Figure 4.7 shows the average outside air temperature during the summer months of the study. Figures 4.8, 4.9 and 4.10 show the temperature development in the summer chip pile at the top, middle and bottom of the pile, respectively. The most fluctuating temperatures were found at the top of the pile (Figure 4.8). Temperatures dropped as low as 12.0°C and went as high as 45.3°C. A possible explanation for this is that the top location was effected to a larger extent by the outside environment than the other 8 locations.

The middle of the pile (i.e. height = middle, depth = 4 m) was the location of

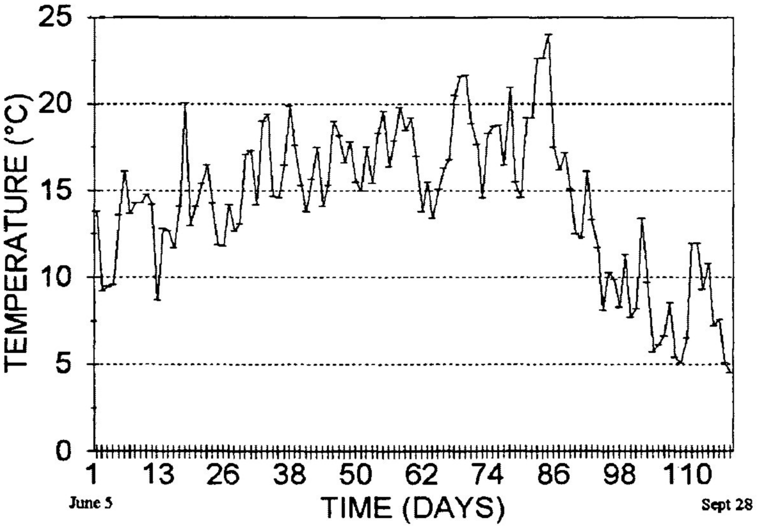

Figure 4.7 Average outside air temperatures during the summer chip pile months.

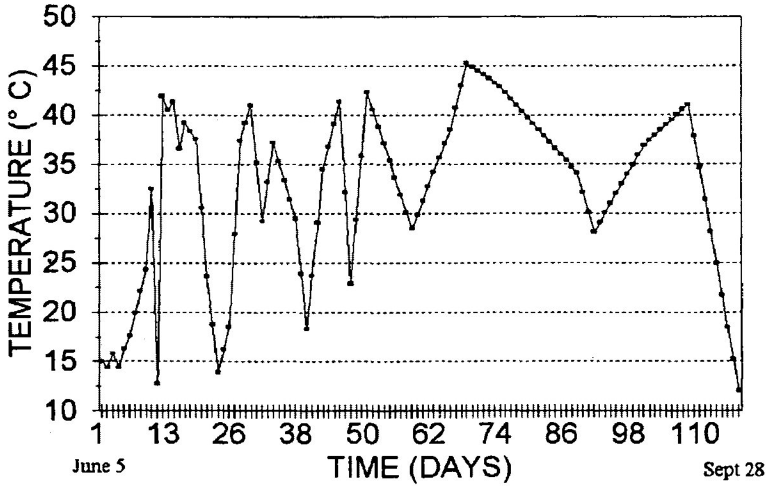

Figure 4.8 Daily average temperatures at the top sampling location in the summer pile.

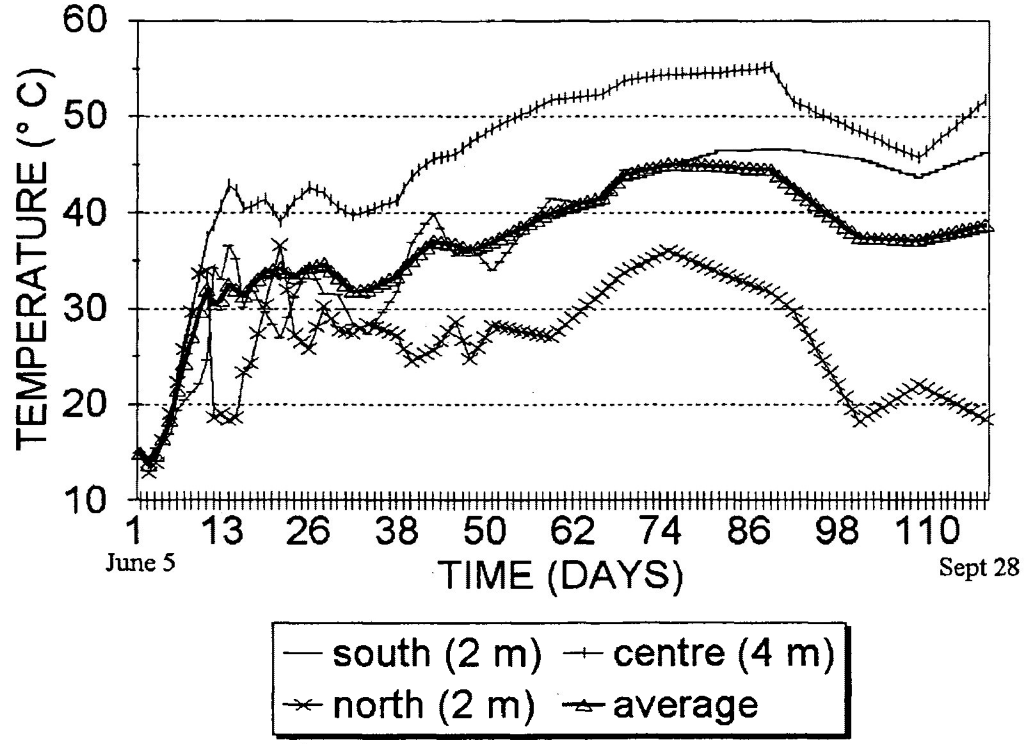

Figure 4.9 Daily temperatures for the middle locations of the summer pile.

the highest temperatures (i.e., max 55.1°C)(Figure 4.9). The middle sampling locations all showed the same general trend of a sharp rise in temperature for the first 12 days and then a gradual levelling off. The initial heating up of the pile (i.e., the first 12 days) was at a rate of 1.5°C/day.

The bottom of the pile (Figure 4.10), at a depth of 6 m, showed the second highest maximum temperature of 46.0°C. Both 2 m locations at the bottom of the pile showed a similar temperature development. They also showed the coolest temperatures of the 5 bottom locations. The two 4 m depth locations also showed similar temperature developments up until around day 80. Temperature readings

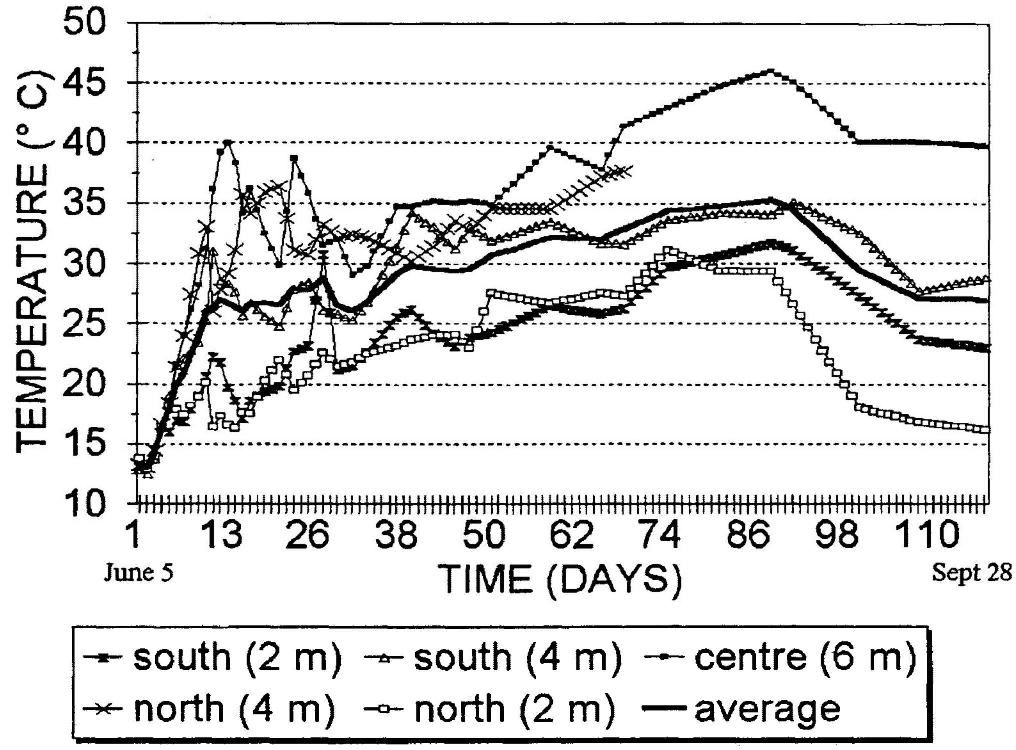

Figure 4.10 Daily temperatures from the bottom locations in the summer pile.

could not be taken on the north bottom 4 m pipe after this period because a fox chewed off the sensor wire that attached the thermocouple to the hand-held thermometer. The sharpest rise in temperature also occurred in the first 12 days, showing a rate of increase of 1.1°C/day. Figure 4.11 shows the average pile temperatures from the top, middle and bottom plotted along with the regression line. Figures 4.12 through 4.16 show the temperature isotherms within the summer pile throughout its development. Figure 4.12 shows the isotherms in the summer pile after 2 weeks; the approximate time that the pile temperatures began to level off. Figures 4.13 through 4.16 show the pile isotherms during each of the subsequent

