

MICROWAVE DRYING OF FLAX FIBRE AND STRAW AND STUDY OF THE STRAW'S USE IN A FIRELOG

BY

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ABSTRACT

Microwave-convective and conventional hot air drying of flax fibre and straw was compared at three drying temperatures (40°C, 60°C and 80°C). Microwave-convective drying ensured a 30% to 70 % reduction in drying time compared to hot air drying. Microwave-convective drying at 80°C was found to be most suitable method in terms of drying time. Drying curve fitting with different mathematical models was carried out. The colour change of the flax fibre and straw upon drying was measured and the brightness and colour change increased with an increase in drying temperature. For both drying processes, the tensile strength and modulus of elasticity of the flax fibre and straw increased with an increase in drying temperature.

The properties of firelogs mixed with different proportions of flax straw, sawdust and wax were assessed. A maximum burning temperature of 231°C was achieved by firelogs containing 60% sawdust and 40% wax binder, followed by firelogs with 70% flax straw and 30% wax (228° C). The burning rate rose with an increase in the percentage of wax, and was lowest (3.18 g min⁻¹) for firelogs containing 35% flax straw, 35% sawdust and 30% wax. Firelogs with only flax straw and wax produced the greater quantity of residues compared to sawdust-only firelogs. The maximum load applied and energy to break a firelog was greatest for 70% flax straw, 30% wax and this combination was considered as the most feasible flax-containing firelog.

Résumé

Le séchage micro-onde/convectif ou à air chaud (conventionnel) de fibres ou de paille de lin fut comparée à trois températures de séchage (40°C, 60°C et 80°C). Le séchage micro-onde/convectif assura une réduction de 30% à 70 % dans le temps de séchage des fibres ou de la paille de lin par rapport au séchage à l'air chaud. Un séchage micro-onde/convectif à 80°C s'avéra le meilleur procédé en terme du temps de séchage. Un ajustement de courbes de séchage à différentes modélisations mathématiques fut entreprise. Le changement de couleur des fibres et de la paille de lin advenant du séchage fut évalué. La clarté (L^*) et le changement de couleur augmentèrent avec une augmentation de la température de séchage. Pour les deux modes de séchage, la résistance à la traction et le module d'élasticité des fibres et de la paille de lin augmentèrent avec une augmentation de la température de séchage.

Les propriétés des bûches artificielles formées avec différentes proportions of de paille de lin, de brin de scie et d'agent liant (cire) furent évaluées. Une température de combustion maximale de 231°C fut atteinte par les bûches comportant 60% de brin de scie et 40% de cire, suivi par les bûches comportant 70% de paille de lin et 30% de cire (228° C). La rapidité de combustion augmenta avec l'augmentation de la proportion de cire. La combustion la plus lente (3.18 g min^{-1}) fut celle des bûches comportant 35% de paille de lin, 35% de brin de scie et 30% de cire. Les bûches comportant seulement de la paille de lin et de la cire produisirent la plus grande quantité de cendres, par rapport aux bûches comportant seulement du brin de scie et de la cire, qui en produisirent le moins. La charge maximale et l'énergie necessary pour briser une bûche fut maximum pour les bûches formés de paille de lin à 70% et de cire à 30%. Considérant tous les facteurs, cette bûche sembla la plus faisable du côté pratique.

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CONTRIBUTIONS OF THE AUTHORS

The work reported here was performed by the candidate and supervised by Dr. G.S.V Raghavan of the Department of Bioresource Engineering, Macdonald Campus of McGill University, Montreal. The entire research work was carried out at the Postharvest Technology laboratory and the work shop, Macdonald Campus of McGill University, Montreal.

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CHAPTER 1

GENERAL INTRODUCTION

According to Flax Council of Canada, Canada is the world's largest producer and exporter of flax (*Linum usitatissimum* L.). Flax seeds have been used as a food ingredient since the earliest ages. Fibres in the plant's stem were once extensively used in the production of linen textiles, but nowadays flax is mainly grown for oil production. Once the seed crop is harvested surface crop residues of flax straw remain in the field, but their long and tough stem fibres decay slowly, so farmers faced with the difficulty of incorporating flax straw into the soil after harvest either destroy the fibrous stems by burning them. More recently combines and straw choppers have been used to chop and spread the flax straw. The straw has also been used as animal bedding, lining for drainage ditches, horticultural mulch or as a fuel source in bale burners. Factories currently exist in Manitoba, Canada, which produce flax fibre for the production of specialty papers for cigarettes, currency, bibles, prayer books and art work books. There is also a flax fibre processing plant in Saskatchewan, where they extract flax fibre for use in specialty paper production. They produce fibre composites to replace the fibreglass presently used to make automotive parts like dashboards.

The flax fibre obtained after retting is of high moisture content of about 70% (w.b). In the wet fibre, the water molecules behaves like a plasticizer, hence the cellulose molecules move freely, which causes low elastic modulus and tensile strength. The decrease in the mechanical properties of the flax fibre may also be due to the development of fungi in the presence of moisture in the internal structure of the fibre (Stamboulis et al., 2001). Consequently, an effective and quick drying process is important in the production of bio-composites. Prior to the processing into a biocomposite material, the flax fibre must be dried properly. High temperature drying of flax fibre results in damage to its structure and colour. Microwave drying with a hot air supply could offer uniform heating, thus retaining the quality of the fibre. Microwave drying is a faster drying process than most conventional methods (Raghavan et al.,

2005). Hence an attempt was made to apply microwave drying to flax straw, evaluate its properties and compare them with conventionally dried samples.

Fireplaces are very common in North America. The problems regarding natural firewood are difficulty in storage due to irregular shape, lack of control over moisture content, difficulty in lighting, etc. To avoid these problems to a great extent, artificial firelogs have been introduced. These are mostly made from wood remains like saw dust, wood chips etc. Artificial fire logs are prepared by compressing a mixture of saw dust and binding agent in a high pressure in a mold of standard size. Artificial firelogs have a specific shape and are very easy to store, easily used in heating and leave less of ash behind (Houck et. al. 2000). The amount of binder can range from 0 to 60%, depending on the type of firelogs. Firelogs with minimum percentage of binder are very dense and difficult to light (De Hoop et al., 2005). In this study, firelogs were made by compressing flax straw, saw dust and wax in different proportions. Burning and tensile strength tests were conducted to find out the optimum percentage of mix for maximum performance.

CHAPTER 2

GENERAL OBJECTIVES

2.1. Problem statement

Flax is a major crop in Canada mainly grown nowadays for its seeds. While it was formerly used in the production of linen textiles, flax plant stems or flax straw, a by-product of growing this oil-crop, is now mainly left behind in the field as a crop residue, and later incorporated by ploughing. More recently some potential uses of the flax straw in terms of its fibres and by-products have led to renewed interest in this material. Drying is a major problem for the storage of flax straw and flax fibre must be dried for the production of bio-composites. Normal drying methods like sun drying and hot air drying are time-consuming, inefficient and uneven, adversely affecting the quality of the product. This problem led to the study of another quicker and more efficient method of drying flax fibre and straw at controlled temperatures.

Flax straw residues which are not being used for fibre production are usually burned or destroyed. These flax straw residues have a high calorific value, so they are ideal for use as a fuel source or in artificial firelogs. The thermal properties of flax straw will determine the combustion efficiency of firelogs made from flax straw.

2.2. Objectives

The added value and cost effectiveness of drying flax fibre and straw using an efficient microwave drying method were studied and the physical properties of the product (strength and colour) were compared to conventionally dried material. By converting the flax straw residues to artificial firelogs, an added value will be conferred to the raw flax straw residues found on harvested fields.

The objectives of the studies were:

- (i) Microwave-assisted drying of flax straw and fibre at controlled temperatures. This involves drying flax fibre and straw by using microwave energy, combined with

convective air and to compare the tensile strength and colour changes of the different samples.

Specifics:

- To establish an optimum product processing temperature for flax fibre and straw.
- To compare microwave-convective drying with hot air drying at the same temperatures.
- To study the tensile and colour profile of flax fibre and straw after various drying processes.

(ii) Evaluation of the properties (combustion, compression) of firelogs made with different proportions of flax straw, sawdust and wax binder.

- To produce fire logs with different proportions of flax straw, sawdust and wax mix.
- To analyse the burning rate, mean and maximum temperature and percentage weight of residues for each sample.
- To conduct puncture tests to find out the maximum load and energy to break the sample.

CHAPTER 3

REVIEW OF LITERATURE

3.1. Flax

Flax (*Linum usitatissimum* L.) is commonly used in food, textile and paper industry. The seed is used in food industry for making oil, cattle feed etc. The stem is used for the production of fibre, bio-composites, paper and many other industrial applications. Flax belongs to the family [Linaceae](#). Flax seeds were consumed as a cereal in the past and were known for their medicinal values. Flax grows in cold areas, with a mean height of 0.20 m to 1.50 m (Hegi, 1925). Flax seed oil has been used as a medium for food frying, in lamp oil and as a preservative material in paints (Vaisey-Genser and Morris, 1997).

There are different varieties of flax that are used in bio-composites, food and feed industries, depending on its properties. The height and growth of the plant depend on the variety, density, soil fertility and climatic conditions. Traditional flaxseed varieties used for human consumption contain large quantities of alpha-linolenic acid (ALA), the essential omega(ω)-3 fatty acid, which constitutes about 57 percent of the total fatty acids in flax. Flax varieties that contain less than 3% ALA are termed as Solin (Oomah and Mazza, 1998).

Studies on flaxseed as a source of alpha-linolenic acid proved that flax-seeds contain oil at 35% of its mass; out of that alpha-linolenic acid (omega-3 fatty acid) is 55% and linoleic acid is 15–18%. Flax-seed is a rich source of omega-3-fatty acids and the richest source of plant lignans (Carter, 1993).

During the research on health, nutrition and functionality of flax, Vaisey-Genser and Morris (1997) stated that flax seeds were consumed as a cereal in the past and were known for their medicinal values. The oil from the seed has been used as a medium for food frying, in lamp oil and as a preservative material in paints (Vaisey- Genser and Morris, 1997).

Lignans, also present in flax seed are phytoestrogens, compounds that are found naturally in plants and show weak estrogenic activity, i.e. the ability to bind to estrogen receptors on cell membranes (Zava et al., 1997).

3.2. Flax fibre

Flax is a dicotyledonous plant. Flax fibres are situated at the inner bark or the walls of phloem cells of the flax stem. The fibres are cellulose in the form of hollow tubes. The fibres are bundles of complex carbohydrates such as pectins, waxes and gums which support the plant. The hollow fibres transport photosynthesized materials from the leaves due to capillary action. Flax fibre is mainly used in the apparel industry for making clothes. The structure of the plant's stalk plays a pivotal role in determining the utility of flax for fabric (Atton, 1989; Judd, 1995).

The structure of flax is non-uniform and the semi cylindrical fibre becomes narrower towards the end (Grishanov et al., 2006; Moskaleva et al., 1981). In the wet fibre, water molecules behave as a plasticizer, allowing the cellulose molecules to move freely, and leading to their low elastic modulus and tensile strength. The decrease in mechanical property of the flax fibre may also be due to the development of fungi in the presence of moisture in the internal structure of the fibre (Stamboulis et al., 2001).

There are two major advantages of flax fibre when used in for linen cloth: one is that the yarn spun from flax fibre is more than twice as strong as cotton and the other is that the hollow flax fibres absorb moisture through wicking (Atton, 1989). Another advantage of hollow flax fibres is the ability to act as a sound proofing material (e.g. the lining of the interior of Roy Thomson Hall, a major concert hall in Toronto, Ontario (Atton, 1989).

Flax fibres are low in density, cheap, and biodegradable. The consistency in quality of the natural fibres is not the same when compared to synthetic fibres, which is one of the major disadvantages of flax fibre over synthetic fibres. The inconsistency of flax fibre may be due to various factors such as climate, the crop variety, the retting process and the equipment used in the process (Thomsen et al., 2006).

3.3. Physical properties of flax fibre

The forms of fibre that is present in flax-reinforced composite materials ranges from fibre bundles, technical fibres, elementary fibres, mesofibres and microfibres on the basis of diameter (Figure 3.1).

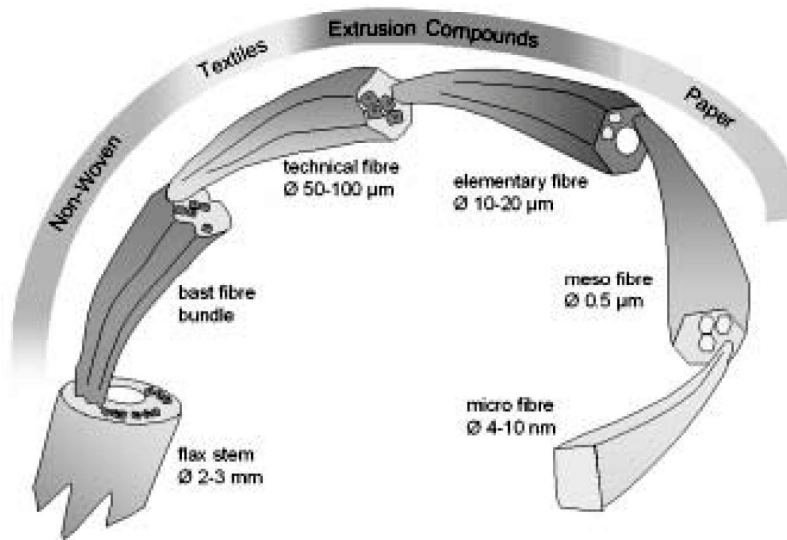


Figure3.1. Stem to microfibre (reproduced from Oever, M. J. A. et al., 1999)

Artificial fibres are very costly and not environment friendly. Natural fibres are as strong as artificial fibre and they are cheaper and friendlier to the environment. Given these characteristics, natural fibres are used in composites. The reinforcement of natural fibre in the bio-composite is comparable to that obtained with fiberglass, but its fibres are much lighter. The strength of the fibre is most important than fibre fineness in the production of composites (Norton et al., 2006).

Comparing the tensile strength of flax fibre with that E-glass (electrical grade glass), the tensile strength of E-glass was found to range between 2000 and 3000 MPa when compared to 345 to 1100 MPa for flax; however, their elastic moduli, λ , showed similar values: 12-85 GPa for flax, and 70 GPa for E-glass (Baley, 2002; Mohanty et al., 2000).

The strength and fineness of the fibre is also determined by the anatomy of the plant. The diameter of the cell and the thickness of the cell wall are the factors affecting the strength of the fibre. A smaller bundle diameter ensures finer fibres because of the

easy splitting process. Plants grown at a higher density cannot take up enough nutrients to grow further and will be branched, resulting in weaker fibres. The time of harvesting also affects the strength of the fibre: immature plants produce weaker fibre and over-mature plants produce stiffer fibre. The retting method also affects the strength of the fibre: longer retting times deteriorate the strength of the fibre. Enzymatic retting is found to be a better process in terms of the quality of the fibre produced, but this method is expensive as compared to other methods.

With the lessening use of linen in recent decades, flax straw first came to be considered a waste, but later producers recognized the potential of flax straw in different commercial applications. Totally unprocessed flax straw in square bales can be used as a simple building/insulation material for livestock wind-break structures, barn wall insulation and containment for feed bunks/grain storage piles. Flax straw in bales, or as loose flax straw, can be used as a geotextile for erosion control on slopes and in water courses, emergency erosion control on wind-swept barren ground, soil stabilization around livestock waters/feed bunks in feedlots, soil stabilization in oil-patch roads and during drilling site preparation and soil remediation/enhancement during road construction. Flax straw can be used as a heating source for yard sites, green houses and grain drying.

Flax straw after chopping or milling is termed ‘partially processed straw.’ These forms of straw can be used as a fibre/filler source in non-structural building materials like deck boards, paving stones, or roofing shingles. The extracted fibre can also be used in the production of speciality papers (for cigarettes, currency, etc). Partially processed flax straw is used as an additive to increase the strength of recycled paper pulp.

Fully processed flax straw can be used as a long lasting, durable and recyclable insulation, as an alternative to other non recyclable fibres. The water retention property of cottonized flax helps to increase the its functionality of feeling cooler to wear in hot environments (Payne, 2010)

3.4. Drying of flax fibre

The flax fibre obtained after retting is of high moisture content; however, the moisture content of flax fibres must be very low for the production of bio-composite materials (Ghazanfari et al., 2006). Sun drying was commonly used in drying natural fibres, but the lack of control of the drying conditions and longer drying times lead to poor fibre quality. Drying must be done before the production of bio-composites, otherwise the moisture in the fibre creates bubbles during the mixing process (Panigrahi et al., 2006).

Drying is a mass transfer process of removal of moisture from a material by means of heat. Drying occurs mainly at the surface and the movement of water from the inner portion of the material to the surface occurs due to the difference in the vapour pressure. The temperature of the process is a major factor in drying of natural fibre. High drying temperatures leads to a change in the physical and chemical properties of the fibre. Thermal degradation begins when the fibre is subjected to heat. Waxy compounds begin decomposing at 120°C and when the temperature reach 150 to 180°C, the hemicelluloses and pectin begin to decompose. At temperatures of 350 to 500°C, cellulose present in the fibre begins to decompose (Knothe and Folster, 1997). In general, the degradation of pure flax fibre begins at a temperature of 200-210°C and continues at temperatures up to 400°C (Powell et al., 2002). The main method of drying of flax straw is by passing hot air through the bales (Pereira et al., 2007).

3.5. Drying of flax straw

In Canada, the dominant regions producing flax are eastern Saskatchewan and Western Manitoba and these areas show a steady increase in the area of production. This increase in production leads to the necessity of proper management of the biomass produced. The producers find difficulty in managing the flax straw, and are forced to burn the crop residues. If flax straw is to be harvested for value added processing, the cutting height of the crop should be high, in order to minimize the loss of straw while harvesting the seeds (Anon, 2004).

The drying of flax straw is often carried in a traditional manner by spreading it in the field, changes in weather may affect the drying process. For storage purposes, the

excess moisture of flax hay bales is usually removed by hot air drying. Early experiments in the drying of rectangular flax hay bales were reported by Miller (1946).

Plue (1990) reported that the rate of air flow needed to dry baled hay was four times greater than that of loosely packed hay, because of the escape of supplied air from the loosely packed hay. Plue (1990) conducted his study using ambient air drying of large round hay bales, a process which proved to be both technically and economically acceptable. The application of microwave technology in the drying of flax straw will be beneficial in terms of quality and reduced duration of drying.