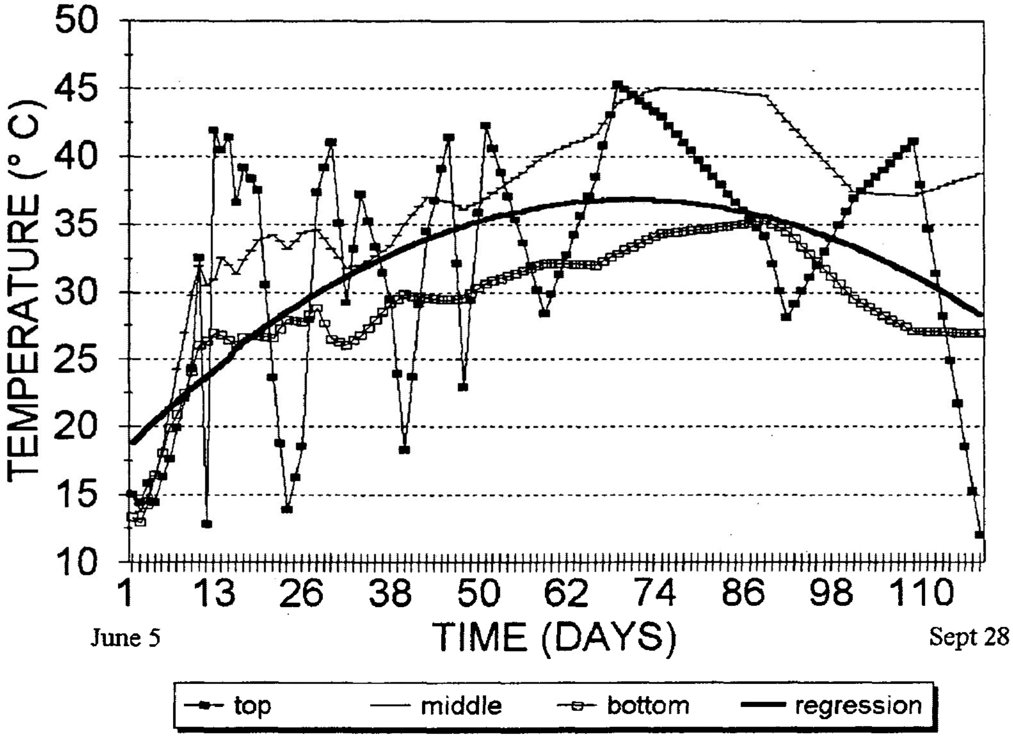

Figure 4.11 Average pile temperature values and their estimated regression line (summer pile).

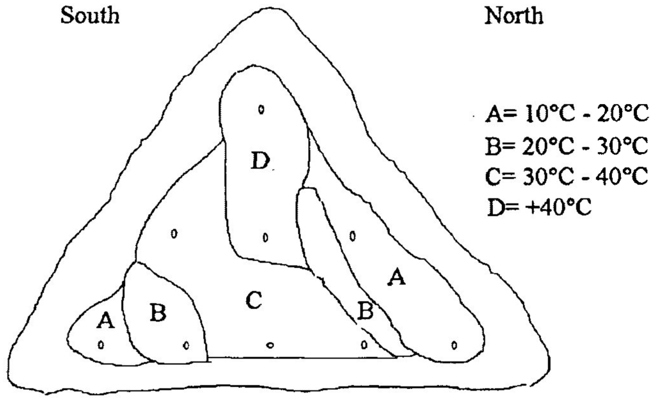

Figure 4.12 Temperature isotherms after 2 weeks in the summer pile.

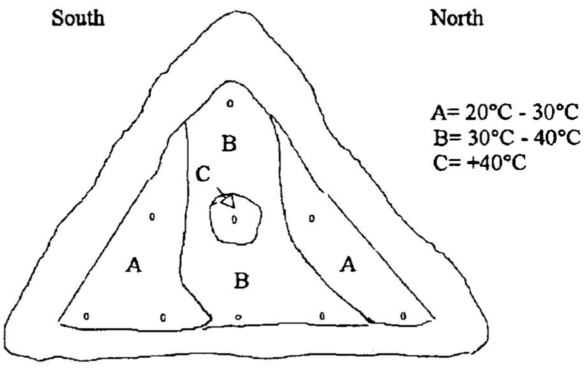

Figure 4.13 Temperature isotherms after 1 month in the summer pile.

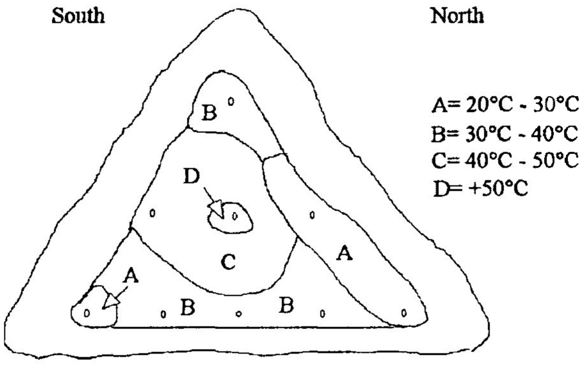

Figure 4.14 Temperature isotherms after 2 months in the summer pile.

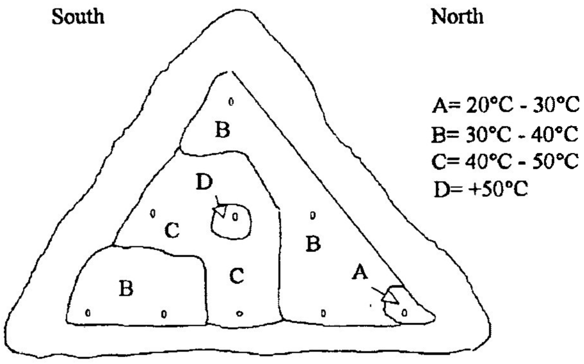

Figure 4.15 Temperature isotherms after 3 months in the summer pile.

months (i.e. month 1 to month 4). The upper portion of the chip pile showed the largest amount of deviation (Figure 4.12, 4.13, 4.14, 4.15 and 4.16) dropping from temperatures greater than 40°C in the first 2 weeks to temperatures ranging from 10°C - 20°C in the fourth month. The hottest area in the pile as shown by figures 4.12 through 4.16 is the middle, which had temperatures greater than 50°C from month 2 to month 4 (i.e. pile break down).

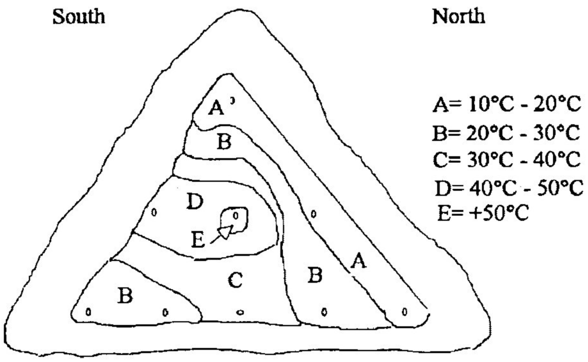

Figure 4.16 Temperature isotherms after 4 months in the summer pile.

Regression analysis on the temperature data yielded the following equation and standard errors (in brackets) :

$$\text{Temp } (^\circ\text{C}) = 18.8 + \underset{(0.04699)}{0.525}\ \text{time (day)} - \underset{(0.0004438)}{0.00382}\ \text{time}^2\ \text{(day)}$$

The r^2 for this equation is 0.341. The ANCOVA table for the regression equation is presented in Table 4.20 and shows the equation to be highly significant.

Table 4.20 ANCOVA table for the summer chip pile temperature regression analysis.

SOURCE	DF	SS	MS	F	P(F)
Regression	2	11440.8	5720.4	89.32	0.000
Error	345	22094.9	64.0		
Total	347	33535.7			

The average values for each level of the pile, as well as their regression equation are plotted in Figure 4.11. The figure shows that the temperatures at each level in the pile and the regression equation all follow the same general trend as the average air temperature outside of the pile (Figure 4.7). Based on the regression equation, a maximum temperature is calculated at approximately day 69.

4.3 WINTER PILE

4.3.1 MOISTURE CONTENT

One of the objectives of this study was to compare a chip pile built in the summer with one built in the winter. The results of the winter pile data show no need for a comparison with the summer pile for two reasons. First, due to the cold temperatures and snowy conditions during pile construction, 5 of the 9 sampling pipes froze up in the time period between pile building and pile break-down and as a result, no intermediate samples could be taken from them for the duration of the study. Secondly, the pile itself also remained frozen throughout. Since the chips were frozen, no significant amounts of extractives, moisture content or basic density losses occurred. The values for the average moisture contents during the winter pile are shown in Figure 4.17. The graph illustrates that there were no significant changes in the pile moisture content. The maximum moisture content being 51.5% and the minimum moisture content being 47.8%.

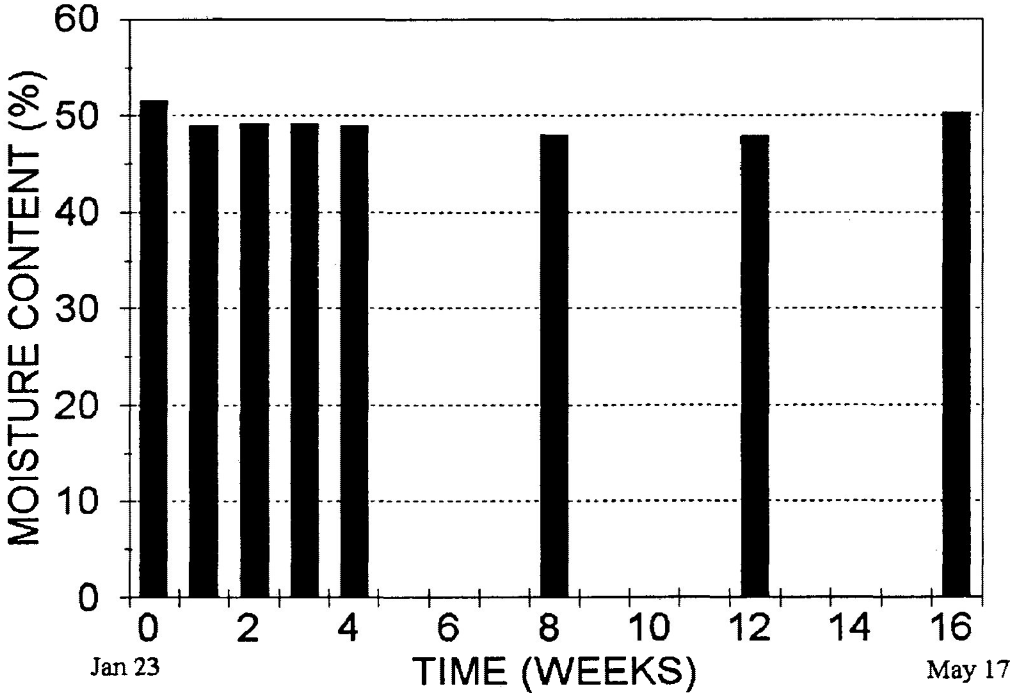

Figure 4.17 Average moisture contents for all sampling locations in the winter pile by week.