Antti (1995) conducted a study on microwave drying of pine (*Pinus* sp.) and spruce (*Picea* sp.) wood from green to 8% moisture content. The microwave power densities employed were 25 to 78 kW m⁻³ and the microwave energy consumption was between 365 to 760 kWh m⁻³.

3.6. Microwave drying

The study on microwave processing of foods revealed that the hot air drying method is very inefficient since the majority of the energy will be wasted in forcing air through the bale. The application of microwave to processing not only occurs in the food industry, but also in the drying of wood, paper, textiles and for the curing of resins in the plastic industry.

Microwaves are electromagnetic waves with short wavelengths in a frequency ranges of 300 MHz to 300 GHz. Microwave technology has been applied in food processing for drying, baking, sterilization, pasteurization, and the extraction of organic compounds. The microwave frequencies used for industrial scientific and medical purposes are known as ISM bands: 433 MHz, 915 MHz and 2450 MHz (Orsat et al., 2005).

Microwave assisted drying is a better processing method for natural composites than hot air drying because of the shorter lesser drying time, and the better mechanical and thermal properties. The practical difficulty of achieving even and controlled heating of relatively large samples is the main limitation of microwave drying (Sgriecchia and Hawley, 2007).

Microwave drying is a faster way of drying than conventional methods (Raghavan et al., 2005) because microwaves penetrated the material to be heated and volumetric rather than surface heating occurs. When microwave energy is applied to a material, the microwave energy begins oscillating water and other polar molecules in the material. This oscillation cause friction and hence heat is developed. The intensity of the heat depends up on the frequency of the microwave, strength of the electromagnetic field and the dielectric properties of the heated material (Beaudry et al., 2003).

Conventional drying combined with microwave drying accelerates the drying process because of the removal of surface moisture developed due to the increase in the internal pressure. It is experimentally proven that microwave drying is a faster way of drying than conventional methods from the experiments conducted on microwave drying of various agricultural commodities (Raghavan et al., 2005).

3.7. Microwave Generation

A microwave generator consists of three basic parts: the magnetron, wave guide and applicator. A cathode filament located at the centre of the magnetron is surrounded by a hollow iron cylinder with an even number of vanes, which acts as an anode. The output frequency of the microwave is controlled by the area between the vanes in the magnetron (Orsat et al., 2005). The schematic diagram of a magnetron is shown in Figure 3.2

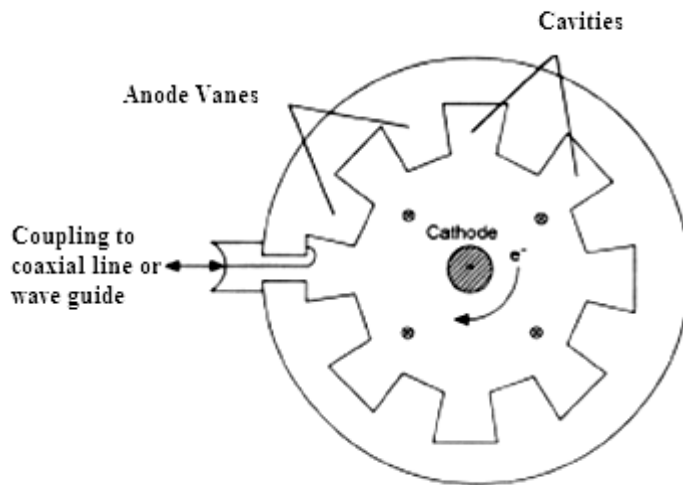


Figure 3.2. Schematic diagram of a magnetron tube (Source: Anonymous, 2007)

The magnetic field inside the magnetron tube is provided by strong permanent magnets situated in the magnetron. The electric and magnetic fields inside the magnetron tube influence the electrons move towards the anode from the cathode. The magnetic field is perpendicular to the direction of movement of the electrons, so the electrons tend to move in a curved path rather than a straight line. These spiral rotating electrons transfer their energy into a wave guide due to their interaction with the resonant cavities (Orsat et al., 2007)

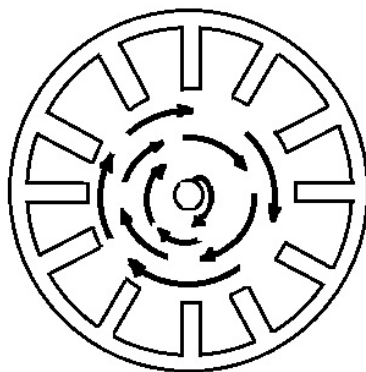


Figure 3.3. The curved path of electron in the magnetron due to the influence of electric and magnetic field (Source: Anonymous, 2007)

The waveguides are generally rectangular-shaped copper pipes which guide the microwaves from magnetron to the site of their application. Wave guide consists of tuners which match the load impedance to the waveguide impedance and there by minimize the reflected power to ensure minimal loss due to reflection (Orsat et al., 2005).

Torrington et al., (1996) conducting a study on the microwave puffing of vegetables found that microwave-convective drying, in which convection drying followed by microwave drying, produced better quality product thanks to a higher drying rate and lower drying time.

In the present study, the drying of flax fibres and flax straw was done using a microwave apparatus designed in the post harvest technology lab, Macdonald Campus, McGill University. The microwave generator operated at 2450 MHz with a variable power setting of 0 to 750 kW. The temperature of the flax was measured with the help of an optical fibre probe (Nortech EMI-TS series, Quebec City, Canada). The temperature probes were connected to an Agilent 34970A data acquisition unit and that unit was connected to a computer (Dev et al., 2007).

Experiments on drying of nettle (*Urtica* sp.) leaves by microwave energy showed a reduction in drying time with an increase in the power level (Alibas, 2007). The greater energy efficiency of microwaves when compared to other conventional method was also reported. The energy is absorbed by the material, the heat dissipates and the material then dries. The internal heat develops an internal pressure which helps water to come out to the surface by diffusion. The surface water is then removed by the air flow (Alibas, 2007).

3.8. Drying characteristics

The drying of biological products takes place in 2 different phases, a falling rate drying phase and a constant rate drying phase. First phase of drying occurs at a declining rate which parallels a sudden decrease in the moisture content, attributable to the removal of loosely bounded top layer moisture from the product due to capillary action and partial pressure differences. In the falling rate drying period, moisture

removal is very slow and occurs at a steady rate, due to the difficulty in removing tightly bound water molecules from the product (Raghavan et al., 1995).

In hot air drying, the moisture removal happens from the surface first, and then the water inside the product moves out due to the diffusion. But in microwave drying, heating is done volumetrically which results in a high internal temperature. As a result, the vapour pressure increases which aids in the movement of water to the surface. This results in higher drying rates under microwave (vs. hot air) drying (Sunjka et al., 2004).

3.9. Mathematical modeling

Mathematical modeling of thin layer solar drying of shelled and unshelled pistachios were conducted by Midilli and Kucuk (2003). Experimental data from eight different models of both thin layer forced air and natural solar drying processes was best described by a logarithmic drying model.

A semi theoretical thin layer drying model was developed by Hii et al. (2008) to describe air drying of cocoa beans with overnight tampering. A combination of Page and a two-term drying model were used. The model fitted best in the temperature range of 60-80° C. Beans dried at 60° C showed lower acidity and better flavour compared to drying at 70°C or 80° C (Hii et al., 2008).

Studies on thin-layer drying of moist flax fibre were conducted at 30°C, 50°C, 70°C, and 100°C at a constant absolute humidity of 0.0065 kg water per kg dry air. Five term solutions of Fick's Second Law of diffusion were used in modeling the drying of fibres under different drying conditions. The drying process was underestimated by the model in the initial stages and overestimated in the final stages. The coefficient of diffusion estimated by using the equations increased linearly with the drying air temperature (Ghazanfari et al., 2006).

Fruit materials have been dehydrated by combined microwave and vacuum drying methods to produce high quality products. The knowledge of the drying kinetics of fruit products has allowed for the design of efficient dehydration systems. Drouzas et al., (1999) studying the drying of orange juice, used a laboratory microwave vacuum drier in drying kinetics experiments with model fruit gels. The operating conditions were

3000 to 5000 Pa and microwave power was 640 to 710 W. Drying rate was used to determine the distribution of the electromagnetic field in the cavity of the microwave oven and the drying rate was determined by weighing the samples in regular intervals. The rate constant (K) was determined by regression analysis of the experimental data and found to increase with an increase in microwave power level (Drouzas et al., 1999).

3.10. Effect of microwave heating on tensile strength and colour of flax

Muller and Krobjilowsky (2004) conducted studies on the effect of temperature on the impact strength of flax fibre reinforced composite material. An increase in strength occurred with an increase in the processing temperature (Mueller and Krobjilowsky, 2003). Flax fibres used in the composites are called technical fibres or fibre bundle. The fibre bundle or technical fibre has low mechanical properties as compared to elementary fibres of lower diameter. The tensile strength values of fibre bundles is around 600–700 MPa , with a Young's modulus of 50–60 GPa (Singleton et al., 2003).

The tensile strength of sisal (*Agave sisalana* Perrine) fibre was measured by using an Instron apparatus (Yuan et al., 2001). The diameter of the single fibre was measured using a Nikon Profile microscope. The area of the fibre was calculated by assuming the fibre to be cylindrical in shape (Yuan et al., 2001).

The color change of flax fibres which are dried by microwave is related to browning brought on by a Maillard reaction, the reaction between an amino acid and a reducing sugar at elevated temperatures. Like caramelization, it is a form of non-enzymatic browning. The reactive carbonyl group of the sugar reacts with the nucleophilic amino group of the amino acid, and forms a variety of interesting but poorly characterized molecules responsible for a range of odours and flavours. (Glomb and Monnier, 1995).

3.11. Compression of flax straw

De Hoop et al. (2005) investigated the production of sawdust firelogs with an agriculture-based binder (soybean wax) and studied the firelog's emission properties. A firelog press was made by modifying a 20 Mg log splitter. Samples with the desired sawdust-wax proportions were placed inside the cylinder and the cylinder heated to 84°C in an oven to melt the soybean wax. The samples were then compressed until the wax squeezed out between the piston and cylinder (De Hoop et al., 2005).

Shaw and Tabil (2005) investigated the handling characteristics of densified biomass with respect to improving its transportation and storage properties. Mechanical properties of biomass are essential in designing a suitable system for compression of biomass. Coefficients of wall friction, adhesion and the asymptotic modulus of peat moss, wheat straw, oat hulls and flax shives were studied. The calorific values of all the samples were calculated. Flax shives showed the highest calorific value (17.71 MJ kg⁻¹) among the samples tested.

Mankowski and Kolodziej (2008) conducted studies seeking to increase the heat of combustion of briquettes made of hemp (*Cannabis sativa* L. subsp. *sativa* var. *sativa*) shives. The lower specific gravity and bulky nature of flax straw leads to problems with its storage, transportation and burning. Flax takes up much more space in storage, but if it is exposed to rain and wind it deteriorates (Mankowski and Kolodziej 2008).

Agricultural wastes made into fuel source briquettes can act as an alternative source of heat, where solid wood resources are unavailable. Wamukonya and Jenkins (1995) conducted studies on the possibility of producing briquettes from household wheat straw and sawdust, which developed into a small-scale industry in Kenya. Burning of the raw materials was thus avoided. Evaluating briquette length expansion over a week period, they found sawdust briquettes to show the least expansion and greatest durability. Wheat straw briquettes showed the least durability and greatest expansion. In the wheat straw sawdust combination, the length of expansion of the briquettes and their durability was related to each other. Briquettes without binder burned poorly (Wamukonya and Jenkins, 1995).

The most common fire logs are made from sawdust and wax, with 40% to 60% of the material being wax and the rest sawdust. The wax used in artificial fire logs is obtained from petroleum industry. Artificial fire logs bear greater heat content (15,700 Btu lb⁻¹) [convert to SI units] than cordwood (8900 Btu lb⁻¹ for Douglas fire). The moisture content of artificial firelogs is much lower than that of cordwood.

Firelogs can be classified into two main types: (i) densified fire logs and (ii) wax-sawdust firelogs. Densified fire logs are not easy to ignite as there is no binder (wax) in them. The maximum temperature attained while burning a fire log is positively related to the wax content (Houck et al., 2000).

Lehtikangas (2001) conducted experiments on pellets made from fresh and stored sawdust, bark and logging residues to assess their moisture content, heating value and ash content. Dimensions and bulk density were also measured. Pellets from logging residues had the highest heating value and ash content, while bark pellets showed highest durability, and sawdust showed the minimum. Ash content from sawdust pellets was lower than from logging residue or bark pellets.

3.12. Fire logs from a flax straw-saw dust-wax mix and its properties

The burning process can be explained in five different steps, which are heating, decomposition, ignition, combustion and propagation (Pearce et al., 1990). An evaluation of the mechanism of ignition and combustion of materials in a heat flow under a variety of practical conditions is possible under laboratory conditions by simulation tests. The change in the combustion rate of wood with time and the change in the mass velocity of combustion with time were analysed. The rate of combustion at the moment of ignition was found to be maximum, then decreasing as the sample burned out. The decrease in temperature was due to the increase in thermal resistance of the carbon layer formed as the sample burned out, and minimised the rate of heat transfer by conduction from the burning surface to the interior of the burning sample (Abduragimov et al., 1986).

Using a piston and press, Chin and Siddiqui (2000) compressed agricultural materials like sawdust, rice husks, peanut shells, coconut fibres and palm fruit fibres at pressures of 5 to 7 MPa in order to study their properties. They assessed the relaxation behaviour of the samples, their mechanical strength and burning characteristics. Among

the samples, sawdust showed the best handling properties. The combustion rate of the sawdust briquettes was found to increase with the level of binder, and to decrease with an increase in the die pressure.

Wang et al. (2009) studied the combustion behaviour under different heating conditions of agricultural biomass alone and blended with coal. Bituminite coal, aspen sawdust and wheat straw were analysed thermogravimetrically individually and in blends and the mixtures behaviours assessed. Wheat straw acted as a substitute for sawdust in terms of heat release by adding 5% by weight of bituminite.

Joshi et al. (1989) conducted studies on thermal parameters of different stove-fuels: wood, crop residues and dung cakes. The maximum heating efficiency was obtained with wood because of its high calorific content.

Studies on cell wall formation and maturation in flax bast fibres, confirmed the presence of lignin in bast fibres (Baley, 2002; Day, 2005). Dry cell wall residues of flax bast fibres bear 1.5% to 4.2% lignin, while flax xylem tissues bear 23.7% to 31.4%. Flax straw contains lignin in the fibres since the polymer is involved in its composition. Lignin acts as a bonding agent to hold the fibres together.

Studies on the influence of surface roughness of paraffin wax surfaces on the static and dynamic contact angle hysteresis were conducted at 20°C using the Wilhelmy method, with water, ethylene glycol as well as ethanol as test liquids. The wax used had a melting point range of 68 to 72° C (Kamusewitz et al., 1999).

Puncture tests (12-mm-diameter puncture probe) were conducted using an Instron materials testing device (Instron 4502, Boston) to measure the texture of potato tubers (Ranganna et al., 1997). The maximum load on the sample and energy to break samples were calculated (Ranganna et al., 1997).

This chapter reviewed the physical properties of flax fibre and straw which are important factors with respect to its potential uses. Drying of flax fibre and straw was further discussed with regard to the optimum temperature for maximum quality. An understanding of microwave generation and microwave drying were also essential to proceed with the microwave drying of flax fibre and straw. In the following chapter

various mathematical models were fitted to drying curves for flax fibres. The tensile strength of fibres, compression of flax straw and the production of firelogs from flax straw are also discussed in this chapter.

CHAPTER 4

MICROWAVE-ASSISTED DRYING OF FLAX STRAW AND FIBRE AT CONTROLLED TEMPERATURES

Abstract

Flax fibres and stems were subjected to microwave drying at controlled temperatures. The rate of drying was then compared with conventional hot air drying. The product temperature was maintained at 40°C, 60°C or 80°C for both microwave and hot air drying. The initial moisture content of flax fibre was about 60% (wet basis); while for flax stem, it was about 70% (wet basis). The microwave drying was conducted in a microwave apparatus which recorded mass, product temperature, incident microwave power, reflected microwave power and inlet/outlet air temperature. The final moisture content for both experiments was set to 9% (wet basis). Microwave-convective drying ensured about 30 to 70% reduction of drying time for drying flax fibre and straw as compared to hot air drying. Curve fitting with different mathematical models were carried out. While a significant difference in colorimeter-assessed colour existed between microwave-convective dried flax fibre and hot air dried flax fibre, such a difference did not exist for flax straw. The tensile strength of flax fibre and straw, measured with an Instron apparatus, increased with an increase in the processing temperature of both processes. Hot air dried flax fibre and straw showed the greatest tensile strength and modulus of elasticity at processing temperatures of 60°C and 80°C.

Key words: microwave, flax straw, flax fibre.

4.1. INTRODUCTION

Flax (*Linum usitatissimum* L.) is used in various food and industrial products. The seeds are used in the food industry for making oil, cattle feed etc. The stem is used for the production of fibre, bio composites, high quality paper and many other industrial applications. Flax plant belongs to the family Linaceae. Flax seeds were consumed as a cereal in the past and were known for their medicinal values. The oil from the flax seed has been used as a medium for food frying, in lamp oil and as a preservative material in

paints (Vaisey-Genser and Morris, 1997). Flax seeds 35% oil by mass, of which α -linolenic acid (an ω -3 fatty acid) represents 55% and linoleic acid represents 15–18%. Flax-seed is a rich source of ω -3-fatty acids and the richest source of plant lignans (Carter, 1993). The flax seeds are primarily used in the food industry, while the plant stems are used in fibre production. The stem height of flax plants range from 0.20 m to 1.50 m (Hegi, 1925). The flax stems contain cellulose fibres (bast fibres) which have a number of mechanical and physio-chemical properties that are useful in textile and biocomposite industries. Flax fibres, which originate from renewable resources, are used as an alternative to mineral fibres. Their low cost together with their lower density, higher specific stiffness and recyclability are the major incentives for their use in composite materials (Baley 2002). Flax fibre is biodegradable, environmentally friendly, renewable, non-abrasive, economical, and easily available in Western Canada (Ghazanfari et al., 2006).

Flax fibre for the textile industry, production of bio-composite materials and the paper industry are produced from flax straw. Drying of flax straw is essential for maintaining its quality during storage. Commonly, natural fibres are dried under sunlight, but this process takes a long time and drying conditions are not easy to control. The quality of fibre by this drying method is frequently not optimal.