4.3.2 EXTRACTIVES

The average extractive content at each of the nine sampling locations (Note;

locations B2, B3, B5, M1 and M2 were sampled only at the start and at the end), along with the overall averages are shown in Figure 4.18. All pile locations, but the top, remained at a relatively constant extractive content. The top location, like the top location in the summer pile, however, showed a dramatic rise at the month 4 sampling period. It continued its steady rise until the end of the study. No reason as to why this occurred could be found in the literature.

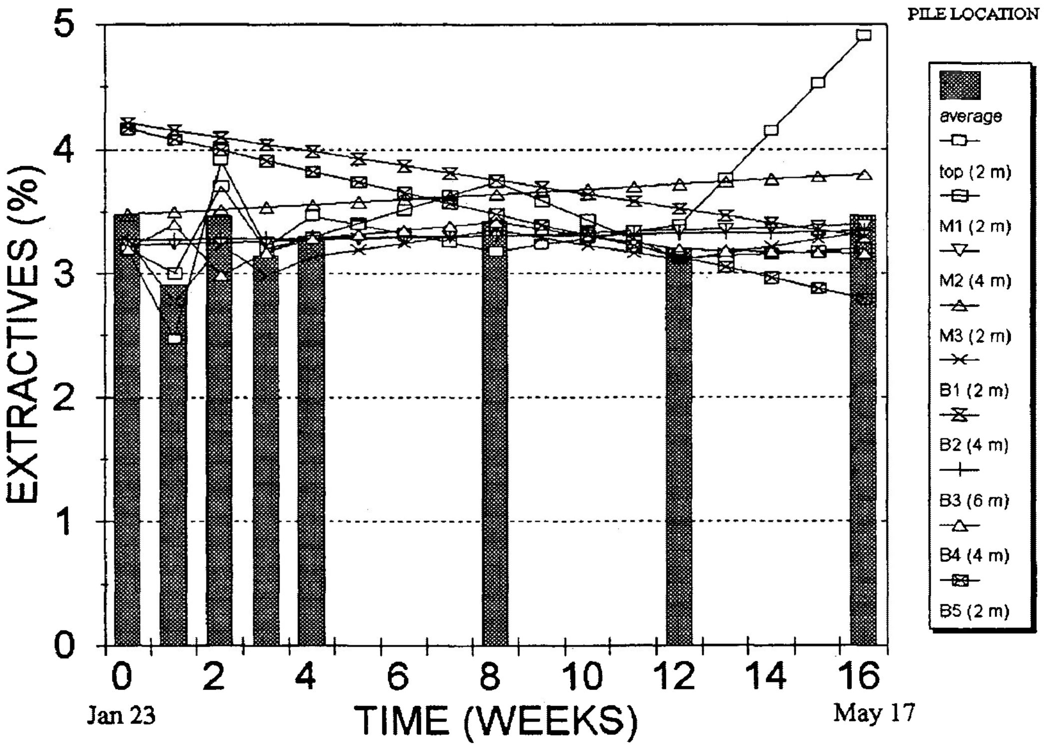

Figure 4.18 Average extractive contents for all sampling locations in the winter pile by week.

4.3.3 BASIC DENSITY

Figure 4.19 shows the same characteristics for basic density as the other two previous winter pile graphs (i.e. remaining relatively stable throughout the entire time period).

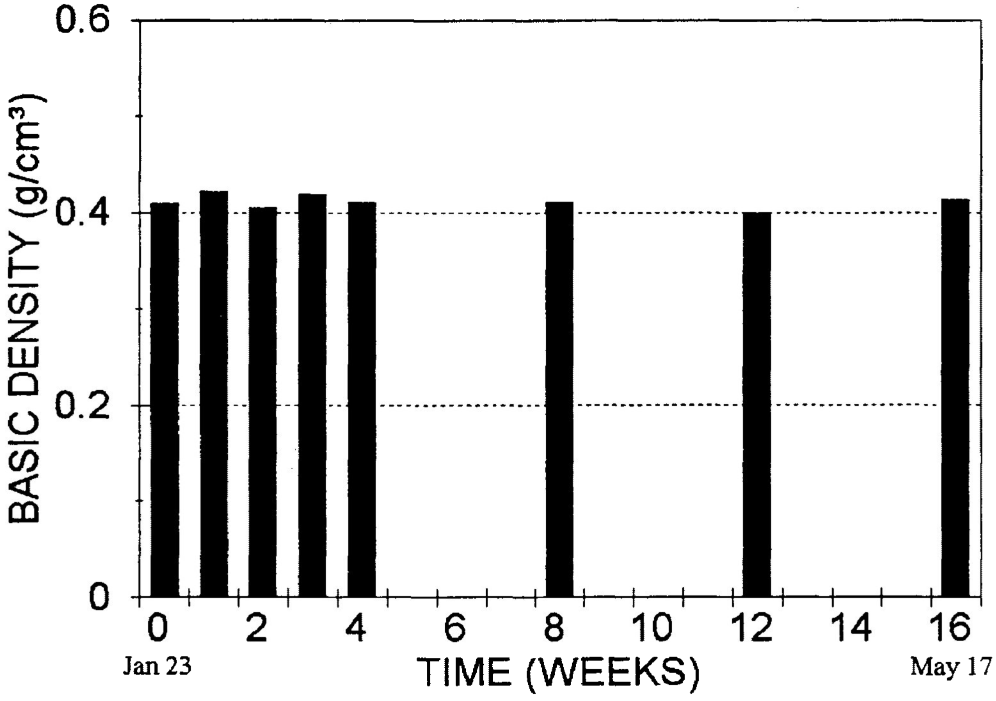

Figure 4.19 Average basic density for all sampling locations in the winter pile by week.

4.3.4 TEMPERATURE

Figure 4.20 shows the average outside air temperatures during the winter months of the study. Figures 4.21, 4.22 and 4.23 (i.e. top, middle and bottom of the pile

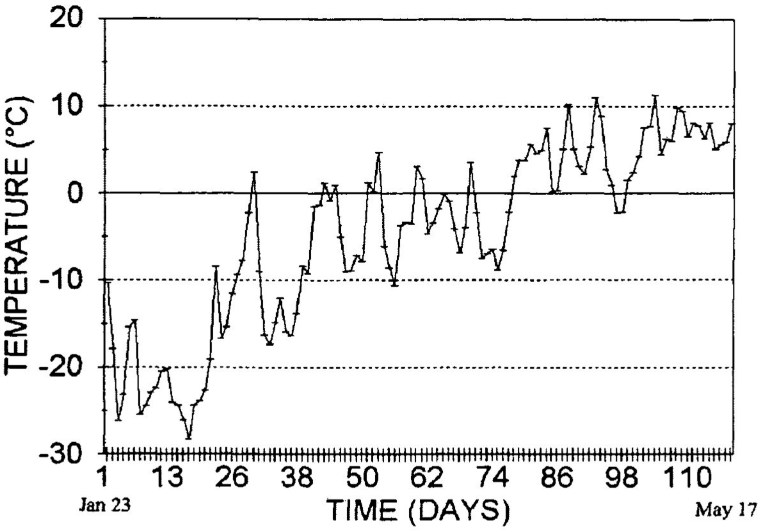

Figure 4.20 Average outside air temperatures during the winter chip pile months.

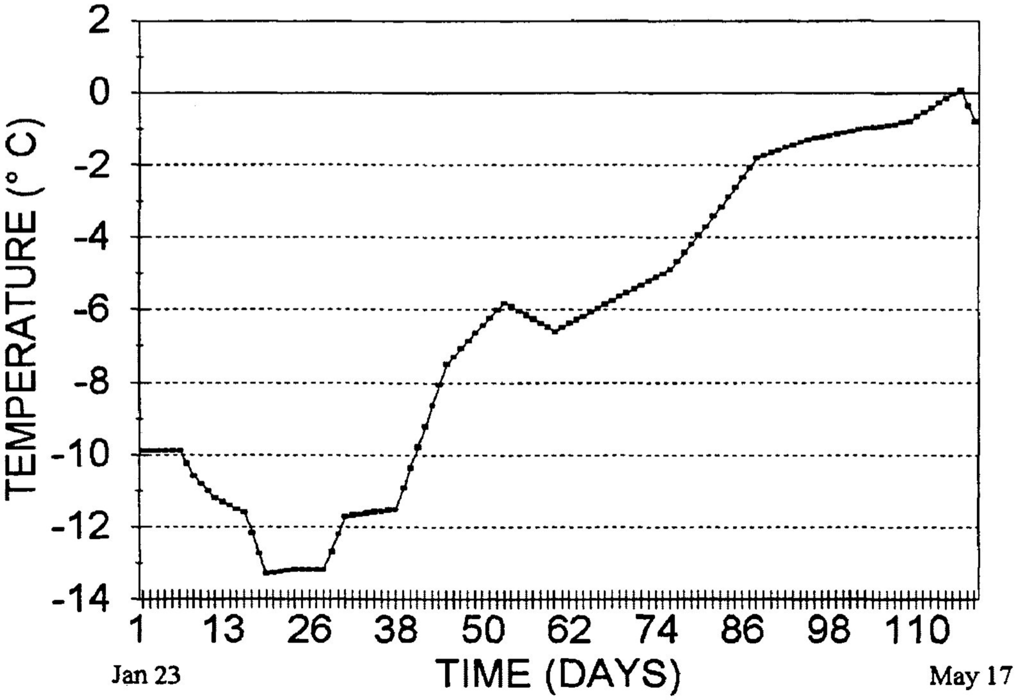

Figure 4.21 Temperature development at the top sampling location in the winter pile.

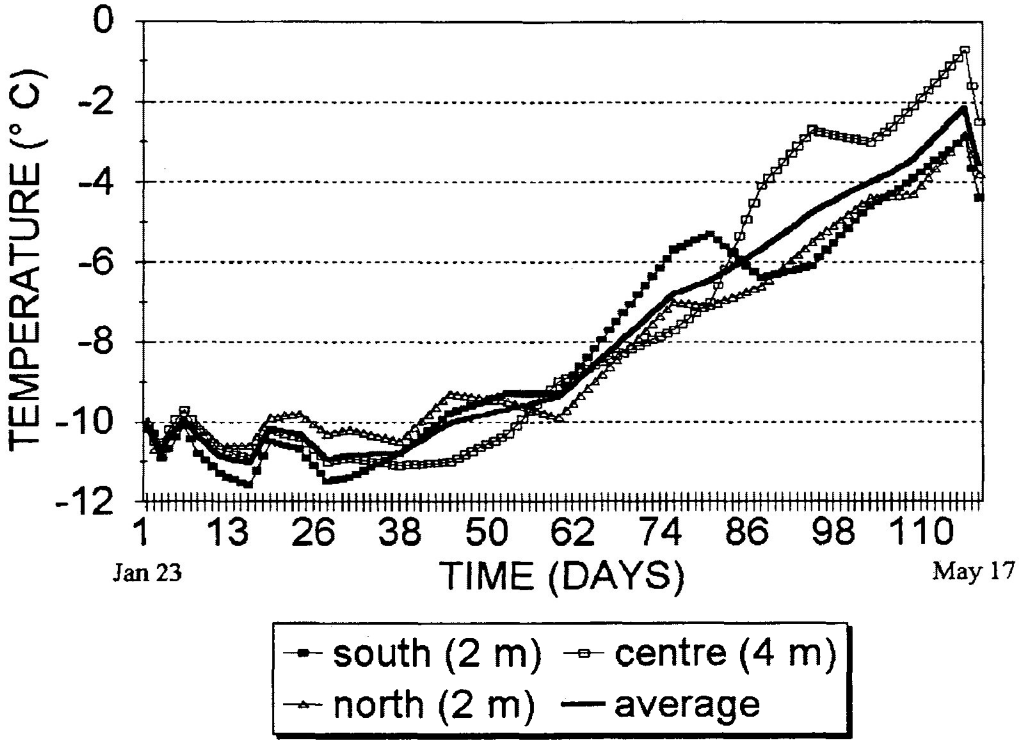

Figure 4.22 Temperature development at each depth in the middle of the winter pile.

respectively) all showed the same gradual thawing trend. At the end of the winter sampling period, all the pile temperatures were just below the freezing point. It is most probable that if the pile were left for an extended period of time, it would gradually thaw and begin to heat up at a similar rate as the summer pile. The middle sampling locations (Figure 4.22) showed that the 4 m depth area had slightly higher temperatures than the two 2 m areas. This would be expected since the 4 m sampling location was not affected to the extent that the 2 m locations were from the outside environment.

During the initial weeks (Figure 4.23), the deeper you went into the pile, the higher the temperatures got. After the first 8 weeks, however, all the sampling location temperatures gradually became more uniform.

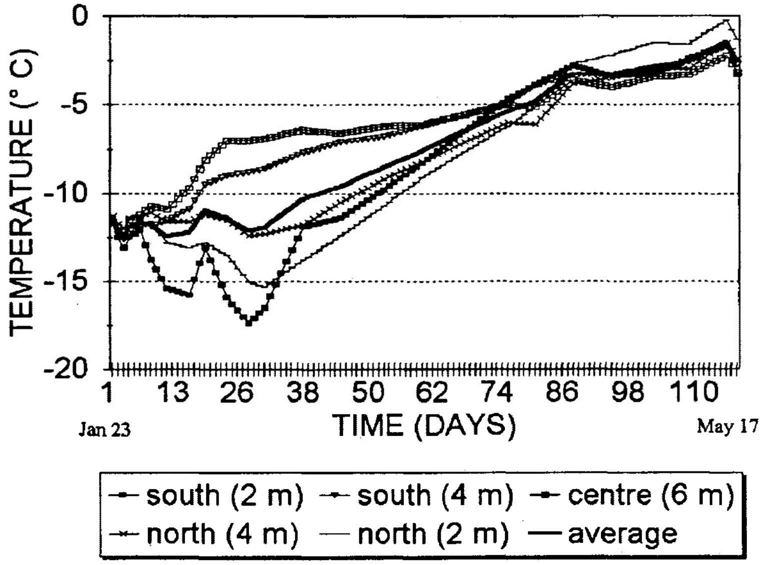

Figure 4.23 Temperature development at each depth at the bottom of the winter pile.

Figure 4.24 shows the average values for each of the 3 sampling heights in the winter pile. The graph shows that for the first month, very little temperature change occurred in the pile in any of the areas. After that period, however, all areas showed a gradual increase in temperature. All three locations in the pile exhibited the same gradual warming trend as the outside air temperature (Figure 4.20). The top portion of the pile showed the highest temperatures of the 3 areas and was the only location to reach a temperature above 0°C (i.e. 1 day at 0.1°C).

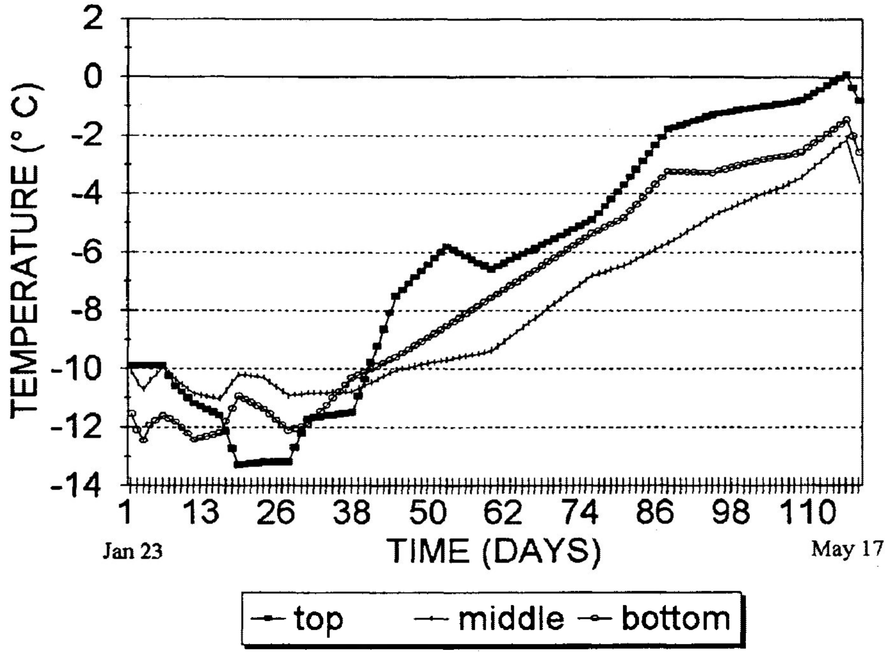

Figure 4.24 The average temperatures at each of the 3 levels in the winter chip pile.

4.4 ROUNDWOOD

4.4.1 MOISTURE CONTENT

A small sample of year-old aspen roundwood was analyzed to determine the moisture content and the extractive content of wood which would typically be used in Avenor's old woodhandling system. Ten bolts of wood were broken down into 5 samples (i.e. 2 bolts/sample). It was found that the average moisture content was 32.3% with a standard error of 0.9913 and a sample size of 30. Since there was quite a bit of fluctuation in the numbers, fairly large sample sizes were required. Table 4.21 shows the required sample sizes under 95% and 99% confidence levels and allowable errors.

Table 4.21 Moisture content sample sizes for the aspen roundwood under different conditions.

	CONFIDENCE LEVEL	
Allowable Error	95%	99%
10%	13	22
5%	46	79

4.4.2 EXTRACTIVES

The average extractive content for the aged roundwood was 2.5% with a standard error of 0.090 and a sample size of 15. Again, a relatively high standard deviation resulted. Table 4.22 shows the required samples sizes under different confidence levels and allowable errors.

Table 4.22 Extractive content sample sizes for the aspen roundwood under different conditions.

	CONFIDENCE LEVEL	
Allowable Error	95%	99%
10%	12	20
5%	40	68

4.4.3 BASIC DENSITY

No basic densities were measured in this experiment since all sampling occurred at the same time period.

4.5 DIGESTER AND FRESH ARRIVING CHIPS

4.5.1 MOISTURE CONTENT

The average moisture content of the fresh arriving chips was 47.9% with a standard error of 0.2959 and a sample size of 96. The average moisture content of the chips entering the digester was 40.2% with a standard error of 0.5408 and a sample size of 60. Using the Student's t-test method for populations with different sample sizes, it was found that the differences between the moisture content averages of the fresh arriving chips and the digester chips were highly significant.

Figure 4.25 shows the daily average moisture contents, on each of the sampling days, for both the fresh arriving chips and digester chips. In all cases but one, the fresh chips had higher daily average moisture contents than did the digester chips. The fresh arriving chips (coefficient of variation (CV) = 6.1%) showed a more uniform moisture content distribution as compared with the digester chips (CV = 10.4%) which had greater fluctuations in moisture content.

4.5.2 EXTRACTIVES

The average extractive content for the fresh arriving chips was 3.6% with a standard error of 0.0625 and a sample size of 90. The digester chips, on the other hand, had a lower average extractive content of 2.9% with a standard error of 0.0360 and a sample size of 59. The t-test value calculated for the extractive means was 8.8

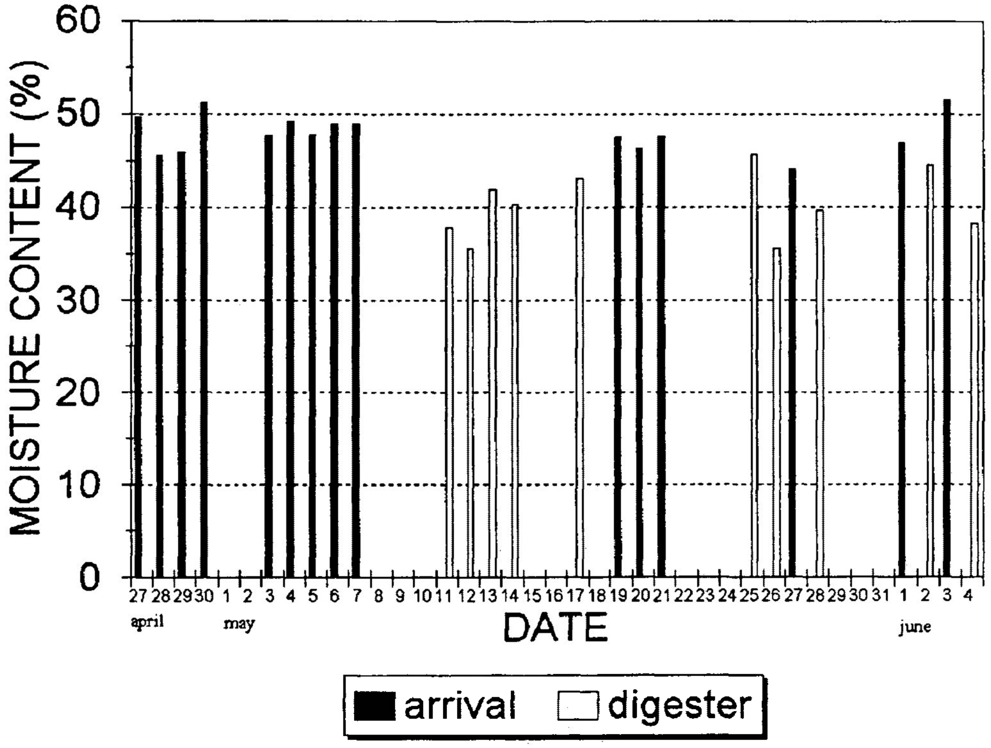

Figure 4.25 Daily average moisture contents for both the fresh arriving and digester chips.

meaning that the fresh arriving chip means and the digester chip means are significantly different from one another.