The low specific gravity and bulky nature of flax straw lead to problems with its storage, transportation and burning. Flax requires a great deal of storage space; however if it remains outdoors or otherwise exposed to the environment, it loses its functional properties (Mankowski and Kolodziej, 2008). The main method of drying of flax straw is by passing hot air through the bales (Pereira et al., 2007). The hot air drying method is very inefficient since the major part of the energy will be wasted in forcing the air through the bale. Microwave drying is a faster way of drying than most conventional methods (Raghavan et al., 2005); therefore, microwave drying of flax straw was tested and compared with hot air drying.

The flax fibre obtained after retting is of high moisture content. The moisture content of flax fibres should be very low for the production of biocomposite materials. In wet fibre, the water molecules behave like a plasticizer, hence the cellulose molecules

move freely, leading to a low elastic modulus and tensile strength. Decreases in the mechanical property of the flax fibre may also be due to the development of fungi in the presence of moisture in the internal structure of the fibre (Stamboulis et al., 2001). Therefore, the development of an effective quick drying method is considered to be important in the production of bio-composites. Drying is necessary for flax straw and fibre because the water contained inside the fibre forms bubbles during the mixing process involved in making biocomposite materials (Panigrahi et al., 2006). Hence, prior to the processing into a biocomposite material, the flax fibre must be dried properly. High temperature drying of flax fibre results in damage to its structure and colour. Microwave drying with a hot air supply could offer uniform heating and retain the quality of the fibre. The objectives of this study were:

- i. To establish an optimum product processing temperature for flax fibres and straw.
- ii. To compare the microwave-convective drying with hot air drying at the same temperatures.
- iii. To study the tensile and colour of flax fibres and straw generated through different drying processes.

4.2. MATERIALS AND METHODS

The material and methods used for the study are discussed in this section.

4.2.1. Initial moisture content

Initial moisture content of the flax straw and fibre on a wet basis (w.b.) were measured by drying 25 g samples in a hot air oven for 24 hours at 105°C, and calculating moisture content (M.C.) of the flax fibre or straw (ASAE S358.2 Dec 93):

$$\text{M.C. (wet basis)} = \frac{M_w}{M_w + M_s} \quad (4.1)$$

where,

M_w is the mass of water in the sample (g),

M_s is the mass of solid in the sample (g)

4.2.2. Sources of flax straw and fibre

The flax straw used in all experiments was grown in a McGill University greenhouse under controlled conditions. The flax seeds of brown variety from local suppliers in Montreal were sown in pots of 24 cm diameter with a volume of 0.006 m³. The number of seeds in the pots was limited to a maximum of eight and the pots were filled with soil and organic manure mix. The flax plants were harvested after 100 days and kept in the green house for 2 weeks for drying. The moisture content of the flax stems was 3.93% w.b.

Flax fibres used for microwave drying were purchased from a local market in Humboldt, Saskatchewan, Canada. The fibre had a moisture content of 4.22% w.b.

4.2.3. Microwave Apparatus

The drying of flax fibre and flax straw was performed by using a microwave apparatus designed in the post harvest technology lab, Macdonald Campus, McGill University (Figure 4.1). The microwave generator operated at 2450 MHz with a variable power from 0 to 750 kW. The temperature of the flax was measured with the help of an optical fibre probe (Nortech EMI-TS series, Quebec City, Canada). The temperature probes were connected to an Agilent 34970A data acquisition unit and that unit was connected to a computer (Dev et al., 2007). An adjustable hot air supply was attached to the microwave oven to pass hot air through the microwave oven to remove the moisture generated by the samples.

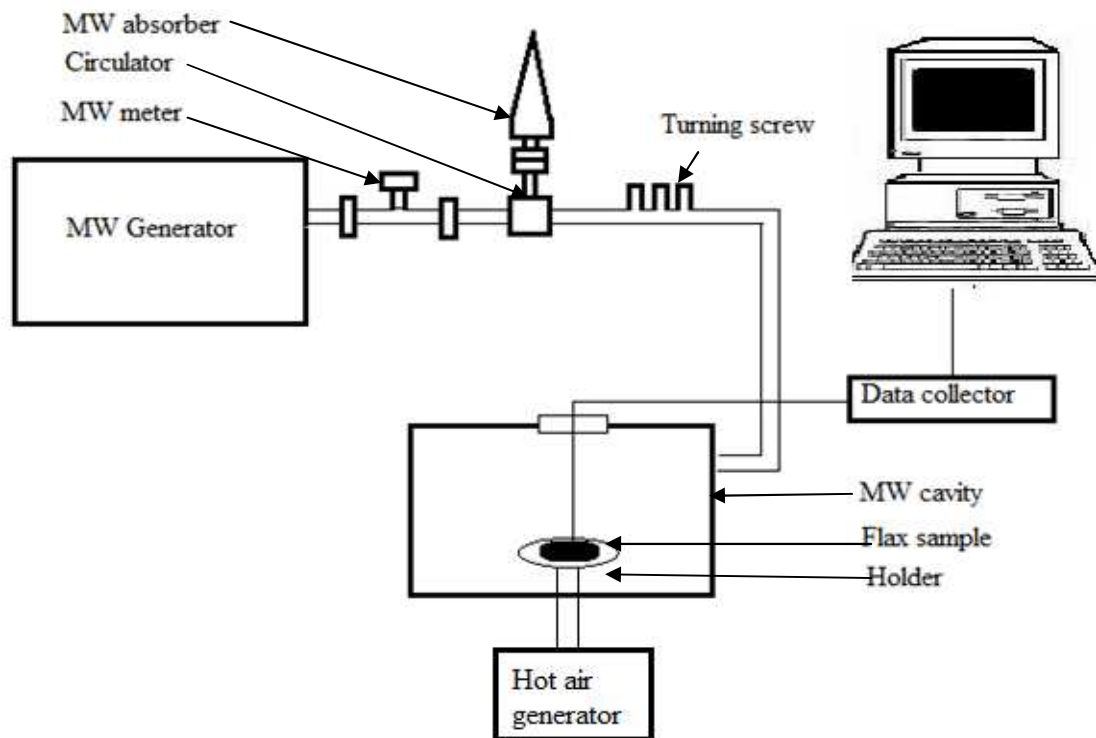


Figure 4.1. Diagram of microwave drying apparatus (Adopted from Dev et al., 2007).

4.2.4. Experimental design

The flax fibre or straw was dried by either microwave-convective drying method or a hot air drying method, each operating at 40°C, 60°C or 80° C. The drying characteristics were recorded and analysed. Three replicates of each test were conducted. The tensile strength, elastic modulus and colour of the dried material were tested for all materials and each set of processing conditions.

4.2.5. Microwave drying of flax fibre

Samples of flax fibre (25 g) bearing an initial moisture content of 4.2% w.b. were kept in a glass jar of volume 0.002 m³ filled with tap water at room temperature for 48 hours. The samples were taken out and the excess water was removed by using a manually-operated centrifugal rotator (salad spinner). Initial moisture content was 60% w.b.. The drying temperatures were set at 40°C, 60°C and 80°C. Air flow of 40°C, 60°C

and 80°C from an air blower was ensured in the microwave oven for the removal of moisture from the samples during the experiment. Throughout drying experiments the microwave reflectance was controlled manually with tuners. The sample temperature, microwave reflectance and sample mass were recorded by the computer at intervals of 30 seconds. The maximum incident power and the maximum reflected power were kept at 100 W and 80 W respectively for all the experiments. The drying was conducted until the material reached a final moisture content of 9% w.b.. Three replicates were done for each test.

4.2.6. Microwave drying of flax straw

Flax straw was cut into 0.15 m lengths. The middle part of the plant stem was chosen for the experiment to ensure uniformity in carrying out drying tests. Flax straw samples (25 g) were placed in a jar full of water for 48 hours at room temperature to ensure fully wet conditions. The wetted samples (68-70% w.b.) had their surface water removed using a manually-operated centrifugal rotator (salad spinner). The samples were then weighed, and transferred to the microwave apparatus. The microwave drying was done at a temperature of 40°C, 60°C or 80°C. While drying, the microwave reflectance was manually controlled using tuners. The temperature, reflectance and mass were recorded by the computer at intervals of 30 seconds. The maximum incident power and the maximum reflected power were kept at 100 W and 80 W respectively for all the experiments. The drying was conducted till it reached a final moisture content of 9 % (wet basis). Three replicates of each test were done.

4.2.7. Hot air drying of flax straw and flax fibre

The same procedure for the sample preparation as per microwave drying was followed. The experiments were done with different temperatures of convective air at 40°C, 60°C and 80°C without microwave incidence inside the same microwave apparatus, with the microwaves off. The mass and product temperature were noted at 30 second intervals.

4.2.8. Mathematical modeling of drying of flax fibre and straw

After collecting the drying data, the drying kinetics were studied on the basis on the following mathematical models on thin layer drying (Ghazanfari et al., 2006).

Table 4.1. Mathematical models used for drying of flax fibre and straw

Model name	Equation	Reference	Equation No.
Newton	$MR = e^{-kt}$	Ayensu (1997)	(4.2)
Page	$MR = e^{-kt^n}$	Karathanos and Belessiotis (1999)	(4.3)
Henderson & Pabis	$MR = a e^{-kt}$	Akpinar et al. (2003)	(4.4)
Modified Page	$MR = e^{(-kt)^n}$	Diamante and Munro (1993)	(4.5)
Logarithmic	$MR = a e^{-kt^n} + c$	Yaldis et al. (2001)	(4.6)
Wang and Singh	$MR = 1 + at + bt^2$	Midilli and Kucuk (2003)	(4.7)

where,

k is the drying rate constant (min^{-1})

n, a, b and c are drying coefficients (unitless) that have different values depending on the equation and the drying curve

t is time (min), and

MR is the moisture ratio, defined as $\frac{M_i - M_e}{M_0 - M_e}$, where M_i , M_0 and M_e are

the moisture contents at time t_i , initial moisture content and equilibrium moisture content, respectively.

However, given that M_e is negligible in both microwave and convective drying (Midili and Kucuk, 2003), the equation for MR reduces to $MR = M_i / M_0$.

Statistical parameters such as the root mean square error (Eq. 4.8). Chi-square (Eq. 4.9) and coefficient of determination R^2 ; (Eq. 4.10) are used to assess the closeness of fit of model curves (Hii et al., 2009).

$$RMSE = \sqrt{\frac{1}{n} \sum_{i=1}^{i=n} (MR_i^{pred} - MR_i^{exp})^2} \quad (4.8)$$

where,

n is the number of observations,

MR_i^{pred} is the predicted MR at time, t_i , and

MR_i^{exp} is the experimentally derived (or measured) value of MR at time t_i

$$\chi^2 = \sum_{i=1}^{i=n} \frac{(MR_i^{pred} - MR_i^{exp})^2}{n - m} \quad (4.9)$$

where,

χ^2 is the chi-squared statistic, and

m is the number of constants in the drying models.

$$R^2 = 1 - \frac{\sum_{i=1}^{i=n} (MR_i^{exp} - MR_i^{pred})^2}{\sum_{i=1}^{i=n} (MR_i^{exp} - \overline{MR}^{exp})^2} \quad (4.10)$$

where,

$\overline{MR}^{\text{exp}}$ is the mean experimentally derived (measured) of MR

4.2.9. Tensile strength of flax fibre and straw

Tensile strength of flax fibre and straw were measured with a tensile testing machine (Instron – 4502, Instron Corporation, USA) controlled by a computer software (Instron series IX, version 8.25). The samples were fixed with two clamps attached to the crosshead and platform, and crosshead set to move at a speed of 10 mm per minute. The flax fibre and straw, obtained under different drying conditions, were tested for their tensile strength and compared (Yuan et al., 2001).

4.2.10. Diameter measurement

The diameter of flax fibre samples, in μm , was measured by using a standard microscope (Micromaster, Fisher Scientific, Canada) with a graduated eye piece. The flax fibre samples were maintained straight between two glass plates and placed on microscope to measure its diameter. Given that the diameter of the flax fibre was not uniform along the length of the sample, 5 readings were taken along its length and averaged. This diameter was used in the calculation of its tensile strength.

4.2.11. Tensile strength of flax fibre

Five samples of flax fibres were randomly selected from the dried samples and each tested three times. For the tensile strength test (Figure 4.2), the desired length of the fibre was 75 mm, with a gauge length of 50 mm.

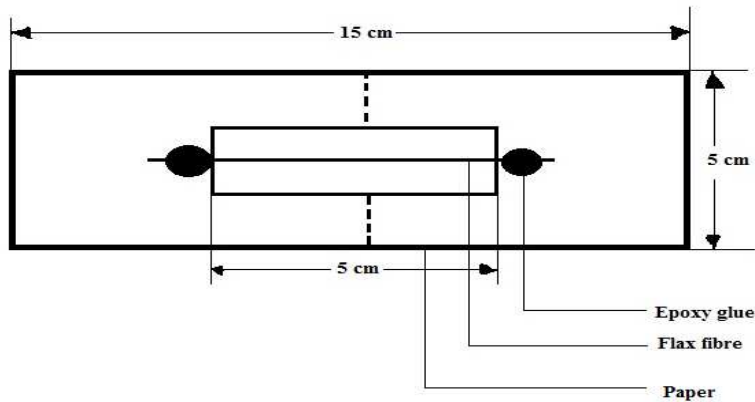


Figure 4.2. Flax fibre for tensile test

The fibre was fixed on a 50 mm × 10 mm sheet of paper with epoxy, so as to maintain the fibre in a straight and extended position (Figure 4,2). The flax fibre attached to the paper was mounted on the tensile test machine (Figure 4.3), the top end being attached to the machine with the help of a high-grip clip and the other end being connected to the chuck at the bottom. After mounting on the machine, the edges of the paper were carefully cut away and force applied in tension until the fibre broke into two. The force and displacement were recorded on an attached computer. Five sets of experiments were done, each with three replicates. The experiments were repeated for both the microwave and hot air dried samples and the results compared.

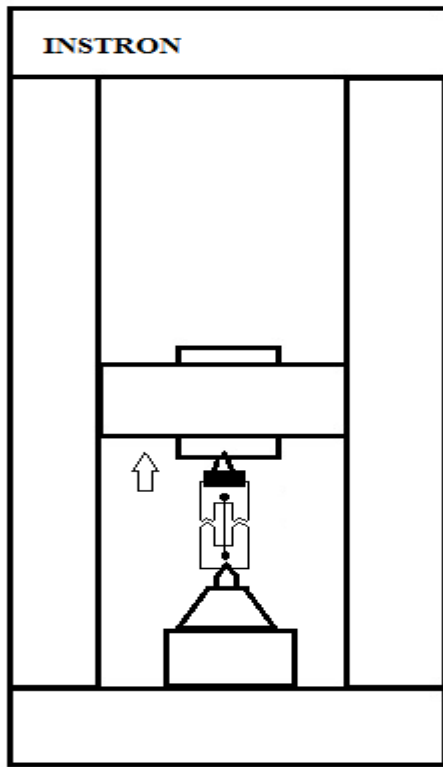


Figure 4.3. The tensile strength test of flax fibre

The tensile test of flax straw was conducted using the Instron tensile test machine only (no paper strip). Dried flax stems were taken and cut into 90 mm lengths and both the ends were inserted into small teflon tubes 20 mm in length and 3 mm in inner diameter and 5 mm in outer diameter. The flax straws were fixed in the tube with the help of a glue gun and allowed to set before conducting the experiments. The gauge length was 5 cm for all the samples. The samples prepared for the tensile strength test of flax straw is shown in Figure 4.4.

The prepared samples were mounted on the Instron tensile test machine (Figure 4.5) and the tension force applied until the sample broke into two. The force and displacement were recorded in the computer attached to the tensile strength machine. Five sets of experiments were done for each of the three replicates. The experiments were repeated for both the microwave and hot air dried samples and the results compared.

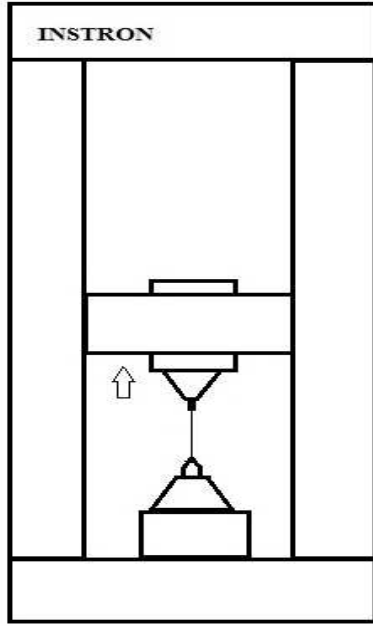


Figure 4.4. Flax stem sample

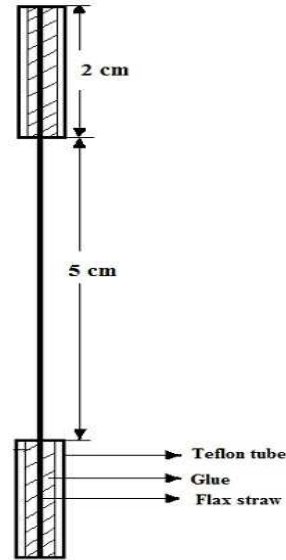


Figure 4.5. Tensile test of flax stem

The tensile strength of the flax fibre and straw were calculated using the equation:

$$\sigma_t = \frac{F_{\max}}{A} \quad (4.11)$$

where,

σ_t is the tensile strength in N mm^{-2} ,

F_{\max} is the maximum force applied (N), and

A is the cross sectional area of the fibre (mm^2)

The modulus of elasticity (E) was then calculated as:

$$E = \frac{\sigma_t}{\varepsilon} \quad (4.12)$$

where,

ε is the tensile strain which is a dimensionless ratio of change in length and original length .

4.2.12. Colorimetric test of flax fibre and straw

Changes in the colour of flax fibres and straw after microwave or hot air drying with respect non-dried samples were compared. Colour was assessed within the CIE 1976 L*, a*, b* colour space (CIE, 2007) using a tristimulus colorimeter (Minolta Co. Ltd., Japan). Colour values, expressed as L* (whiteness or brightness/darkness), a* (redness/greenness) and b* (yellowness/blueness) were determined for all samples.

Forty millimeter thick flax fibre or straw samples were placed on a table, and the L*, a*, b* values measured by pressing the measuring head on the top of the samples. Three replicates of each sample were measured. Non-treated flax fibre and straw samples were taken as standards and colour change after the drying of the flax fibre and straw was calculated with respect to the standards. Colour difference values of ΔL^* , Δa^* and Δb^* were calculated by subtracting the respective standard colour values from the measured values for each set of dried experimental samples.

The target colours in this experiment are L, a, and b of the non dried flax fibre and straw. The total colour difference ΔE is measured as (Minolta, 1991):

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4.13)$$

4.2.13. Statistical analysis

The statistical analysis of variance (ANOVA) of the samples was performed to assess which treatments had significant ($P \leq 0.05$) effects on measured parameters using Statistical Analysis Software (SAS 9.2 SAS Institute Inc., Cary, NC, USA). Means were compared using Duncan's Multiple Range test ($P \leq 0.05$).

4.3. RESULTS AND DISCUSSION

4.3.1. Flax fibre

The microwave drying of flax fibre at different temperatures 40° C, 60° C and 80° C are shown in Figure 4.6.

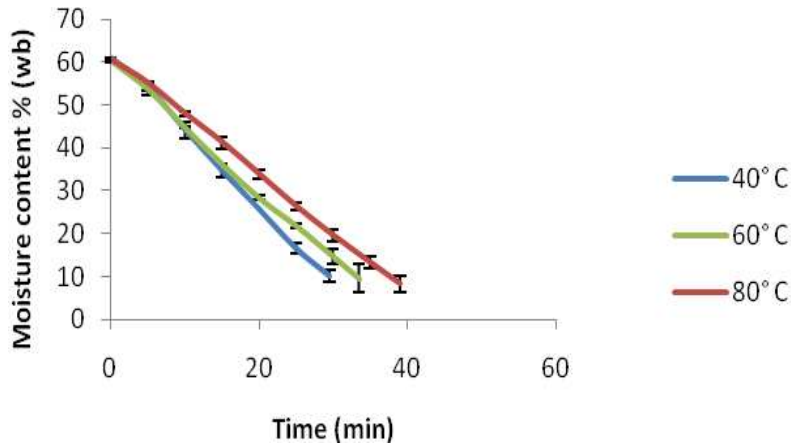


Figure 4.6. The temporal variation of moisture content during microwave drying flax fibre at 40° C, 60° C and 80° C

The microwave drying of flax fibre at 80°C, 60°C and 40°C took 28.5, 34.0 and 37 min, respectively, to reach a final moisture content of 9% w.b. from 60% w.b. Thus drying time decreased with increasing drying temperature. At 40°C, 60°C and 80°C microwave drying took, respectively, 70.4%, 55.6% and 59.6% less time to dry flax fibre than hot air drying (Figures 4.7, 4.8 and 4.9, respectively). Microwave power heated the flax fibre sample volumetrically and the surface moisture was removed by convective air simultaneously (Alibas, 2007), consequently, flax fibre samples dried more quickly under microwave- convective drying than hot air drying.

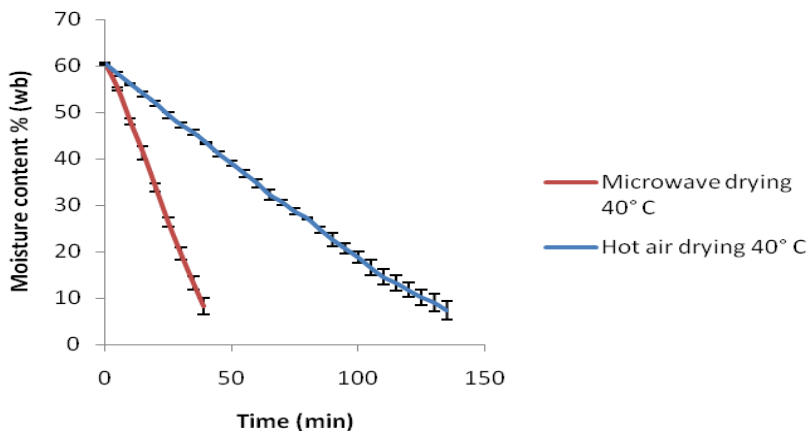


Figure 4.7. Comparison of microwave and hot air drying of flax fiber at 40° C

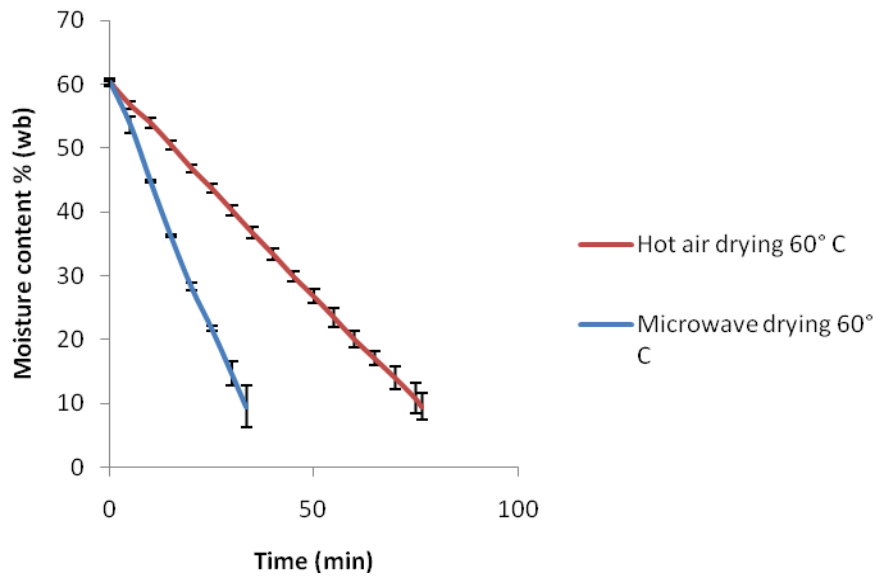


Figure 4.8. Comparison of microwave and hot air drying of flax fiber at 60° C

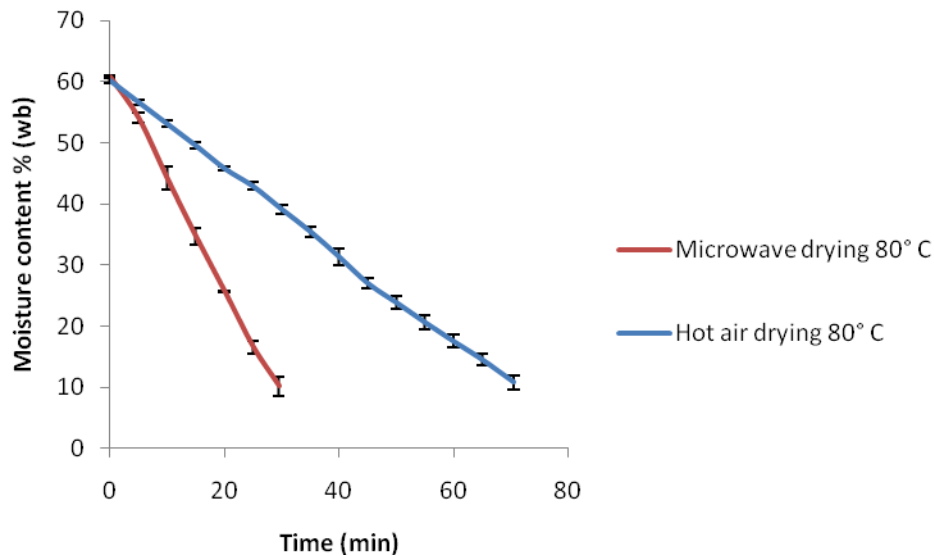


Figure 4.9. Comparison of microwave and hot air drying of flax fiber at 80° C

Thus, in all the three temperature ranges, microwave-convective drying showed a considerable reduction of drying time when compared to the hot air drying. Similar observations were presented Alibas (2007) and Torringa et al. (1996). Shivhare, et al. (1990) showed that, as a result of initial excess moisture removal, the initial drying rate

of flax fibre samples under hot air drying was higher than the final drying rate. Comparatively, under microwave drying, this difference in drying rates was minimal. The microwave–convective drying and hot air drying were compared, and the microwave-convective drying at 80° C avers itself the suitable method for flax fibre in terms of residence time in the drying chamber.

4.3.2. Flax straw

The microwave drying of flax straw from 70% w.b. to 9% w.b. moisture content took 70 min at 40°C, but only about 30 min at 60°C and 80°C; thus, the drying rate increased with an increase in temperature between 40°C and 60°C (Figure 4.10). At 40°C, 60°C and 80°C microwave drying took, respectively, 48%, 48%, and 42% less time to dry flax straw than hot air drying (Figures 4.11, 4.12 and 4.13, respectively)

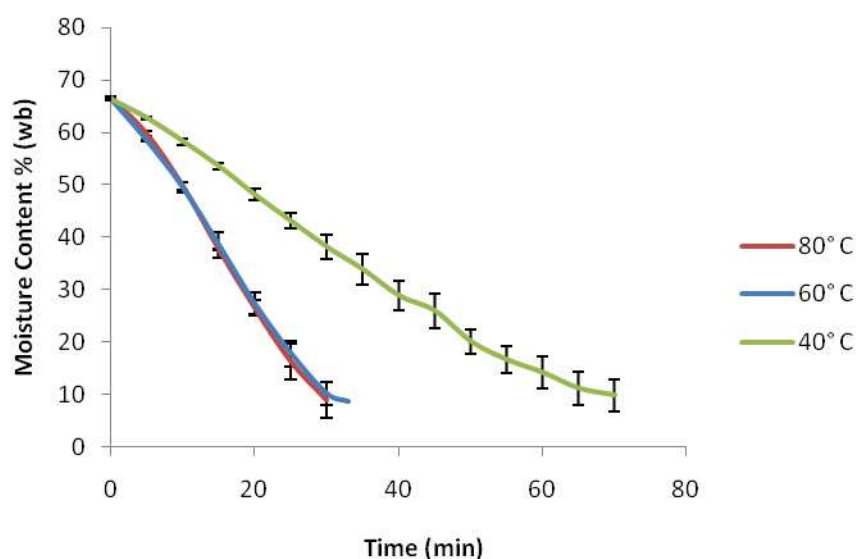


Figure 4.10. The temporal variation of moisture content during microwave drying of flax straw at temperatures of 40°C, 60°C and 80°C

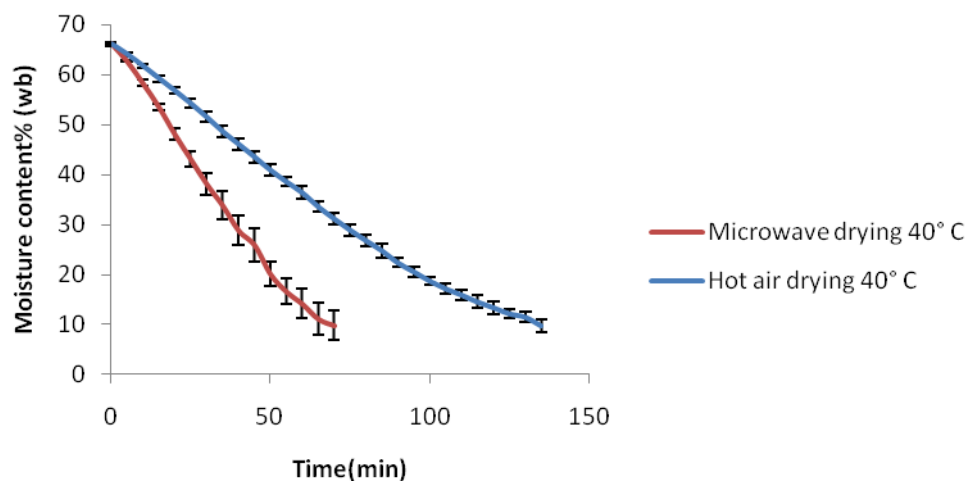


Figure 4.11. Comparison of microwave and hot air drying of flax straw at 40° C

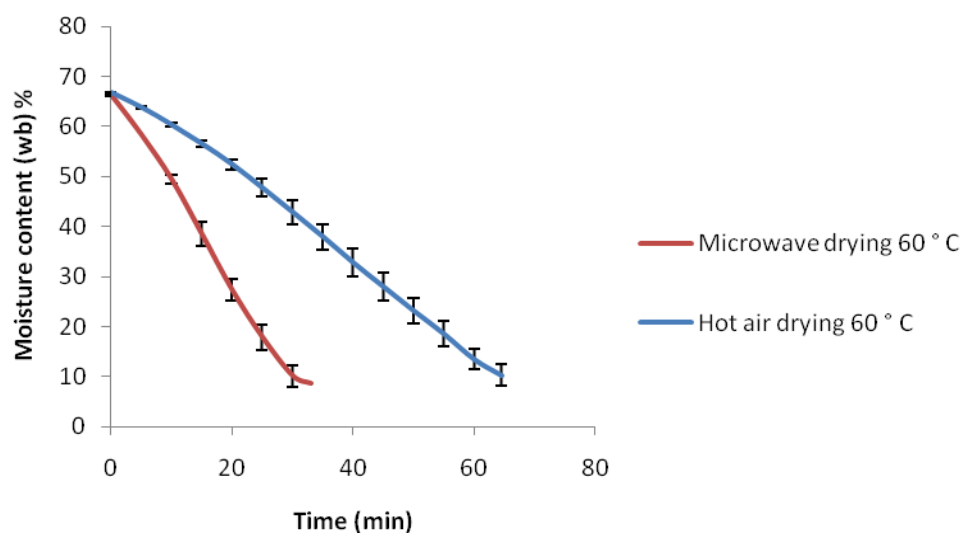


Figure 4.12. Comparison of microwave and hot air drying of flax straw at 60° C

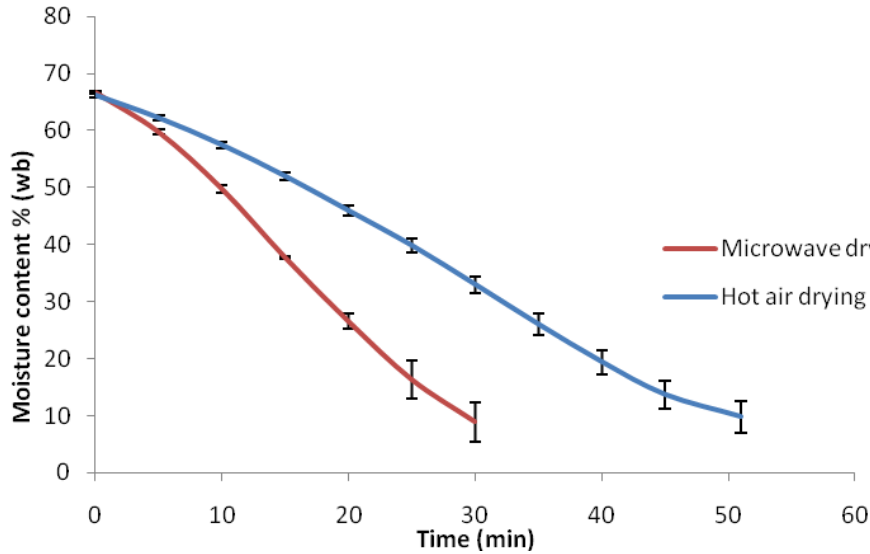


Figure 4.13. Comparison of microwave and hot air drying of flax straw at 80° C

Previous studies showed hot air convective drying of flax straw followed by microwave drying produced a better quality product with higher drying rate and shorter drying time (Alibas, 2007; Torringa et al., 1996). Shivhare et al. (1990) showed the initial drying rate of flax straw samples to be higher than the final drying rate under hot air drying as a result of the initial removal of excess moisture; however, under microwave drying, the difference of drying rates was much less. In terms of residence time, microwave-convective drying at 80° C was found to be the most suitable method for drying flax fibre and straw

4.3.3. Mathematical Modeling

The models [Eqs. 4.2-4.7] were fitted to the flax fibre and flax straw drying curves and statistics reflecting model accuracy were calculated (Table 4.2.). For both straw and fibre, both drying methods, all temperatures and all models, values of R^2 exceeded 0.9945

The drying constant k in both microwave and hot air drying increased with an increase in the processing temperature. (Table 4.2). During the microwave drying of flax fibre and straw, the power level was kept constant throughout the experiments. Others have shown that k increases with an increase in microwave power level (Drouzas et al., 1999). For

flax fibre, the χ^2 values were low for both microwave and hot air drying, ranging from 6.10×10^{-6} to 1.10×10^{-3} , while RMSE values ranged from 3.00×10^{-2} (Newton model, 40°C microwave drying) to 4.60×10^{-3} (Newton mode, 40° C hot air drying). X^2 values ranged from 0.009 to 0.0000067 and RMSE values ranged from 0.06 to 0.001 for flax straw samples dried using microwave energy and hot air. For flax straw, the χ^2 values were low for both microwave and hot air drying, ranging from 6.70×10^{-6} to 9.00×10^{-3} , while RMSE values ranged from 6.00×10^{-2} to 1.00×10^{-3} .

All 6 models fitted drying data well for both microwave-convective and hot air drying of flax fibre. For 40°C microwave-convective drying of flax fibre, the Wang and Singh model showed the best result in fitting the experimental data, while at 60°C and 80°C the Page and modified Page models gave best the best fit (Table 4.2). The logarithmic model showed the best fit for hot air drying of fibre at 40°C, 60°C and 80°C (Table 4.2). For flax straw the Page and modified Page models gave the best result in fitting microwave drying at 40°C, at 60°C the Wang and Singh model gave the best, while at 80°C the modified Page model gave the best fit. For hot air drying the Page model showed the best fit for drying at 40°C, while the Wang and Smith performed best at 60°C and 80°C (Table 4.2).