The digester chips showed a more even distribution of extractive content (CV = 9.5%) compared to the fresh arriving chips (CV = 16.5%) which showed a higher daily average fluctuation. Figure 4.26 shows the daily average extractive contents for both the digester and fresh arriving chips.

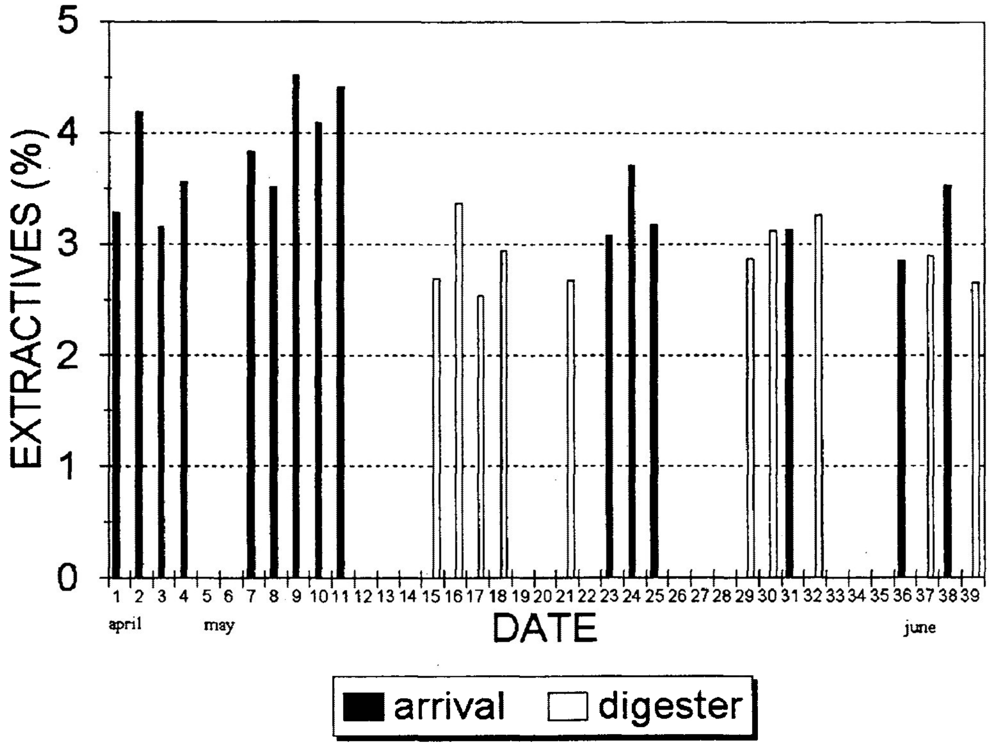

Figure 4.26 Daily average extractive contents for both the fresh arriving and digester chips.

4.5.3 BASIC DENSITY

The average basic density for the fresh arriving chips was 0.41 g/cm^3 with a standard error of 0.0038 and a sample size of 235. The average basic density for the digester chips was also 0.41 g/cm^3 with a standard error of 0.0026 and a sample size of 150. There was no significant difference between the fresh arriving chip basic densities and the digester chip basic densities. The coefficient of variations of the fresh arriving chips and the digester chips were 14.1% and 7.9%, respectively. The fresh arriving chips had higher variability then the digester chips. Figure 4.27 shows the daily average densities for both the fresh arriving and digester chips.

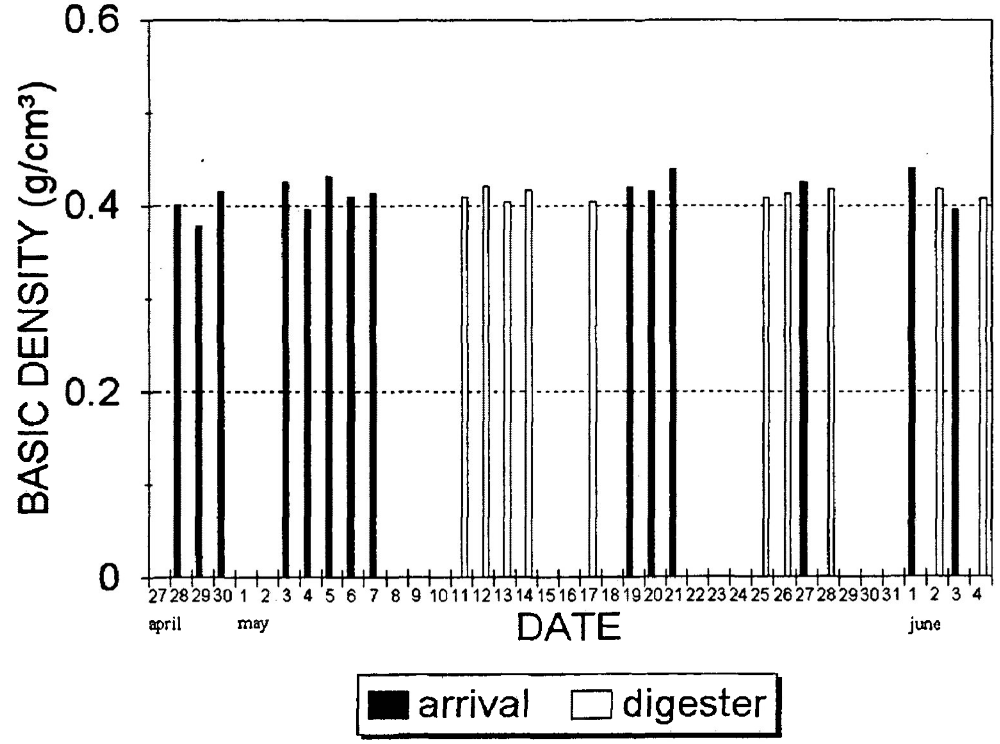

Figure 4.27 Daily average basic densities for both the fresh arriving and digester chips.

4.6 MAXIMUM EXPOSURE DATA

4.6.1 MOISTURE CONTENT

A regression analysis of moisture content on time was performed resulting in the following regression equation, showing each of the coefficient's standard errors in brackets :

$$\text{Moisture Content }(\%) = 47.5 - \underset{(0.1375)}{3.73}\text{ time (days)} + \underset{(0.005313)}{0.0948}\text{ time}^2\text{ (days)}$$

The r^2 value for this equation is 0.921. Figure 4.28 shows the daily average moisture content data plotted along with the regression equation.

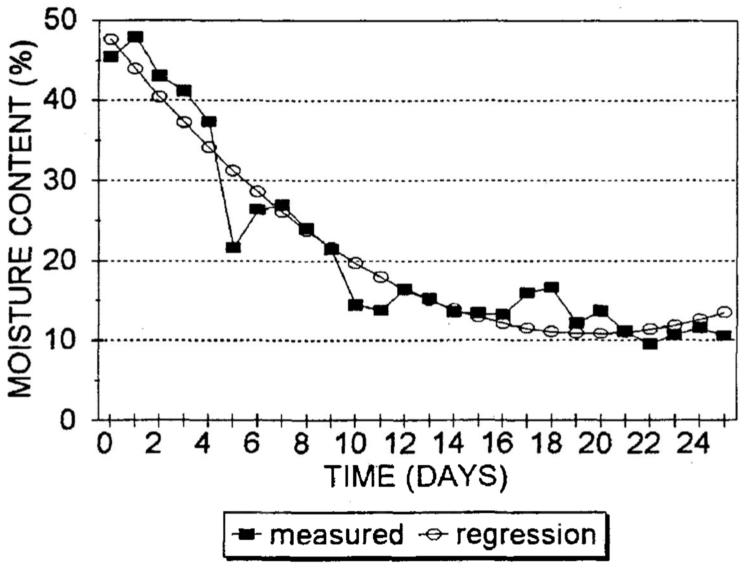

Figure 4.28 Average daily moisture contents and estimated regression line for the maximum exposure data.

From the regression equation, the minimum moisture content was determined at approximately day 20. The moisture contents, as calculated from the regression equation, decrease from 47.5% at day 0 to 10.8% at day 20. This represents an overall absolute decrease of 36.7% (i.e. 77% of the original moisture content). Again, this regression line fits the data quite well until it reaches its minimum point, where it starts to increase. The equation begins to lose its accuracy when trying to estimate times that are greater than day 20 since the moisture contents of the actual maximum exposure pile remained relatively unchanged from that time onwards. To overcome this, the minimum value will be used for all times greater than day 20.

An analysis of covariance (ANCOVA)(Table 4.23) shows the regression equation to be highly significant.

Table 4.23 Regression analysis for the moisture content maximum exposure data.

SOURCE	DF	SS	MS	F	P(F)
Regression	2	19802.4	9901.2	892.20	0.000
Error	153	1697.9	11.1		
Total	155	21500.3			

4.6.2 EXTRACTIVES

Regression analysis was also performed on the daily extractives data. The regression equation and standard errors for each coefficient (in brackets) is as follows:

$$\text{Extractive Content (\%)} = 2.95 - \underset{(0.005869)}{0.0610}\ \text{time (days)} + \underset{(0.0002269)}{0.00172}\ \text{time}^2\ \text{(days)}$$

This regression equation yielded an r^2 value of 0.727. Figure 4.29 shows the daily extractives data plotted along with its regression equation. The model again starts to rise at the point of the minimum value on the regression line. To over come this, the minimum value will be used for all points above the minimum time period on the

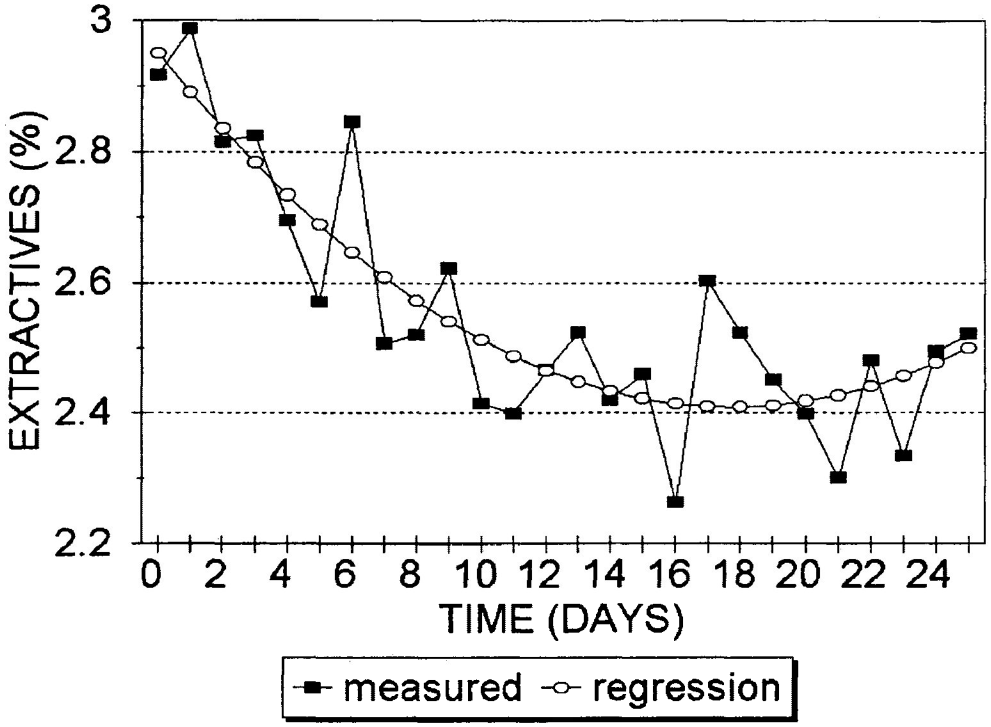

Figure 4.29 Maximum exposure extractive content data plotted with the estimated regression equation.

line.

A minimum extractive content is reached at approximately day 18 (i.e. 17.7 days). The regression extractive content decreases from 3.0% at day 0 to 2.4% at day 18. This represents an absolute decrease of 0.6% or 20% of the original extractive content. The ANCOVA table for this equation is presented in Table 4.24. It shows the regression equation to be highly significant.

Table 4.24 Regression analysis for the extractive content maximum exposure data.

SOURCE	DF	SS	MS	F	P(F)
Regression	2	1.99491	0.99746	98.67	0.000
Error	74	0.74809	0.01011		
Total	76	2.74301			

4.6.3 BASIC DENSITY

No basic densities were measured in this experiment since the duration of the experiment was only 26 days and as such, no significant amounts of basic density loss would likely occur.

4.7 STEAMING

4.7.1 MOISTURE CONTENT

No analysis was done for the moisture contents of the steaming data since they are not critical to this study.

4.7.2 EXTRACTIVES

The means for the extractive content steaming data and their standard errors are shown in Table 4.25.

Table 4.25 Means and standard errors for the extractive content steaming data.

Time	Mean	Standard Error
0 hours	3.3	0.1057
3 hours	3.7	0.1057
6 hours	3.8	0.1057

The extractive content rose from 3.3% at the start of the experiment to 3.8% at hour 6. This represents a 15.2% increase in the overall extractive content. The following regression equation, and standard errors (in brackets) for each coefficient was used to describe the extractive content during steaming :

$$\text{Extractive Content } (\%) = 3.30 + \underset{(0.08979)}{0.210\ \text{time (hours)}} - \underset{(0.01438)}{0.0210\ \text{time}^2\ \text{(hours)}}$$

The resulting r^2 value for the equation is 0.134. According to Table 4.26, the regression equation is highly significant.

Table 4.26 ANOVA table for the extractive content steaming regression equation.

SOURCE	DF	SS	MS	F	P(F)
Regression	2	4.5221	2.2611	6.75	0.002
Error	87	29.1342	0.3349		
Total	89	33.6564			

Figure 4.30 shows the net extractive changes in the steamed chips, as well as the control chips. The control data represents how the extractive content changes when the wood chips are left to sit at room temperature with maximum exposure to air. The graph shows a net increase in extractives over time with steaming, while for the control chips it remained relatively unchanged. Figure 4.31 illustrates how the extractive content of the chips continues to rise over a period of 24 hours.

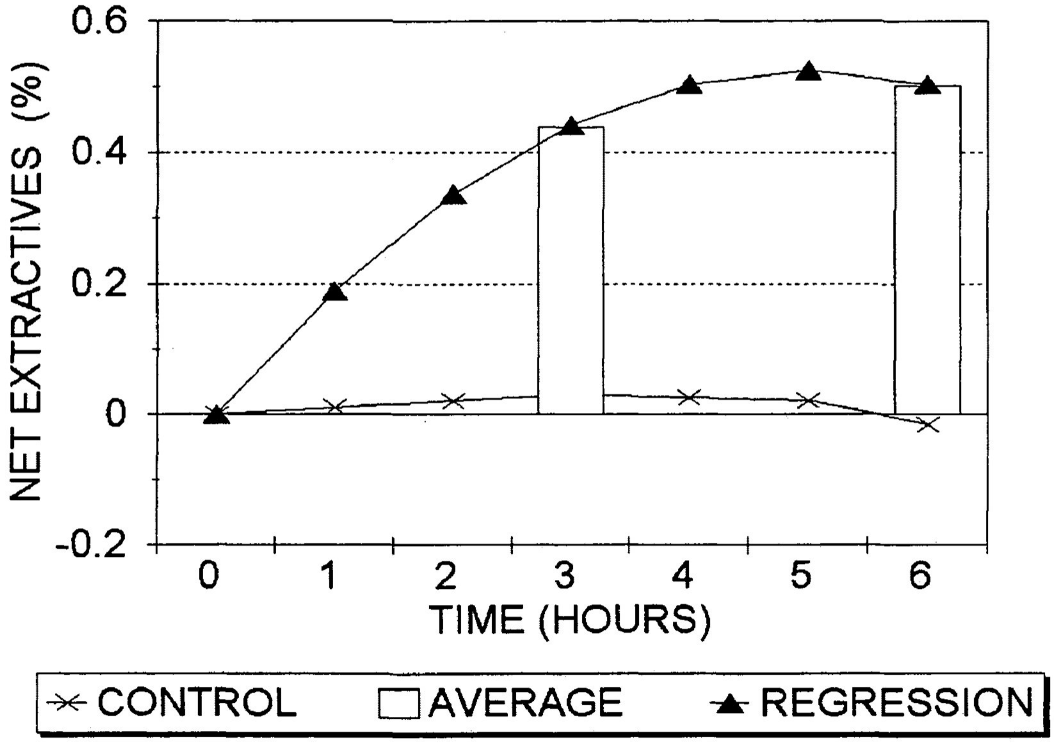

Figure 4.30 The net extractive content change for the average steamed chips and the control chips.

The ANOVA table for the steaming data is presented in Table 4.27. It shows that the time factor is highly significant.

Table 4.27 ANOVA table for the extractive content steaming data.

SOURCE	DF	Seq SS	Adj SS	Adj MS	F	P
time	2	4.5221	4.5221	2.2611	6.75	0.002
Error	87	29.1342	29.1342	0.3349		
Total	89	33.6564				

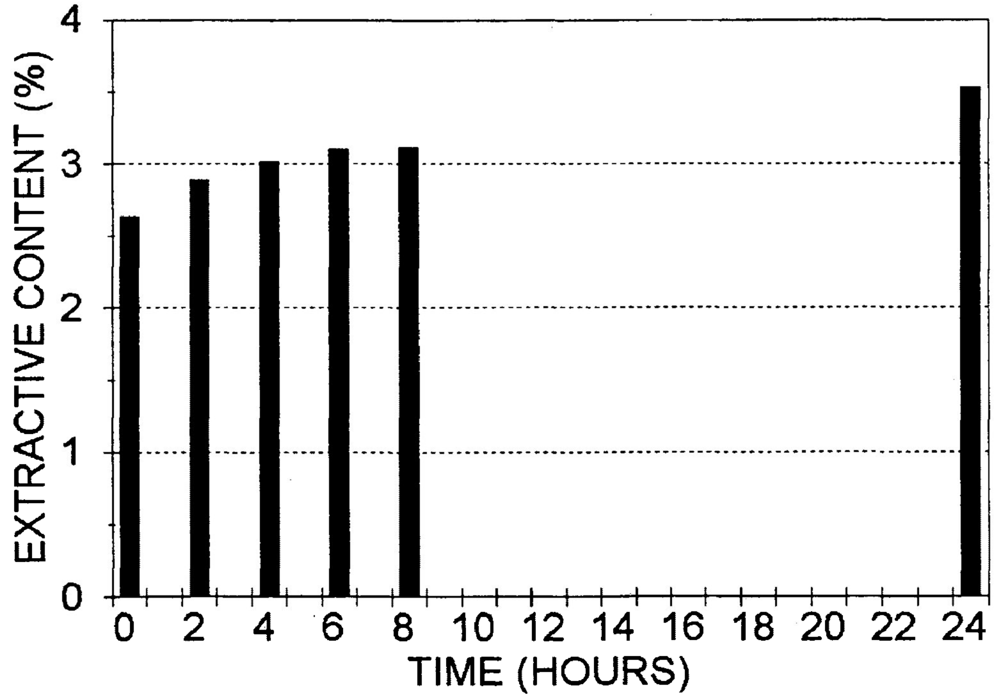

Figure 4.31 The extractive contents of wood chips over a 24 hour steaming period.

4.7.3 BASIC DENSITY

No basic densities were measured in this experiment since no significant amounts of basic density loss would likely occur due to its short duration.

5 DISCUSSION

5.1 SUMMER CHIP PILE

One result that all the analyses had in common was that time was a very important covariate. This is illustrated by the fact that in each ANCOVA and ANOVA table, time and time^2 represent a large percentage of the Error sums of squares, meaning that much of the variation could be explained by time. The replication values for each of the variables is 18 (MC), 9 (Ext) and 30 (Den).

It was an objective of this study to compare a chip pile built in the summer to one built in the winter. The winter chip pile remained frozen and showed minimal change in chip characteristics over 4 months therefore there is no need to statistically compare the results for the two piles.