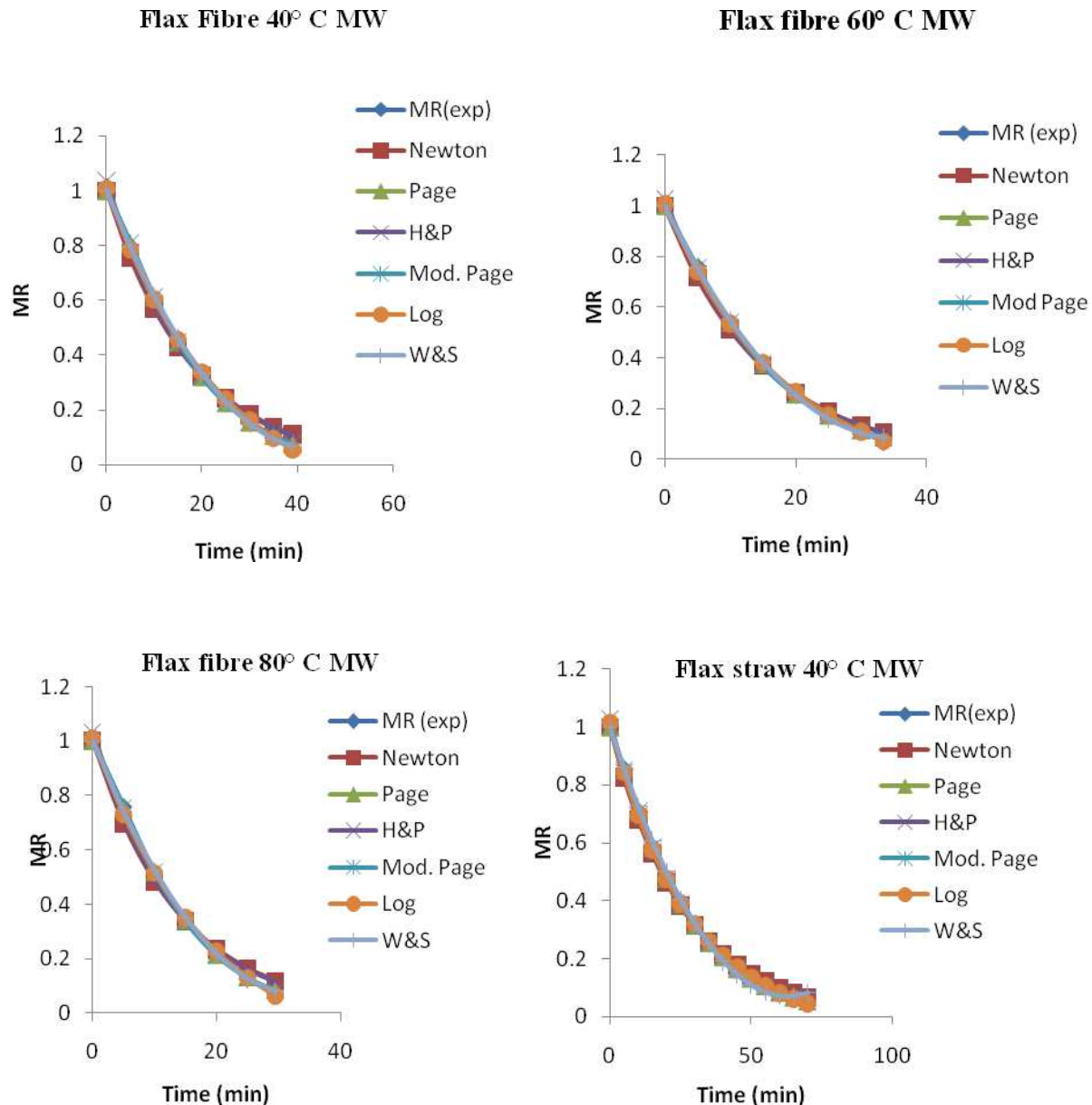
Table 4.2. Estimated values of drying constants, RMSE values and Chi- square (χ^2) values for different models in flax fibre and flax straw drying.

MATERIAL	DRYING	TEMP (°C)	STATISTICS / COEFFICIENT	Models												
				NEWTON	PAGE		HENDERSON & PABIS		MODIFIED PAGE		LOGARITHMIC		WANG & SINGH			
				k	k	n	a	k	a	K	n	a	K	c	a	B
Fibre	MW	40	Coeff.	5.64×10^{-2}	3.05×10^{-2}	1.21	1.04	5.84×10^{-2}	5.55×10^{-2}	1.21	1.19	4.15×10^{-2}	-0.18	-4.33×10^{-2}	4.98×10^{-4}	
			χ^2	1.15×10^{-3}	1.36×10^{-4}			1.05×10^{-3}	1.36×10^{-4}			6.71×10^{-5}			7.31×10^{-6}	
			RMSE	3.19×10^{-2}	1.03×10^{-2}			2.86×10^{-2}	1.03×10^{-2}			6.68×10^{-3}			7.54×10^{-3}	
		60	Coeff.	6.67×10^{-2}	4.44×10^{-2}	1.15	1.02	6.83×10^{-2}	6.57×10^{-2}	1.15	1.11	5.52×10^{-2}	-0.10	-5.23×10^{-2}	7.47×10^{-4}	
			χ^2	6.10×10^{-4}	7.96×10^{-5}			5.74×10^{-4}	7.96×10^{-5}			1.41×10^{-4}			2.54×10^{-4}	
			RMSE	2.31×10^{-2}	7.73×10^{-3}			2.08×10^{-2}	7.73×10^{-3}			9.37×10^{-3}			1.38×10^{-2}	
		80	Coeff.	7.26×10^{-2}	3.87×10^{-2}	1.23	1.03	7.50×10^{-2}	7.14×10^{-2}	1.23	1.19	5.39×10^{-2}	-0.18	-5.61×10^{-2}	8.43×10^{-4}	
			χ^2	1.30×10^{-3}	5.65×10^{-5}			1.28×10^{-3}	5.65×10^{-5}			2.81×10^{-4}			1.29×10^{-4}	
			RMSE	3.34×10^{-2}	6.35×10^{-3}			3.03×10^{-2}	6.36×10^{-3}			1.27×10^{-2}			9.60×10^{-2}	
Fibre	HA	40	Coeff.	1.82×10^{-2}	1.38×10^{-2}	1.07	1.01	1.8×10^{-2}	1.8×10^{-2}	1.07	1.07	1.53×10^{-2}	-0.08	-1.42×10^{-2}	5.50×10^{-5}	
			χ^2	2.62×10^{-4}	1.55×10^{-4}			2.51×10^{-4}	1.54×10^{-4}			2.43×10^{-5}			4.92×10^{-4}	
			RMSE	1.59×10^{-2}	1.20×10^{-2}			1.53×10^{-2}	1.20×10^{-2}			4.66×10^{-3}			2.14×10^{-2}	

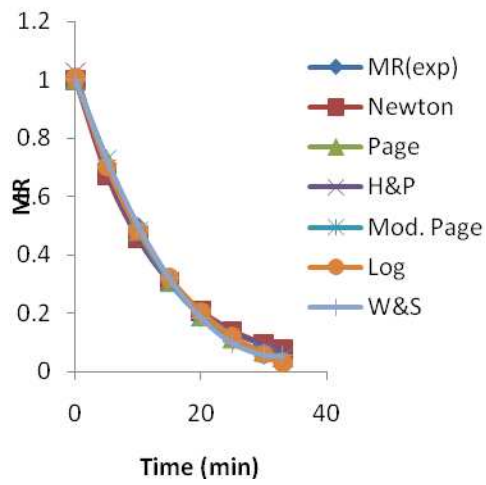
MATERIAL	DRYING	TEMP (°C)	STATISTICS / COEFFICIENT	Models											
				NEWTON		PAGE		HENDERSON & PABIS		MODIFIED PAGE		LOGARITHMIC		WANG & SINGH	
				<i>k</i>		<i>k</i>	<i>n</i>	<i>a</i>	<i>k</i>	<i>K</i>	<i>n</i>	<i>a</i>	<i>K</i>	<i>c</i>	<i>a</i>
Straw	40	MW	Coeff.	2.85×10 ⁻²	1.99×10 ⁻²	1.10	1.02	2.91×10 ⁻²	2.83×10 ⁻²	1.10	1.11	2.26×10 ⁻²	-0.12	-2.25×10 ⁻²	1.39×10 ⁻⁴
			χ ²	4.29×10 ⁻⁴	1.93×10 ⁻⁴		4.06×10 ⁻⁴	1.93×10 ⁻⁴			2.10×10 ⁻⁵		3.00×10 ⁻⁴		
	80	RMSE	2.01×10 ⁻²	3.44×10 ⁻²		1.89×10 ⁻²	1.31×10 ⁻²			4.20×10 ⁻³		1.63×10 ⁻²			
			Coeff.	3.03×10 ⁻²	2.18×10 ⁻²	1.09	1.02	3.09×10 ⁻²	3.01×10 ⁻²	1.09	1.11	2.43×10 ⁻²	-0.12	-2.42×10 ⁻²	1.63×10 ⁻⁴
Straw	40	MW	χ ²	2.3×10 ⁻⁴	6.06×10 ⁻⁵		1.95×10 ⁻⁴	6.30×10 ⁻⁵			7.90×10 ⁻⁶		6.82×10 ⁻⁵		
			RMSE	1.47×10 ⁻²	7.25×10 ⁻⁴		1.30×10 ⁻²	7.39×10 ⁻³			2.53×10 ⁻³		7.69×10 ⁻³		
	60	RMSE	Coeff.	3.84×10 ⁻²	2.75×10 ⁻²	1.10	1.03	3.95×10 ⁻²	3.80×10 ⁻²	1.10	1.06	3.58×10 ⁻²	-0.04	-2.97×10 ⁻²	2.37×10 ⁻⁴
			χ ²	2.74×10 ⁻⁴	2.62×10 ⁻⁵		1.90×10 ⁻⁴	2.62×10 ⁻⁵			8.44×10 ⁻⁵		4.16×10 ⁻⁴		
Straw	60	RMSE	1.60×10 ⁻²	4.77×10 ⁻³		1.28×10 ⁻²	4.77×10 ⁻³			8.22×10 ⁻³		1.90×10 ⁻²			
			Coeff.	7.87×10 ⁻²	4.71×10 ⁻²	1.19	1.03	8.06×10 ⁻²	7.66×10 ⁻²	1.19	1.11	6.42×10 ⁻²	-0.10	-5.94×10 ⁻²	9.33×10 ⁻⁴
	80	RMSE	χ ²	9.00×10 ⁻⁴	1.04×10 ⁻⁴		9.14×10 ⁻⁴	1.04×10 ⁻⁴			1.91×10 ⁻⁴		8.14×10 ⁻⁵		
			Coeff.	2.81×10 ⁻²	8.84×10 ⁻³		2.62×10 ⁻²	8.84×10 ⁻³			1.09×10 ⁻²		7.82×10 ⁻³		
Straw	80	RMSE	Coeff.	7.87×10 ⁻²	3.73×10 ⁻²	1.28	1.04	8.02×10 ⁻²	7.67×10 ⁻²	1.28	1.19	5.85×10 ⁻²	-0.17	-5.97×10 ⁻²	9.37×10 ⁻⁴
			χ ²	1.76×10 ⁻³	1.21×10 ⁻³		1.77×10 ⁻³	1.86×10 ⁻⁵			4.65×10 ⁻⁴		8.96×10 ⁻⁵		
	80	RMSE	3.88×10 ⁻²	2.94×10 ⁻²		3.56×10 ⁻²	3.65×10 ⁻³			1.63×10 ⁻²		8.00×10 ⁻³			

MATERIAL	DRYING	TEMP (°C)	STATISTICS / COEFFICIENT	Models											
				NEWTON	PAGE		HENDERSON & PABIS		MODIFIED PAGE		LOGARITHMIC		WANG & SINGH		
				k	k	n	a	k	n	K	a	K	c	a	B
Straw	HA	40	Coeff.	2.07×10^{-2}	1.80×10^{-2}	1.03	1.03	2.10×10^{-2}	2.06×10^{-2}	1.03	1.02	2.05×10^{-2}	-0.01	-1.61×10^{-2}	7.00×10^{-5}
			χ^2	3.83×10^{-5}	6.10×10^{-6}			1.96×10^{-5}	1.02×10^{-4}			1.24×10^{-5}		6.85×10^{-4}	
			RMSE	6.08×10^{-3}	2.39×10^{-3}			4.27×10^{-3}	9.75×10^{-3}			3.34×10^{-3}		2.52×10^{-2}	
			60	Coeff.	3.40×10^{-2}	1.54×10^{-2}	1.23	1.05	3.57×10^{-2}	3.35×10^{-2}	1.23	1.22	2.45×10^{-2}	-0.20	-2.61×10^{-2}
			χ^2	1.32×10^{-3}	1.16×10^{-4}			1.05×10^{-3}	5.24×10^{-3}			6.86×10^{-5}		1.95×10^{-5}	
			RMSE	3.51×10^{-2}	9.99×10^{-3}			3.00×10^{-2}	6.70×10^{-2}			7.34×10^{-3}		4.09×10^{-3}	
		80	Coeff.	4.48×10^{-2}	2.08×10^{-2}	1.24	1.05	4.69×10^{-2}	4.41×10^{-2}	1.24	1.21	3.27×10^{-2}	-0.19	-3.43×10^{-2}	3.09×10^{-4}
			χ^2	1.46×10^{-3}	1.24×10^{-4}			1.25×10^{-3}	1.24×10^{-4}			1.53×10^{-4}		6.70×10^{-6}	
			RMSE	3.64×10^{-2}	1.01×10^{-2}			3.19×10^{-2}	1.01×10^{-2}			1.05×10^{-2}		2.35×10^{-3}	

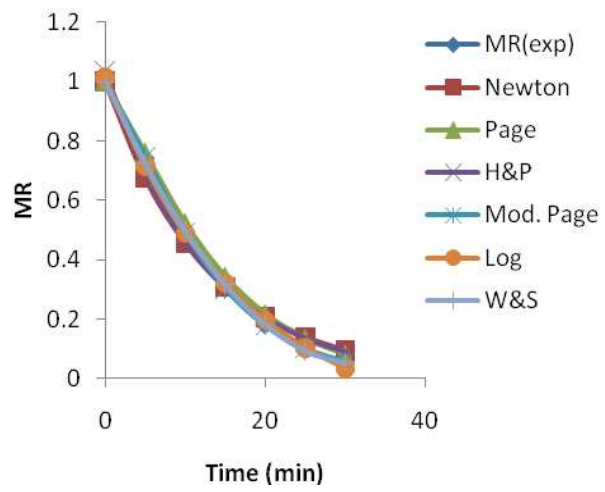
From Figure 4.14, which shows a comparison of the predicted and experimental values for various treatments and models, it is clear that the models were able to describe the drying curve closely for both microwave-convective and hot air drying, for both flax fibre and straw. These models are thus applicable in thin layer drying of agricultural commodities.



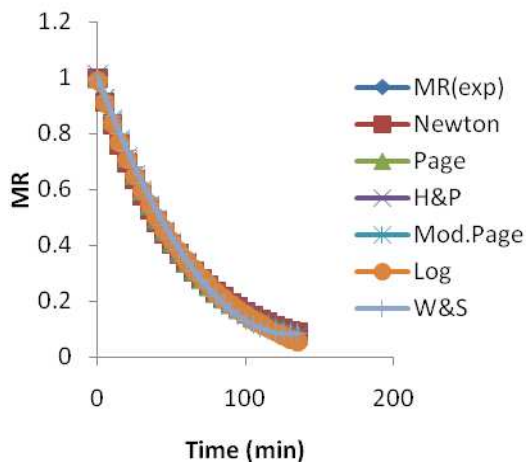
Flax straw 60° C MW



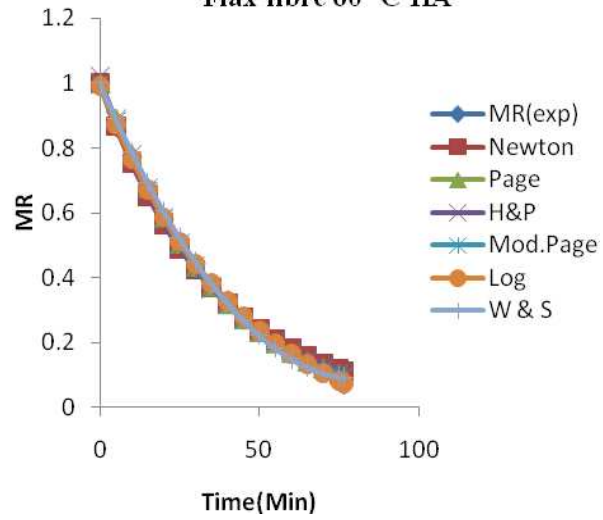
Flax Straw 80° C MW



Flax fibre 40° C HA



Flax fibre 60° C HA



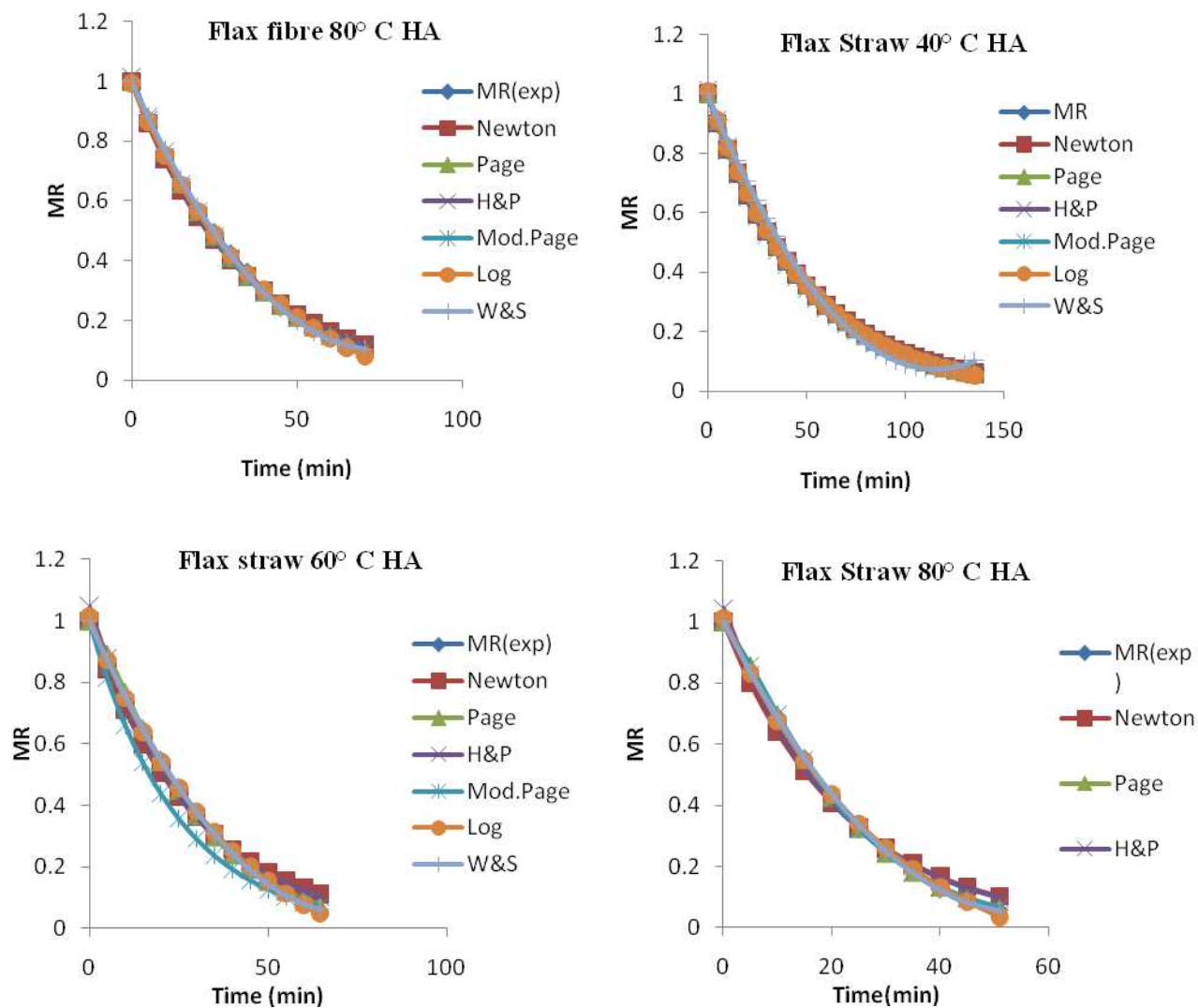


Figure 4.14 Mathematical models of microwave and hot air drying of flax fibre and flax straw

4.3.4 Colorimetric test of flax fibre and flax straw

4.3.4.1 Flax fibre

The L^* value of control/untreated flax fibre samples was roughly $+62$, while for microwave-convective and hot air dried samples, it ranged from $+63$ to $+68$ (Table 4.2). The change in L^* value was greatest for the 60°C microwave- convective dried flax fibre samples followed by the 80°C microwave-convective dried sample. The minimum change in L^* was recorded for 60°C hot air drying.