Because of its importance to this study, the majority of the discussion will deal mainly with extractive contents and their losses.

5.1.1 MOISTURE CONTENT

Although it was found (Table 4.1) that the aspect and the height of the pile (depth in this case was not significant) were highly significant with respect to moisture content loss, from a practical point of view, the difference between means in Table 4.2 is probably not that noteworthy (the largest absolute difference being 7.0%). The most important factor would not necessarily be the actual means themselves but the variation in the moisture content of chips entering the mill process.

5.1.2 EXTRACTIVES

As in the previous discussion (moisture content), all factors considered for the loss of extractives (Table 4.6) were highly significant. The extractives loss trend is quite typical of other studies. The summer pile in this study showed a convection column type heating pattern in the pile , which resulted in the increase of extractives at the top of the pile. This trend is also seen in the reports of Bjorkman and Haeger (1963), Hajny (1966), Erskine and Galganski (1967), Hajny *et al.* (1967), Assarsson (1969), Assarsson *et al.* (1970), and Springer *et al.* (1974).

The absolute rate of extractives loss was, on average, 0.3%/month for the 4 month study period. However, during the first month, the absolute extractive content decreased 1.0% (i.e., 31% of the original) while at the end of the second month it dropped by 1.2% (i.e., 39% of the original). There was no change in the average extractive content from month 2 to month 4. Based on the regression equation a minimum extractive content is reached at 11 weeks (when the summer wood chip pile regression equation is used).

From Tukey's comparison of the chip pile means, it was found that the bottom middle location (height = bottom, depth = 6 m) showed the most significant difference, as well as the lowest extractive content when compared with the rest of the location means (i.e., extractive content of 1.8%) (Table 4.7). Taking this into account, a method of chip pile reclaim that takes only the bottom middle position chips would be preferred, thereby eliminating the possibility of using a surface chip reclaimer. An auger could be positioned underneath the pile in the middle to take only those chips (Figure 5.1). This would result in collection of the chips that had the lowest and most uniform extractive contents. As the bottom middle position chips were collected, the top, middle and side position chips would fill in the openings. Bulldozers or front-end loaders could also be used to push the sides into the middle of the pile. Caution must be used when pushing the chips into the middle of the pile to minimize the amount that the machinery travels on the pile. This will avoid any undue compaction of the chips, as well as unnecessary damage to the chips. This method of chip pile reclaim would use up all regions of the chip pile on a continuous basis thereby reducing the chance of producing a build-up of microorganisms which cause chip decay and deterioration. This method could also be used in the winter since the pile would never be completely frozen. As long as there were some regions left that were still producing heat, the problems of freezing that occurred in this study would be eliminated and faster chip seasoning would result.

For a fresh pile of aspen chips, a long term seasoning period of 2 weeks would be recommended to allow for the maximum amount of seasoning.

Avenor requires 1.1 million m^3 of aspen annually for its kraft mill. The kraft mill runs aspen for 250 days each year, however, all calculations will be based on a full year since the chips will be piled for that time. This results in a chip consumption of 21 154 m^3/week. This 21 154 m^3/week of aspen wood chips will come from the bottom middle location of the chip pile. A minimum pile size of 42 308 m^3 will be required to satisfy the 2 week seasoning period. A pile size of 63 462 m^3 (i.e., 3 weeks inventory) would be preferred since it would give the mill an extra

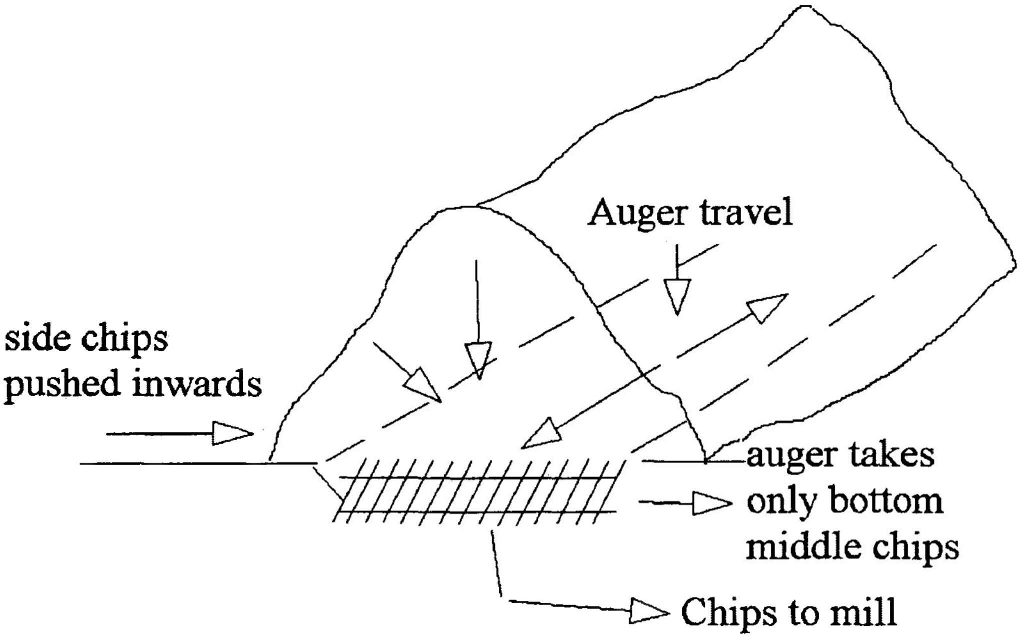

Figure 5.1 Diagram illustrating the proposed chip reclaim system.

weeks buffer of chips. The auger speed should be set so that 21 154 m^3 of aspen chips are taken from the bottom middle location of the pile each week. The gaps left by the augered chips are filled in by chips from other locations of the pile to start seasoning, thus a continuous cycle occurs. The benefit of this system is that all the aspen chips removed from the bottom middle location of the pile will have been aged. No waiting time is required. It is important to note that wood harvested in the winter or early spring may require longer seasoning times and therefore larger chip inventories.

From the roundwood experiment, an extractive content of 2.5% was calculated. This figure represents the extractive content that was entering the mill using the old method of ageing roundwood for one year and then introducing the wood into the mill process. Using the summer wood chip pile regression equation, approximately 1.5 weeks was required to season the fresh wood chips to a point where they were at the same extractive content as the aged roundwood. This seasoning period may be misleading though since the extractive contents of the roundwood and of the chips in the pile were taken at different times in the summer. Due to seasonal variation, as noted by Dunlop-Jones *et al.* (1991), extractive content will be larger in spring and early summer than it would in mid to late summer. It is safe to say, however, that this represents an increased savings over roundwood ageing due to smaller inventories, and lower handling and seasoning costs, to name a few. In general, the wood is cheaper when it is aged in chip form.

5.1.3 BASIC DENSITY

The loss of basic wood density during the summer pile was 2.4%/month, on average. It is a general rule of thumb with softwood chips, that a loss in wood substance of 1%/month can usually be expected (Hajny *et al.* 1967, Hatton 1969, Giffin 1970, Bergman 1972, Hulme 1975, Close 1986, Zreloff 1986). Hulme (1975) stated that the initial weight losses of 1% are not likely to be serious since the loss is probably due to the depletion of non-structural materials such as extractives, starches and sugars. Time was the only thing that was significant when it came to basic density loss. A total basic density loss for the 4 month study period was 9.6%, which is quite significant. No relation between position in the pile and basic density loss could be found. The data does reveal, though, that the longer the chips are left in the pile, the more they break down.

5.1.4 TEMPERATURE

The summer chip pile temperatures showed, as expected, that the closer to the middle of the pile one went, the hotter the temperatures became. The summer pile clearly showed a convection type heating column as stated in the reports of Bjorkman and Haeger (1963), Hajny (1966), Erskine and Galganski (1967), Hajny *et al.* (1967), Assarsson (1969), Assarsson *et al.* (1970), and Springer *et al.* (1974).

5.2 DIGESTER AND FRESH ARRIVING CHIPS

5.2.1 MOISTURE CONTENT

The moisture content results of the fresh arriving aspen chips agree with previous analyses done by other authors. The moisture contents measured in this study fell within the range of 45%-51% that Allen *et al.* (1991) stated was the average for fresh aspen chips. Branch (1971) also stated that the moisture content of fresh aspen chips is at 48.8%. To ensure good pulping results in the mill, the variation in moisture content should be as small as possible. The results of this experiment indicate that the variation in moisture content increased from fresh chips (CV=6.1%) to the digester chips (CV=10.4%). A possible explanation for this increase is the fact that the fresh chips were mixed with aged chips in a 50:50 ratio.

The aged chips would likely have lower moisture contents than the fresh chips and when mixed, one would expect the variation to be larger then it would be if only fresh chips or aged chips were used. This would indicate that the practice of mixing fresh and stored chips is questionable from the point of view of moisture content variation.

5.2.2 EXTRACTIVES

The average extractive content for the fresh aspen chips in this study was 3.6% using acetone as the solvent. It is quite difficult to try and compare figures between studies because the literature shows that no one particular solvent was used. Other solvents that have been used are benzene, ether and alcohol and hot water. Each of these solvents removes a different percentage and different kind of the extractives from the wood, and as a result the final extractive percentages cannot be accurately compared. Levitin (1970) stated that the acetone extractives of poplar range from 2.7%-3.2%. Allen (1988), on the other hand, stated that the acetone extractive content of aspen wood from a freshly cut tree was found to be 4.5%. It is not possible to compare the average extractive content value for this study (i.e., 3.6%) with the value obtained from Allen (1988) since the value obtained by Allen (1988) was from a freshly felled aspen tree. The aspen wood chips used in this study would not be considered fresh since there was probably some time between felling and chipping, as well as some drying out time as the chips were transported from the harvest site to the mill. Dunlop-Jones *et al.* (1991) talked about natural variation among trees and how this affects the total extractives. The natural variation is separated into variation within clones and between clones. There are also seasonal variations in the extractive content of the aspen wood. During the spring, when the wood is growing, more extractives will be present than during the period when the tree's growth is slowing down (Levitin 1970, Dunlop-Jones *et al.* 1991). This trend also appears in Figure 4.26. It shows the extractives in April (3.8%) to be, on average, higher than in June (3.2%). Because of this seasonal variation in extractive content, no comparison can be made between the different methods of wood handling (i.e., roundwood only, roundwood and chips, and chips only) since the data collected for each was gathered at different times in spring and summer.