Table 4.3 Colorimetric values (L^* , a^* , b^*) of untreated, microwave-convective dried or hot air dried flax fibre (40°C, 60°C or 80°C), and colour difference (ΔE) of treated from untreated fibre. (Mean \pm Standard deviation). Column-wise values followed by the same letter are not significantly different. Test used: Duncan's Multiple Range test ($P > 0.05$)

Drying method	Temperature (°C)	CIELAB colorspace parameters			
		L^*	a^*	b^*	ΔE
Untreated	—	61.84 \pm 3.69 b	4.82 \pm 0.79 a	29.52 \pm 1.35 a	—
Microwave	40	64.64 \pm 0.88 ab	3.72 \pm 0.10 b	25.49 \pm 0.91 b	5.12 bc
	60	67.41 \pm 1.98 a	3.32 \pm 0.26 b	25.13 \pm 0.19 b	7.23 a
	80	66.48 \pm 1.01 a	3.25 \pm 0.36 b	24.90 \pm 0.49 b	6.72 ab
Hot air	40	64.95 \pm 0.91 ab	3.33 \pm 0.27 b	25.67 \pm 1.05 b	5.17 bc
	60	63.38 \pm 2.19 ab	3.70 \pm 0.54 b	26.20 \pm 1.70 b	3.82 c
	80	64.86 \pm 2.50 ab	3.16 \pm 0.27 b	25.27 \pm 1.17 b	5.47 abc

The L^* value of the untreated sample was significantly lower ($P \leq 0.05$) than that of the 60°C microwave-convective and 80°C microwave-convective dried flax fibre samples; however, there was no significant difference in the L^* value among dried flax fibre samples. There was a tendency at 60°C and 80°C for microwave-convective dried flax fibre samples to exhibit higher L^* value than their respective hot air dried samples, possibly due to the shorter duration of microwave-convective drying (Table 4.3)

The highest a^* value, +4.82, was recorded for untreated flax fibre sample, all dried samples had significantly lower a^* values, but no significant differences were found amongst the dried samples (Table 4.3). The significant difference in a^* value between dried and non-dried samples was because of the effect of the loss of moisture on the fibres.

The b^* value of untreated flax fibre sample was +29.5 (yellow), while those of dried samples ranged from +26.20 to +25.13 (Table 4.3). While microwave-convective and hot air drying showed no significant difference between them in terms of flax fibre b^* values ($P > 0.05$), b^* values of dried flax fibres were significantly lower ($P \leq 0.05$; i.e., less yellow)

than those of untreated fibres, and both the treated flax fibre samples showed a significant difference with the untreated flax fibre sample.

The change in colour, ΔE was greatest under 60°C microwave-convective drying, and least was under 60°C hot air drying. Colour changes of microwave-dried samples were due to browning and might be attributable to glucose or other sugars derived from cellulose. The ΔE for 60°C microwave-convective dried samples was significantly greater than that obtained by drying at 40°C by either method, or by microwave-convective drying at 80°C. The ΔE for 60°C hot air dried samples was significantly less than that for microwave-convective drying at either 60°C or 80°C. Thus, colour change was mainly affected by microwave power and temperature of the samples.

The colour changes in the microwave-convective dried flax fibre samples at 60°C and 80°C were amongst the largest. The colour of the flax fibre changed from grey to a brighter yellowish grey after drying, but those changes were difficult to perceive by the naked eye.

4.3.4.2 Flax straw

Dried flax straw samples had significantly higher ($P \leq 0.05$) L^* values than untreated samples, but no significant differences were found among dried samples by either method or temperature (Table 4.4)

The a^* values of the untreated straw were significantly greater ($P \leq 0.05$) than those of any dried samples. Among the dried samples only the 40°C and 80°C microwave-convective drying regimes showed significant differences between drying treatments (Table 4.4). The a^* values of the flax straw samples were dependent on the temperature and time of drying of the sample. In general, within the drying temperature range of 40°C to 80°C, the magnitude of change in a^* value depended on the time of heating, i.e., the residence time of the samples in the drying chamber.

Table 4.4 Colorimetric values (L^* , a^* , b^*) of untreated, microwave-convective dried or hot air dried flax straw (40°C, 60°C or 80°C), and colour difference (ΔE) of treated from untreated fibre. (Mean \pm Standard deviation). Column-wise values followed by the same letter are not significantly different. Test used: Duncan's Multiple Range test ($P \leq 0.05$)

Drying method	Temperature (°C)	CIELAB colourspace parameters			
		L^*	a^*	b^*	ΔE
Untreated	—	63.02 \pm 0.28 b	5.68 \pm 1.48 a	26.93 \pm 2.68 a	—
Microwave	40	72.33 \pm 1.33 a	1.95 \pm 0.34 c	20.45 \pm 0.47 cd	11.94 a
	60	71.36 \pm 3.75 a	2.39 \pm 0.32 bc	22.64 \pm 0.52 bc	9.94 a
	80	70.69 \pm 2.80 a	3.27 \pm 0.09 b	22.97 \pm 0.72 b	8.97 a
Hot air	40	72.82 \pm 0.97 a	2.88 \pm 0.43 bc	21.68 \pm 0.62 bcd	11.47 a
	60	73.20 \pm 1.50 a	2.30 \pm 0.27 bc	20.17 \pm 0.13 d	12.68 a
	80	73.32 \pm 2.48 a	2.81 \pm 0.19 bc	21.39 \pm 1.01 bcd	12.05 a

At ⁺27, the b^* value of untreated samples significantly exceeded that of any dried sample (Table 4.4.). Hot air drying at 60°C resulted in a lower b^* value of dried flax straw than microwave-convective drying at either 60°C or 80°C, but did not differ significantly from hot air drying at 40°C or 80°C. The untreated flax straw samples showed the highest b^* value, i.e., they were more yellow.

There was no significant difference in colour change (ΔE) among the different flax straw drying treatments (Table 4.4). But the colour change tended to be least under microwave-convective drying at 60°C and 80°C, possibly as a result of the shorter residence time of those samples compared to hot air treatments. Overall, microwave-convective drying of flax straw samples at 80° C was the most suitable method in terms of colour and drying rate.

4.3.5 Strength of flax fibre and straw

4.3.5.1 Flax fibre

The tensile strength of flax fibre samples were tested by using an Instron testing machine. The results are shown in Figure 4.15. From the graph, The tensile strength of both microwave-

convective dried flax fibre and hot air dried flax fibre increased significantly with an increase in the process temperature from 40°C to 80°C ($P \leq 0.05$), but of all other upward temperature shifts, only the 60°C to 80°C for hot air drying showed a similar increase in tensile strength of dried samples. Only drying at 80°C, by either method, increased the tensile strength significantly above that of the untreated fibre. Mueller and Krobjilowsky (2003) conducted a study on the effect of temperature on the impact strength of flax fibre reinforced composite material found an increase in the strength with an increase in the processing temperature. The flax fibre samples dried at 80°C by the microwave- convective method showed a significantly lesser tensile strength than those hot air dried at the same temperature ($P \leq 0.05$).

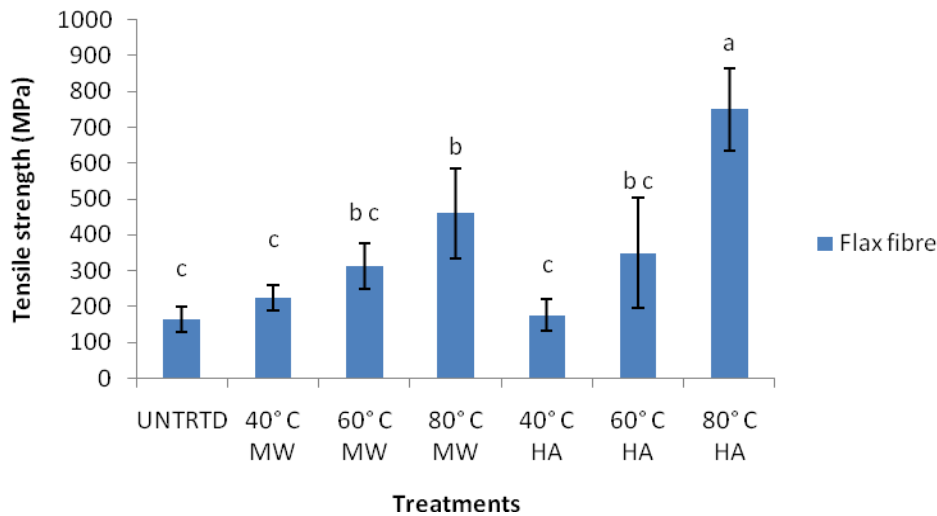


Figure 4.15 Comparison of tensile strength of untreated, as well as microwave-convective and hot air dried flax fibres.

An increase in tensile strength with an increase in drying temperature was shown for flax fibres. However, there is no literature addressing why the tensile strength of the 80°C microwave- convective dried flax fibre sample would be lower than that of fibres hot air dried at the same temperature (Figure 4.15).

Drying at 40°C by either the microwave-convective and hot air method had no significant ($P > 0.05$) effect on the elastic modulus of flax fibres; however, under both heating methods increases in drying temperature from 40°C to 60°C and from 60°C to 80°C both led to significant increases in elastic modulus (Figure 4.16). The elastic modulus of flax fibre was

significantly greater after drying at 80°C by hot air than by microwave-convective means, but this difference was not significant at either 60°C or 40°C.

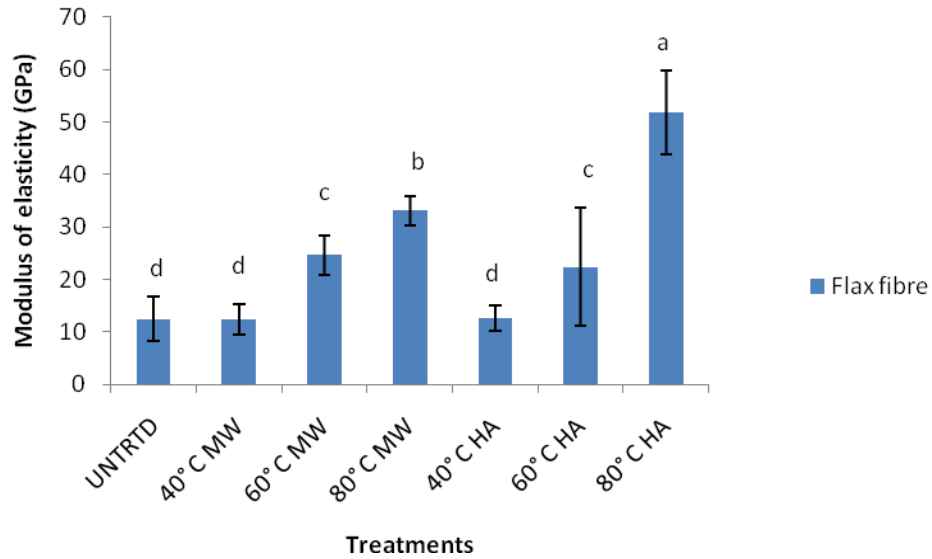


Figure 4.16 Comparison of elastic modulus of untreated, as well as microwave-convective and hot air dried flax fibre

4.3.5.2 Change of elastic modulus of the fibre with diameter

The diameter range of the flax fibres selected for the test was from 0.07 mm to 0.13 mm. For both microwave-convective and hot air dried flax fibre samples, there was a decreasing trend in the modulus of elasticity with an increase in the diameter of the fibre (Figures 4.17, 4.18). The 40°C hot air dried flax fibre samples showed the weakest inverse relationship between diameter and modulus of elasticity.

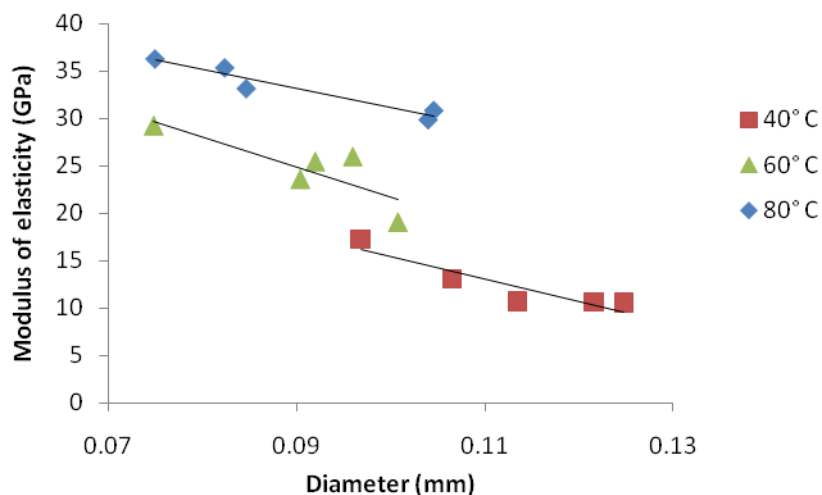


Figure 4.17 Relation between diameter of the microwave-convective dried fibres and their modulus of elasticity.

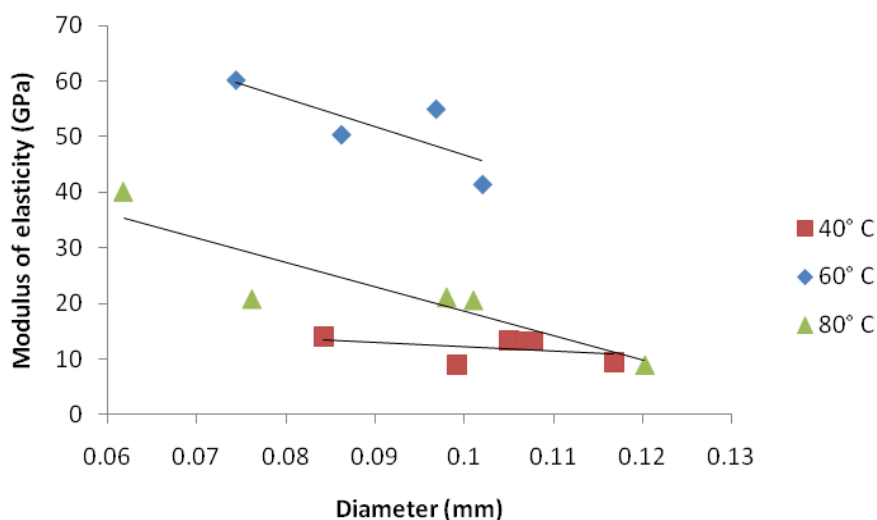


Figure 4.18 Relation between diameter of hot air dried fibres and their modulus of elasticity

4.3.5.3 Flax straw

The tensile strength of untreated flax straw was significantly lower than that of hot air dried at 60°C or 80°C, but not significantly different from straw hot air dried at 40°C or from straw microwave-convective dried at any temperature (Figure 4.19). For microwave-convective

dried straw, the drying temperature had no significant effect on tensile strength, whereas for hot air drying, tensile strength after drying at 80°C was significantly greater than at either 60°C or 40°C. At both 60°C and 80°C (but not 40°C) drying temperatures, the tensile strength was greater after hot air drying than microwave drying.

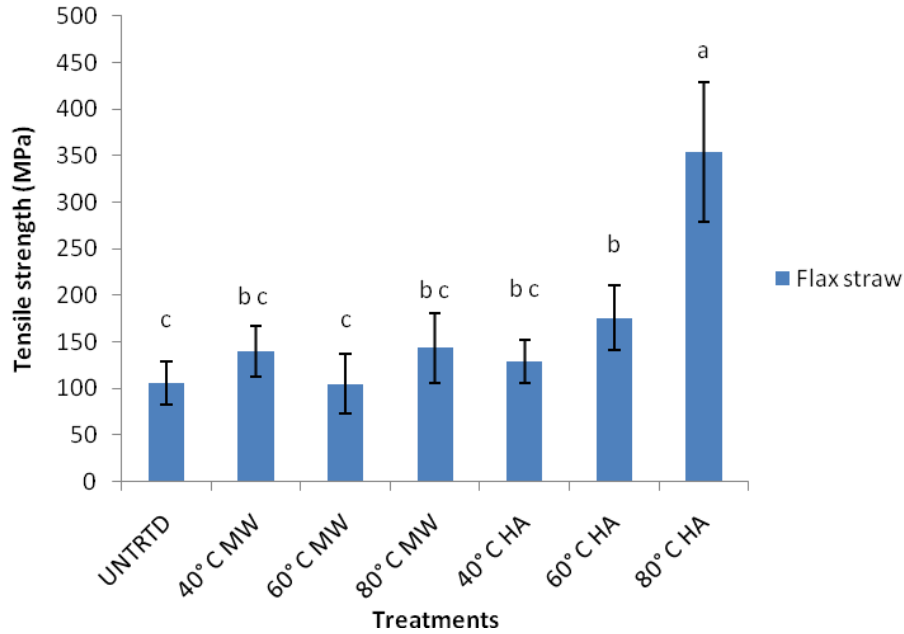


Figure 4.19 Comparison of tensile strength of untreated, as well as microwave-convective and hot air dried flax straw

The modulus of elasticity of the flax straw was compared for the different drying treatments in Figure 4.20. The modulus of elasticity of 80°C hot air dried samples was greater ($P \leq 0.05$) than that of any other samples treated or untreated, which showed no significant differences in the modulus of elasticity amongst themselves.

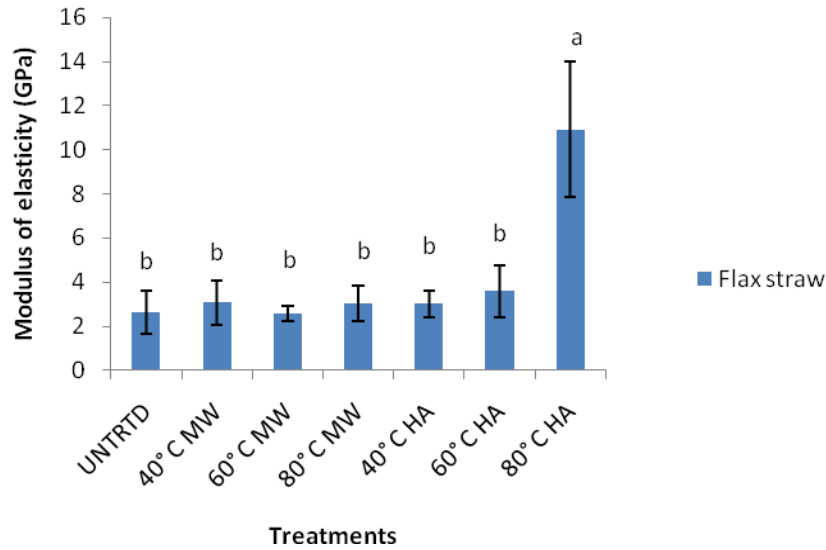


Figure 4.20 Comparison of elastic modulus of untreated, as well as microwave-convective and hot air dried flax straw

The diameter of the flax straw and modulus of elasticity for both the microwave-convective and hot air dried sample were compared in Figure 4.21 and 4.22. The sample diameter ranged from 0.7 mm to 1.3 mm. Generally speaking, microwave-convective dried and hot air dried flax straw samples showed a decrease in the modulus of elasticity with an increase in the straw diameter (Figure 4.21, 4.22).

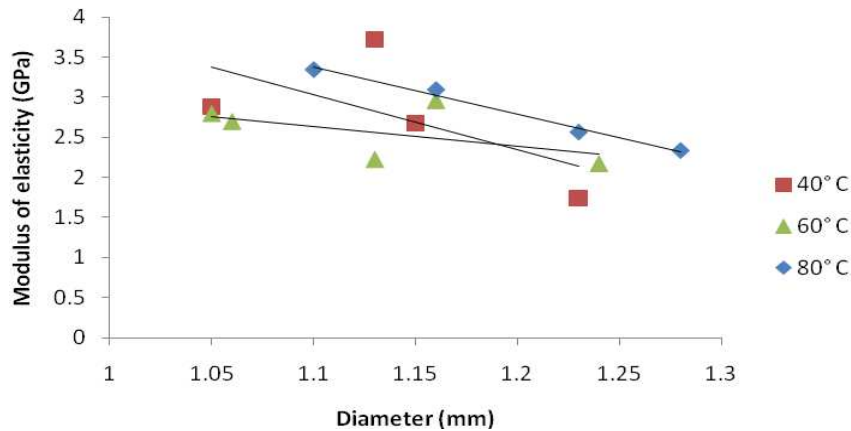


Figure 4.21 Relation between diameters of the microwave- convective dried flax straw and its modulus of elasticity

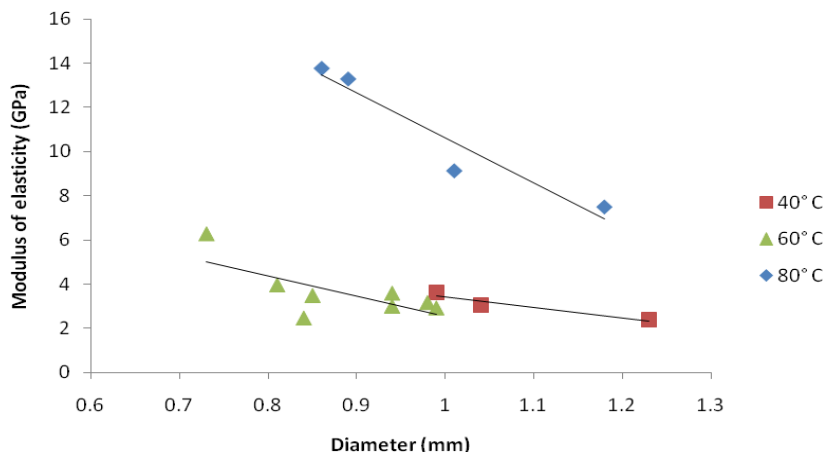


Figure 4.22 Relation between diameters of the hot air dried flax straw and its modulus of elasticity.

4.4 CONCLUSIONS

The microwave-convective drying of flax fibre and straw was conducted at 40°C, 60°C and 80°C, and results were analysed and compared with hot air convective drying. At the drying temperatures of 40°C, 60°C and 80°C microwave convective drying took 30.8%, 54.8% and 48.5 % less time, respectively, than hot air drying. Among the microwave- convective drying conditions tested, drying at 80°C method proved to be the most suitable in terms of drying time.

Thin layer drying models were fitted to the drying data of both microwave and hot air convective drying of flax fibre and straw. All six models showed suitable fit to the experimentally generated drying curves (i.e., high R^2 values, low χ^2 and root mean square error values).

The color change of the flax fibre and straw were studied in comparison with the initial untreated samples and microwave convective drying tended to result in greater colour change than did hot air convective drying. There was a significant difference in the color change between microwave-convective and convective drying of flax fibre. But in the case of flax straw, there was no significant difference in the colour change between microwave and hot air convective dried samples.

Tensile properties of microwave-convective and hot air dried flax fibre and straw were studied and compared with an initial non-dried sample. The tensile strength and modulus of elasticity increased with an increase in the temperature for both flax fibre and straw, but the tensile strength and modulus of elasticity of hot air dried flax fibre and straw were higher than that of microwave dried samples. The modulus of elasticity was found to decreasing with an increase in diameter of both flax fibre and straw.

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CONNECTING TEXT

After studying the effect of controlled temperature microwave drying on flax straw and fibre physical properties for the storage and production of bio-composites, studies were conducted on the compression of flax straw to produce an artificial fuel source. The flax straw has the potential to act as an artificial heating source because of its heating value. The study included the comparative evaluation of firelogs made from various proportions of flax straw, sawdust and wax.

CHAPTER 5

STUDY OF PROPERTIES OF FIRELOGS MADE WITH FLAX STRAW, SAWDUST AND WAX

Abstract

Compression of flax straw into fuel briquettes is a value addition process for flax producers. The objective of this study was to compare the properties of firelogs mixed with different proportions of flax, sawdust and wax. The maximum firelog burning temperature of 231° C was obtained by samples with 60% saw dust and 40% wax (00:60:40), followed by a firelog composed of 70% flax straw and 30% wax (70:00:30) at 228° C. The same trend was shown in the average burning temperature of 150.4° C for 00:60:40 samples and 143.1° C for 70:00:30 samples. The burning rate was increased with an increase in the percentage of wax. The burning rate was minimum (3.18 g min⁻¹) in 35:35:30 fire logs. For the same wax content, firelog samples made with 100% flax straw produced the more residues than sawdust-based firelogs. The maximum load/energy applied to break a firelog was obtained for the 70:00:30 firelog samples. Hence, after considering all the key factors, the most feasible firelog composition is one of 70:00:30, i.e. 70% flax straw and 30 % wax.

Key words: Firelog, flax straw, sawdust, wax.

5.1 INTRODUCTION

Flax (*Linum usitatissimum* L.) is a major crop in Canada. Flax plant belongs to the Linaceae family. Flax seeds are mainly used for the production of oil or in the food industry, while the plant stems are used for fibre production. Flax straw was formerly considered a waste after the harvesting of flax seeds. Later the producers recognized the potential of flax straw in different commercial applications. Totally unprocessed flax straw in square bales can be used as a simple building/insulation material for livestock wind-break structures, barn wall insulation and containment for feed bunks/grain pile storage. The lower specific gravity and bulky nature of flax straw leads to the problems in the storage, transportation and disposal. Flax uses much more

space in a storage area, but if it is exposed to the outside environment, rain and wind will destroy its properties (Mankowski, 2008). Flax straw in bales, or as loose flax straw, can also be used as geotextile for erosion control on slopes, for soil stabilization around livestock water bunks in feedlots and soil enhancement during road construction. Flax straw can be used as a potential heat source as its calorific value is high when compared to sawdust. The conversion of flax straw into a source of heat is a value addition process by which the farmers would benefit.

Fireplaces using biomass to heat rooms during the winter season are very common in North America. Fire wood is selected on the basis of its capacity to produce energy while burning in the fireplaces. Commonly used firewoods are maple (*Acer* sp.) and oak (*Quercus* sp.) in North and Central America. However, natural firewood is difficult to store given its irregular shape, lack of control over moisture content, difficulty in lighting, etc. Largely to avoid these problems, artificial firelogs were introduced. These are mostly made from wood remains like saw dust, wood chips etc. Binding agents are mixed with sawdust or other burning components to activate the burning and the material compressed in a mold to ensure greater strength and greater convenience in storage. They are easy to heat with, and leave behind less ash is left after burning. Usually firelogs are made from sawdust by using wax as a binder (Houck et. al. 2000). The percentage of binder can vary from 0 to 60% depending on the type of firelogs. A firelog with a minimum percentage of binder (wax) is very dense and difficult to light (De Hoop et al., 2005).

Experiments were conducted on firelogs produced with different proportions of flax straw, sawdust and wax to find the optimum proportion of flax and wax binder in terms of the firelog's thermal properties and strength.

The objectives of the study were:

- a) To produce firelogs with different proportions of flax straw, sawdust and wax mix.
- b) To analyse the burning rate, mean and maximum burning temperature and percentage weight of residues for each samples.
- c) To conduct a puncture test to find out the maximum load and energy to break the firelog.

5.2 MATERIALS AND METHOD

5.2.1 Initial moisture content

Initial moisture content of the flax straw and fibre on a wet basis (w.b.) were measured by drying 25 g samples in a hot air oven for 24 hours at 105°C, and calculating moisture content (M.C.) of the flax fibre or straw (ASAE S358.2 Dec 93):

$$\text{M.C. (wet basis)} = \frac{M_w}{M_w + M_s} \quad (5.1)$$

where,

M_w is the mass of water in the sample (g),

M_s is the mass of solid in the sample (g)

5.2.2 Flax straw

In these experiments, flax straw was tested as the major flammable raw material for the production of firelogs. Flax straw samples for the experiments were collected from local farmers near Montreal. The initial moisture content was found to be 4.1% w.b.. The flax straw was cut in small pieces of 40 to 50 mm length to ensure easy and uniform mixing with sawdust and wax.

5.2.3 Sawdust

Sawdust was also used in the production of firelogs as a basis for comparison with the results obtained with firelogs made from flax straw. Wooden pellets made from pine tree sawdust purchased from local market in Montreal, were used for the experiment. The pellets were crushed into the form of saw dust for use in producing fire logs. The wood pellets were crushed in such a way that the individual particle size did not exceed 10 mm. The initial moisture content of the sawdust was calculated by using the oven drying method, and found to be 4.6% w.b.

5.2.4 Wax

Paraffin wax was used as the binder material for the production of firelog samples. Paraffin wax used for the experiments was purchased from a local market and had a melting point range of 68 to 72° C (Kamusewitz et al., 1999). Wax was heated inside a funnel to above 72°C with the help of a gas torch and mixed with the flax straw and sawdust before compressing the material to produce a firelog.

5.2.5 Mold

The mold for the production of a firelog was a cast iron cylinder and piston (Figure 5.1). The cylinder was 50 mm in inner diameter and 30 cm in length. The dimension of the piston was 350 mm in length and 4.9 cm in diameter, giving a 1 mm clearance for the cylinder (Chin and Siddiqui, 2000).

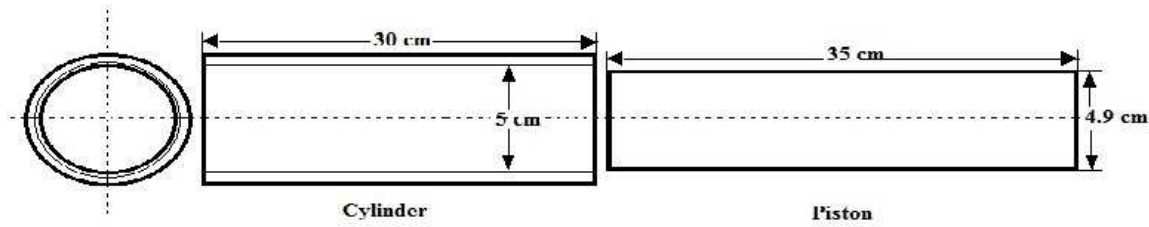


Figure 5.1 Diagram of mold for producing firelog

5.2.6 Hydraulic compressor

The flax straw-sawdust wax mixture was compressed in a manual hydraulic compressor with a maximum compressive strength of 60 tonnes. The cylinder filled with the flax straw-sawdust mixture was placed in the hydraulic compressor and pressure was applied by moving the handle up and down. The piston was compressed till the sample reached the desired volume and the pressure was released by turning a handle.

5.2.7 Chimney

A double-wall insulated steel chimney (Figure 5.2) was used for burning the firelog samples. The chimney was placed on a perforated tray with a support made from steel to provide sufficient air circulation while burning. The dimension of the chimney was 180 mm inner diameter and 290 mm outer diameter. The length of the chimney was 450 mm. The volume inside the chimney was 0.011 m³.



Figure 5.2 The chimney for burning the firelog samples

5.2.8 Thermometer and thermocouple

An Omega HH147 RS 232 Data Logger Thermometer attached to a thermocouple was used for the measurement of the temperature 0.3 m above the firelog samples. The temperature was recorded at 1 min intervals.

5.2.9 Instron Machine for measuring maximum load

The maximum load and energy required to break the firelog samples were calculated by pushing a 5 mm diameter probe into the samples (Figure 5.3), using an Instron materials testing machine (Instron – 4502, Instron Corporation, USA) controlled by Instron series IX, version 8.25 computer software. The machine head speed was maintained at kept at 25 mm min⁻¹.



Figure 5.3 Instron setup for puncture test of firelogs

5.2.10 Experimental design

The firelogs were prepared with three types of materials; unadulterated flax straw, unadulterated saw dust and a 50:50 mixture of flax straw and sawdust. These three flax straw-saw dust proportions bound together with three different percentages of wax content (20%, 30% or 40%). Three replicates of each test were conducted. The following characteristics were analysed after the production of firelog.

1. Maximum temperature of combustion
2. Mean temperature of combustion
3. Burning rate of firelogs
4. Percentage weight of residues after burning
5. Maximum load tolerated in puncture test
6. Energy required to break the firelog

Table 5.1 Experimental design

Base material	Binder (wax) content (%)	Treatment designation
Unadulterated flax straw	20	80:0:20
	30	70:0:30
	40	60:0:40
50: 50 Flax straw : Saw Dust	20	40:40:20
	30	35:35:30
	40	30:30:40
Unadulterated saw dust	20	0:80:20
	30	0:70:30
	40	0:60:30

5.2.11 Preparation of firelog samples

Firelog samples for the experiments were produced by mixing flax straw, sawdust and wax in various proportions. Mixing was done on a 0.70 m × 0.70 m metal sheet. The flax straw samples were cut in small pieces of 40 to 50 mm to ensure uniformity in size. Sawdust particles ranged from were 0.5 to 1.5 mm in diameter. The total weight of the firelog sample was maintained at 100 g for all experiments. To produce an 80:0:20 firelog sample, 80 g of flax straw was spread on the metal sheet. Wax (20 g) was placed in a conical steel funnel, heated by using a gas torch, and then poured on the flax straw. The flax straw and wax were mixed manually and loaded into the cylinder. The bottom of the cylinder was closed by placing it on a cast iron platform. The cylinder with the platform was kept on the hydraulic compressor. The piston was inserted in to the cylinder and the material compressed using the hydraulic compressor (De Hoop et al., 2005). The compression proceeded until the samples reach a mean height of 50 mm. After

5 seconds the whole system was released from compression and the sample was removed by pushing the piston after removing the base. The same procedure was repeated for all other samples

5.2.12 Burning of firelog samples

The burning of firelog samples was done in a double walled insulated chimney with an inner diameter of 0.18 m, an outer diameter of 0.29 m and a height of 0.45 m. The chimney was placed on a metal base support with a perforated iron sheet to provide aeration while burning the samples. The firelog sample was placed on the perforated tray base inside the chimney. The sample was ignited using a gas torch and the time and temperature was recorded manually by using stop watch and thermometer. After burning, the residues were weighed to measure the percentage weight of residues.

5.2.13 Puncture test of firelogs

Puncture tests of the firelogs were performed by using an Instron machine (Instron – 4502, Instron Corporation, USA) controlled by a computer software (Instron series IX, version 8.25). The diameter of the probe used for the experiment was 50 mm. The firelog sample was placed on the platform and the probe was guided automatically at a speed of 25 mm min⁻¹. The load, displacement and energy were recorded through an attached computer (Ranganna et al., 1997).

5.3 RESULTS AND DISCUSSION

The diameter of the fire logs produced was 5 cm. The length varied from 48 to 60 mm depending on the pressure applied on the piston. The fire log samples were taken out and their densities calculated by measuring their dimensions. The density of the fire logs varied from 0.79 g cm⁻³ to 1.02 g cm⁻³ (Table 5.2) depending on the pressure applied and the materials to be compressed.

Table 5.2 Burning of fire logs of different composition

Fire log (flax: sawdust:wax)	Pressure applied for compression (Pa)	Density (g cm ⁻³)	Maximum Temperature (°C)	Rate of Burning (g min ⁻¹)	Weight of Residue (g)
80:00:20	6500	0.79	114.5	3.35	13.94
40:40:20	6500	0.83	152.5	3.42	12.72
00:80:20	6500	0.94	174.0	3.51	5.82
70:00:30	5000	0.80	228.5	4.03	13.5
35:35:30	5000	0.94	190.5	3.19	11.28
00:70:30	5000	1.02	184.0	4.15	3.08
60:00:40	3600	0.80	214.5	3.94	10.79
30:30:40	3600	0.97	206.5	3.70	8.94
00:60:40	3600	0.88	231.0	4.44	1.37

5.3.1 Burning of fire logs with different proportion of flax straw saw dust and wax

5.3.1.1 Fire logs with 20% wax

Firelogs with proportions of 80:0:20 (straw:sawdust:wax), 40:40:20 and 0:80:20 (80%, 40%, 0% flax straw) were burned and temperatures monitored 0.30 m above the firelog (Figure 5.4). The 0:80:20 firelogs reached a maximum temperature 174° C in 3 minutes and took 28 min to burn completely (Table 5.2). The 80:0:20 firelogs reached a maximum temperature of 105° C in 3 minutes and took 29 min to burn completely. This higher burning temperature of firelogs with sawdust was mainly attributable to the higher calorific value of wood, 20.5 kJ kg⁻¹ d.w.b. (Lehtikangas, 2001) when compared to flax straw, 17.7 kJ kg⁻¹ d.w.b. (Shaw and Tabil, 2006).