The problem of trying to correlate operating problems in the kraft mill with the extractive content of the chips entering the mill process is a difficult one. No written accounts of any pitch problems in the mill were kept so all this study has to go on is the sampling of the chips that were entering the digester. The average extractive content of the aged wood that was entering the digester (2.9%) was higher than the average extractive content of the fresh chips used for the start of the summer chip pile. An explanation for this might be that the digester chip's average extractive content was measured earlier in the summer before the summer pile was started. As a result, a higher average extractive content would be more likely due to seasonal variation in the extractive content of the wood. The extractive contents of the digester chips showed a more even distribution (CV=9.5%) than in the fresh chips (CV=16.5%). This is important since proper pulping of the wood chips requires the chips to be as uniform as possible.

5.2.3 BASIC DENSITY

The average wood basic density for fresh wood chips was found to be 0.41 g/cm^3. The literature shows a very wide range of aspen wood density. The grouped density averages for aspen is in the range of 0.32 g/cm^3-0.45 g/cm^3 (Clermont and Schwartz 1951, Hale 1958, Besley 1960, Fogh 1961, Besley 1966, Amidon 1981, Singh 1986, Thomas 1987, Wong and Eng 1987, Horng *et al.* 1988, Koran 1989, Allen *et al.* 1991). The digester basic wood density was found not to be statistically different from the fresh arriving chip basic densities.

5.3 MAXIMUM EXPOSURE PILE

5.3.1 MOISTURE CONTENT

The maximum exposure pile data can be used to illustrate what would happen if the aspen chips had maximum exposure to air and were spread out completely so no compaction (heating) occurs, and no effects from the sun or the wind are present. In this case, the moisture content dropped from 47.5% at day 0 to a minimum moisture content at day 20 of 10.8%.

5.3.2 EXTRACTIVES

More important to this study, a minimum extractive content would be reached in the maximum exposure pile by day 18 (2.4%). This shows that maximum exposure of the chips to air may be more efficient and faster when compared to seasoning the wood chips by piling them. This method of OCS, however, would not be practical when large volumes of chips are involved, since a very large area would be required to make the piles as thin and long as possible. The method could, however, lead to methods of better pile aeration. One possibility would be to place a series of open ended perforated pipes through the chip pile to allow air to flow through the pipe, as well as into the pile (Figure 5.2).

In order to reach an extractive content equal to that of the aged roundwood (2.5%), an estimate of approximately 9 days using the maximum exposure regression equation would be required.

5.4 STEAMING

It was hypothesized at the beginning of this study, that shorter seasoning times would result if the chips were steamed, as compared to seasoning times in piles. Nugent *et al.* (1977) also stated that a commercial steamer was available that could rapidly age (i.e., reduce extractives) wood chips in a period of up to 72 hours with hot air temperatures of 55°C. The results of this study, however, show the opposite. The longer wood chips were steamed in a closed vessel, the higher the moisture content (expected) and extractive content went. A possible explanation for this is that steaming resulted in minor degradation of the wood components, resulting in increased solubility with acetone or water.

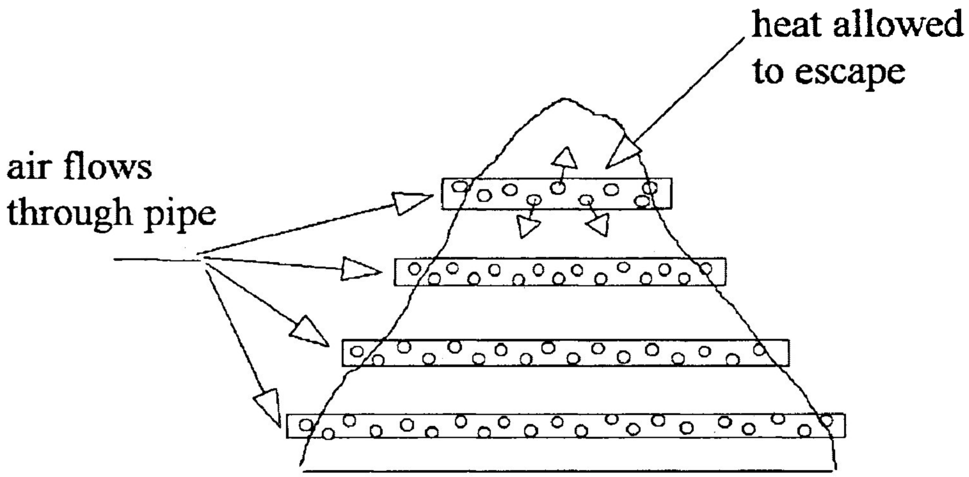

Figure 5.2 Diagram illustrating the chip pile aeration procedure.

6 CONCLUSION

The summer chip pile temperatures showed, as expected, that the closer to the middle of the pile one went, the hotter the temperatures became. It also showed a convection column type heating pattern in the pile. The pile showed a large increase in temperature during the first 12 days. From this period on, the pile remained at a relatively constant temperature. The middle location in the pile showed the highest temperature of 55.1°C, whereas the top position showed the lowest temperature of 12°C. The chip pile built in the winter had only one day where it reached a temperature above 0°C for the entire 4 month period. This temperature however, was only 0.1°C.

The loss of chip moisture content in the summer pile showed the same gradual decrease as with the extractives loss. The middle of the pile showed the lowest moisture content after 4 months (i.e., 38.2%), whereas the top of the pile showed the highest moisture content after the 4 months (i.e., 45.2%).

The rate of extractives loss in the summer chip pile was, on average, 0.3%/month for the four month study period. The first two months, though, showed monthly losses of 1.0%/month (i.e., 31% of original value) and 0.6%/month (i.e., 39% of original value) for the first and second month, respectively. No comparison of the summer chip pile was made with the winter chip pile since the winter chip pile remained frozen for the four month period and no significant changes in the chip characteristics occurred.

Time was shown to be a highly significant covariate in the seasoning of the aspen wood chips. The bottom middle location of the summer pile showed the greatest amount of extractives loss.

This study has shown that air drying the wood chips in small piles to eliminate the effects of compaction and heating may be a faster and more efficient method of seasoning than ageing chips in large piles. Taking this fact into account, methods to better aerate the chips should be developed. Placing open ended perforated pipes through the chip piles in the summer would allow air to flow through the pipe, as well as into the pile, thus allowing the chips to dry out much faster then in a normal chip pile (Figure 5.2). This method would not be used in the winter since the build-up of heat is needed to start the seasoning process.

It was calculated that it would only take 1.5 weeks in the summer pile, using the regression equation, to reduce the extractives to a point where they equalled the extractive content of roundwood aged for 1 year (i.e., 2.5%). A minimum of emphasis should be placed on this 1.5 week figure, though, since the sample size used to calculate the roundwood's extractive content was quite small. A much larger sample would be required in order to be able to draw any firm conclusions as to the extractive content of the aged roundwood.

The measured basic wood density from the summer pile (0.41 g/cm^3) was in agreement with the findings of other studies. The rate of basic density loss calculated for this study was 2.4%/month. This is higher than the usual rule of thumb for softwoods of a loss of 1%/month.

The average moisture content of the fresh arriving chips was 47.9%, whereas the moisture content for the digester chips was 40.2%. This would be expected since the digester chips where comprised of a mixture of fresh chips and aged chips.

The fresh arriving chips had an average extractives content of 3.6%, whereas the digester chips had an extractives content of 2.9%. The average extractive content was on average higher in April (i.e., 3.8%) than it was in June (i.e., 3.2%). This trend is also seen in Levitin (1970) and Dunlop-Jones *et al.* (1991).

The average basic densities for both the fresh arriving chips and the digester chips were the same (i.e., 0.41 g/cm^3).

From the steaming portion of this study, it can be concluded that steaming does not yield quicker extractives losses than either seasoning of wood chips or roundwood. This study found that the longer the chips were steamed, the more the extractive content of the chips increased. This could possibly be due to minor degradation of the wood components, resulting in increased solubility with acetone or water.

This study managed to satisfy all but two, of the listed objectives. The study was able to determine the rates of moisture content, extractive content and basic density losses at different locations within a pile, as well as determine the temperature development within a chip pile built in different seasons (i.e., summer and winter). Data was obtained on the quality of fresh wood chips that were arriving at the mill, as well as on the chips that were entering the mill process. The study also determined that there were differences in seasoning of piles that were built in the summer and in the winter. The study was not able to show a relationship between steaming of the wood chips and shorter seasoning times or correlate operating problems in the kraft pulping process with the extractive content of the wood chips entering.

6.1 FURTHER STUDY

There are a number of areas found in this project that need more in depth study. These are :

1. More thorough chemical analysis of the changes in resins during the ageing process.
2. Pre-steaming the wood chips before they are piled in the winter to see if this aids in the initiation of the chemical changes within the pile.
3. More documentation at the mill in regards to pitch problems.
4. Further study on seasonal variation and its effect on moisture content and extractive content.
5. Further study is required to determine the mechanisms of physical and chemical changes involved during the steaming of wood chips.
6. Further study is required on the aeration of chip piles and its effect on seasoning times.
7. Evaluation of the loss of "good" wood (i.e., cellulose, hemicellulose, etc.) during seasoning.

6.2 RECOMMENDATIONS

A method of chip reclaim using a travelling auger under the pile and not a surface chip reclaimer is recommended. This method would succeed in taking only the bottom middle chips from the pile. The top chips in the pile would then fill in the openings. Bulldozers and front-end loaders would be needed to push the sides of the pile into the middle periodically. For maximum extractives loss a continuous seasoning time of 2 weeks is recommended. Travel on the pile by the bulldozers and front-end loaders should be kept to a minimum to prevent compaction and therefore better aeration of the pile. This chip reclaim procedure could also be used in the winter since the pile centre would always be hot and therefore the problem of frozen chips and no seasoning encountered in this study could be avoided. A chip pile consisting of a minimum of 42 308 m^3 would be sufficient to allow for a 2 week inventory of chips, however, a chip inventory of 3 weeks is recommended (i.e., 63 462 m^3 to allow for an extra weeks buffer).

It is also recommended that Avenor consider continuous monitoring of the digester extractive contents to know that they are sufficiently low, as well as to try and correlate any operating problems in the mill with the extractive content of the chips that had entered. The data presented in this study should be used to specify the storage period required.

Successful implementation of these recommendations will lead to a better understanding and decrease in the occurrence of pitch problems in the kraft mill which in turn will result in an increase in savings for the mill.

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