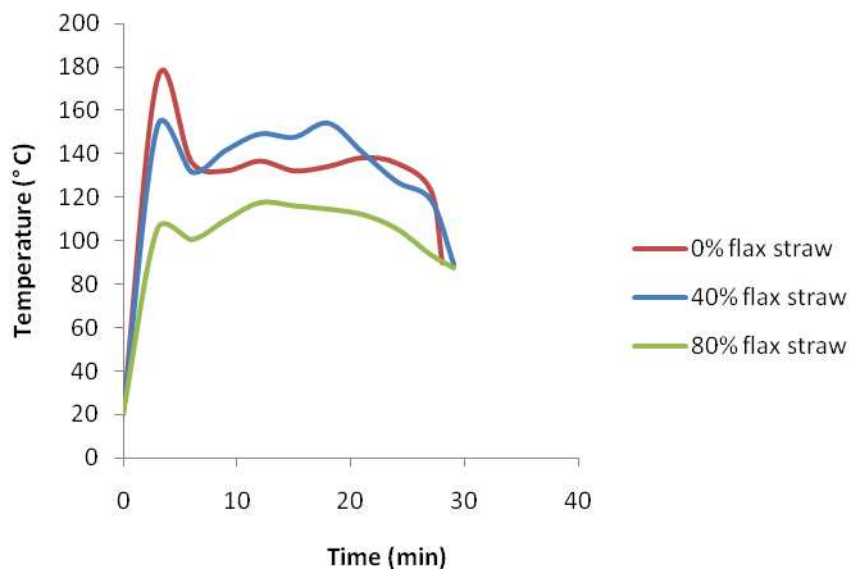


Figure 5.4 Comparison of temperatures 0.30 m above burning firelogs made with 20% wax

The 40:40:20 firelog took 29 minutes to burn fully. The full burning time was largely equal for the three samples containing 20 % wax.

5.3.1.2 Firelogs with 30% wax

The firelogs with proportions of 70:0:30, 35:35:30 and 0:70:30 were tested and the temperature 0.30 m above them monitored during burning (Figure 5.5). The maximum temperature achieved by 70:00:30 firelogs was 192°C within 3 minutes and a total burning time of 25 min. The 35:35:30 firelogs reached their maximum temperature (188°C) in 6 minutes and had a total burning time of burning of 31 minutes. The 0:70:30 samples burned completely in 23 minutes and reached a maximum temperature of 184°C in 3 minutes. Thus the 35:35:30 samples burned longer than the other 30% wax firelogs. Compressed flax straw took more time to burn completely than compressed saw dust with 30% wax, due to the higher calorific value of the wood (Joshi et al., 1989).

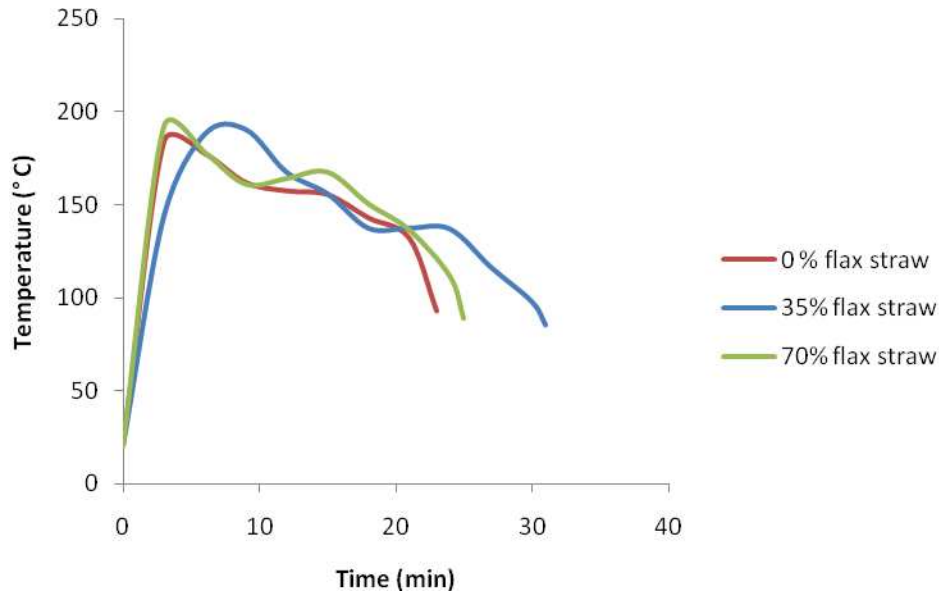


Figure 5.5 Comparison of temperatures 0.30 m above burning firelogs made with 30% wax

5.3.1.3 Firelogs with 40% wax

The firelogs with proportions of 60:0:40, 30:30:40 and 0:60:40 were tested and the temperature 0.30 m above them monitored during burning (Figure 5.6). The maximum temperature achieved with the 60:00:40 firelogs was 223°C within 6 minutes, with a total burning time of 25 min. The 30:30:40 firelogs reached their maximum temperature of 206° C in 3 minutes, with a total burning time of 26 min. The 00:60:40 samples burned completely in 20 minutes and had a maximum temperature of 231° C reached in 3 minutes. The increased temperatures reached by the 40% wax firelogs was attributable to the high percentage of wax binder, a finding similar to that of Chin and Siddiqui (2000).

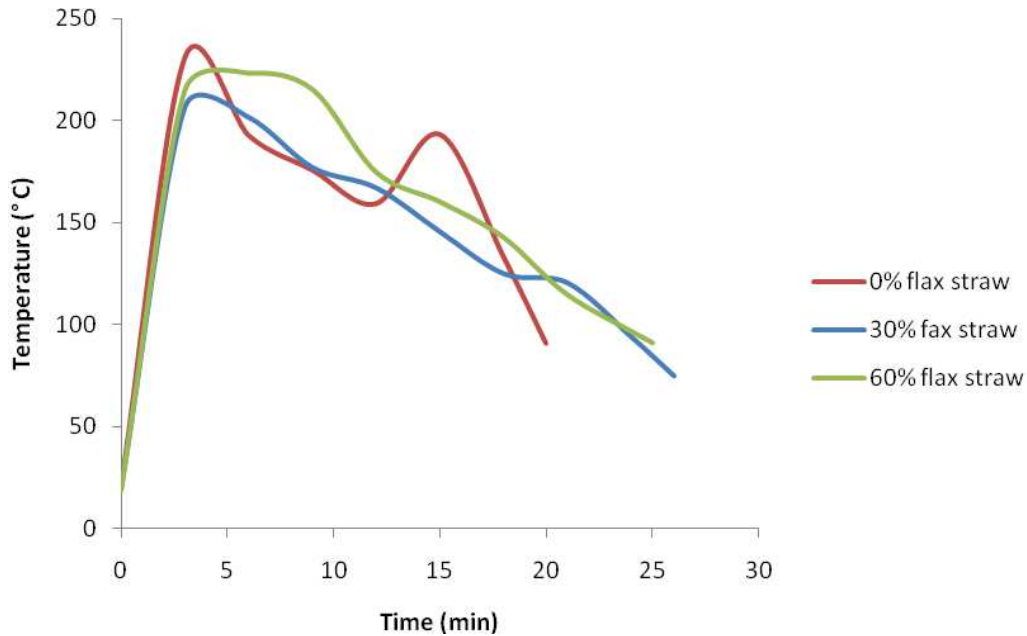


Figure 5.6 Comparison of temperatures 0.30 m above burning firelogs made with 40% wax

Amongst all samples the burning time was greatest for the firelogs with equal parts flax straw and saw dust. Comparatively, firelogs with flax straw and wax only took more time to burn than firelogs with only sawdust and wax. The burning temperature of all the firelog samples reached a peak at the beginning of the burning process, and temperature decreased thereafter. The decrease in the temperature was due to the increased thermal resistance of the carbon layer, formed as the sample burned out, which minimised the rate of heat transfer by conduction from the burning surface to the interior of the burning sample (Abduragimov et al., 1986).

The rate of burning of the fire log samples are shown in Table 5.2. The rate of burning with wax percentage is shown in Figure 5.7.

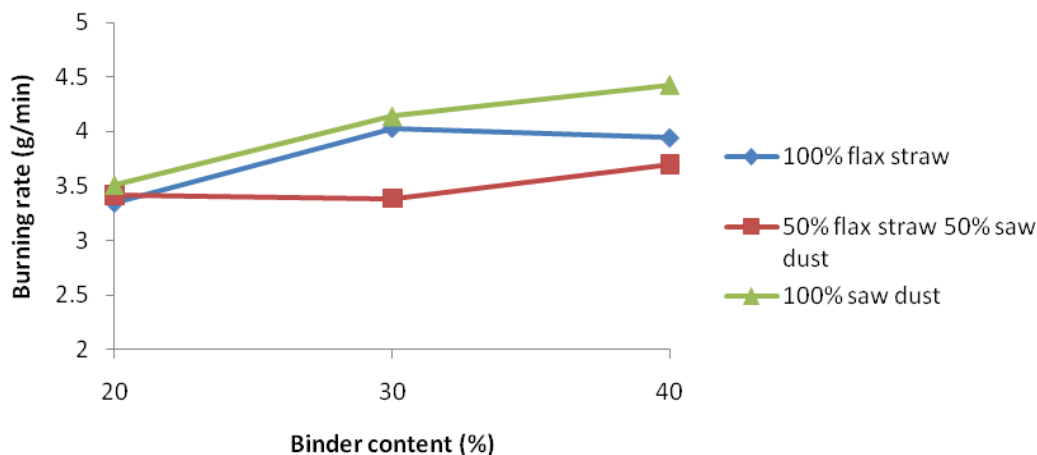


Figure 5.7 Rate of burning with wax binder content.

Chin and Siddiqui (2000) found the burning rate of pure saw dust firelogs to be the highest, and that it increased with an increase in the percentage of wax binder. However, unadulterated flax straw firelogs showed a lower burning rate than unadulterated saw dust firelogs. At 20% wax, all the samples showed almost the same burning rate. In terms of burning rate alone, the 100% flax straw, with 20% binder (80:00:20) firelogs were the best; however, their mean temperature was minimal at 98.5°C. The 35:35:30 firelogs showed the lowest burning rate and a mean temperature of 132°C, and was thus more acceptable in terms of burning rate and temperature.

5.3.2. Maximum temperature attained while burning

The maximum burning temperature 0.30 m above the firelogs with least for 20% wax firelogs, and greatest for the 40% wax firelogs (Figure 5.8). The highest maximum temperature overall (231°C) was obtained with 0:60:40 firelogs, but 70:00:30 and 60:00:40 samples showed similar maximum temperatures of 228°C and 214°C, respectively. The maximum temperature of burning of firelog was positively related with the wax content which initiated burning and ensured even burning of the samples (Houck et. al., 2000).

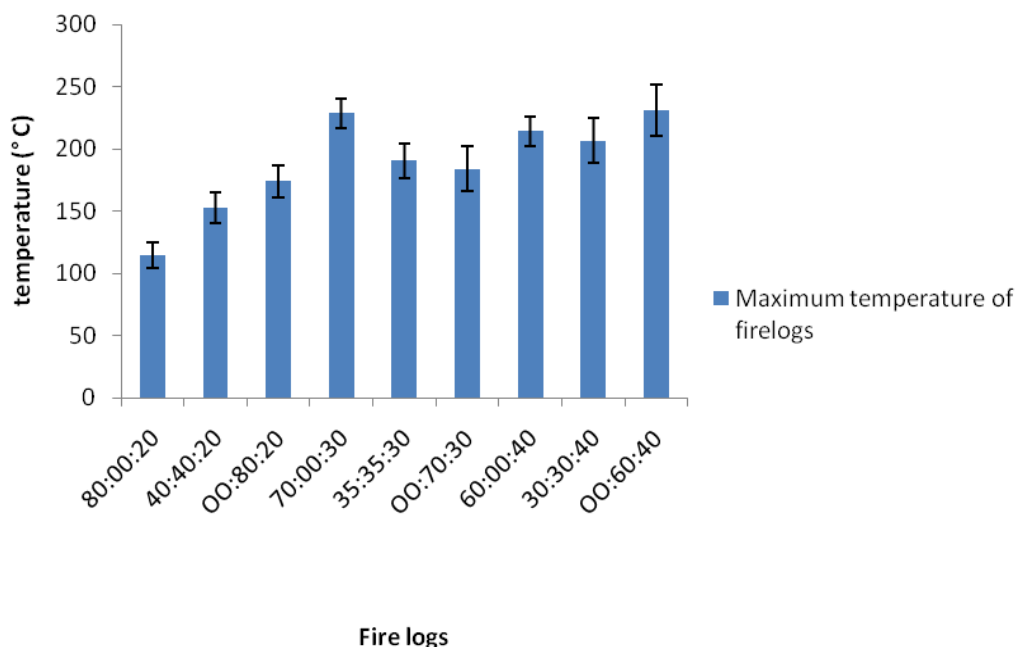


Figure 5.8 Maximum temperature attained during burning

5.3.3 Mean temperature of burning of fire logs

The average temperature of burning of different fire logs is shown in Figure 5.9. The 60:0:40 firelogs had the greatest mean burning temperature of 150.4°C, while the 70:0:30 had a mean burning temperature of 143.1° C. However, the 80:0:20 firelog showed the lowest mean burning temperature at 98.5°C.

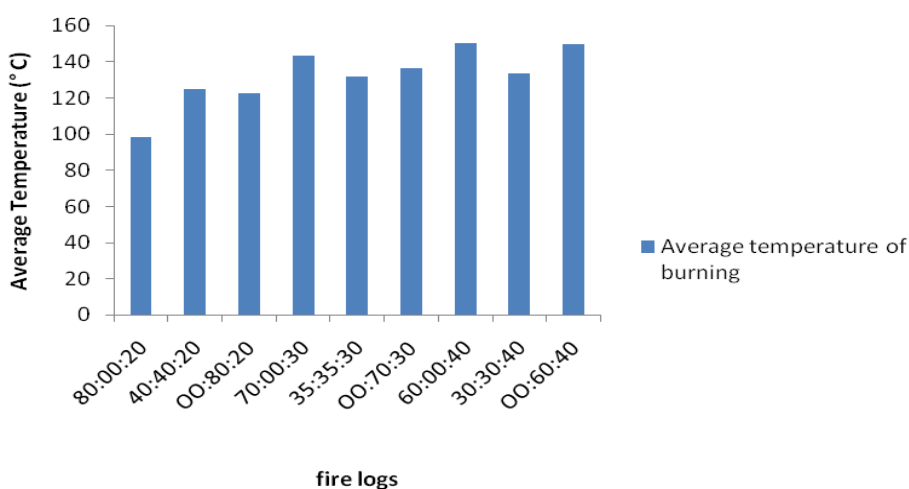


Figure 5.9 Mean burning temperature 0.30 m above fire logs

Thus, in terms of mean burning temperature and rate, the 60:00:40 firelogs were most suitable, followed by the 70:00:30 firelogs. The higher burning temperature of the firelogs related to the property of wax which helped in continuous burning of the firelog. The heat content of a wax mixed firelog is higher than that of normal wood (Houck et al., 2000; Wang et al., 2009). This explained the increase in the burning temperature with an increase in the wax percentage.

5.3.4 Rate of burning of fire logs

Rate of burning was least for the fire log samples with 20% wax content (Figure 5.10). As the percentage of wax increased, the rate of burning also increased (Chin and Siddiqui, 2000). In this study, firelogs 50% flax straw and 50% saw dust showed the lowest burning rate and lowest mean temperature for both 30% and 40% wax content.. The lower burning rate might be attributable to the uneven burning of flax straw and saw dust due to the differences in their calorific values.

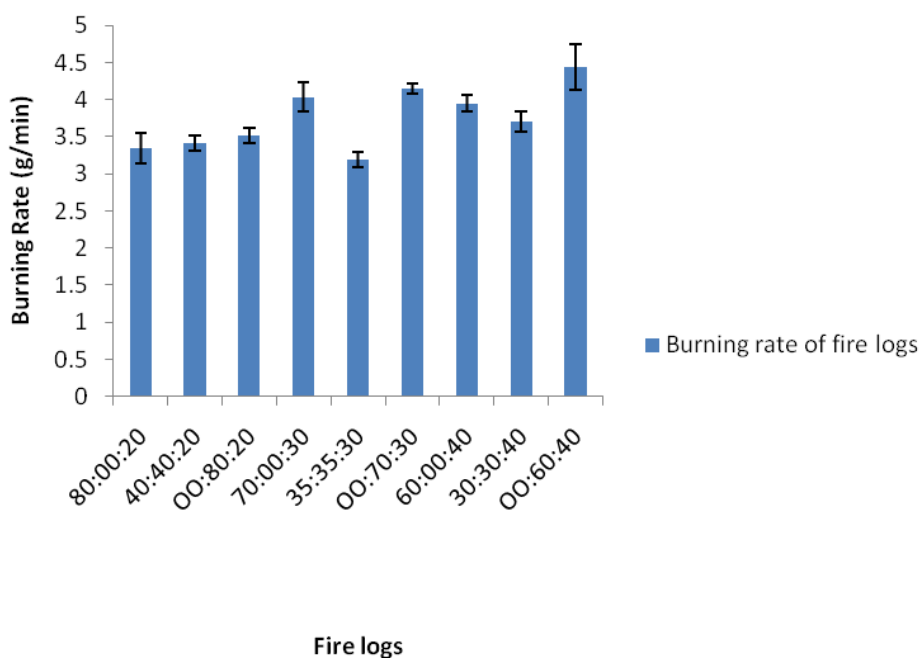


Figure 5.10 Rate of burning of different fire logs

5.3.5. Weight of residue after burning of fire logs

Firelogs with only flax straw produced the most ash of all firelogs, followed by 50% flax straw and 50% saw dust firelogs (Figure 5.11). The minimum amount of residue was found in fire logs with 0% flax straw. This is because it is the nature of sawdust to produce less amount of ash or residue after burning (Lehtikangas, 2001).

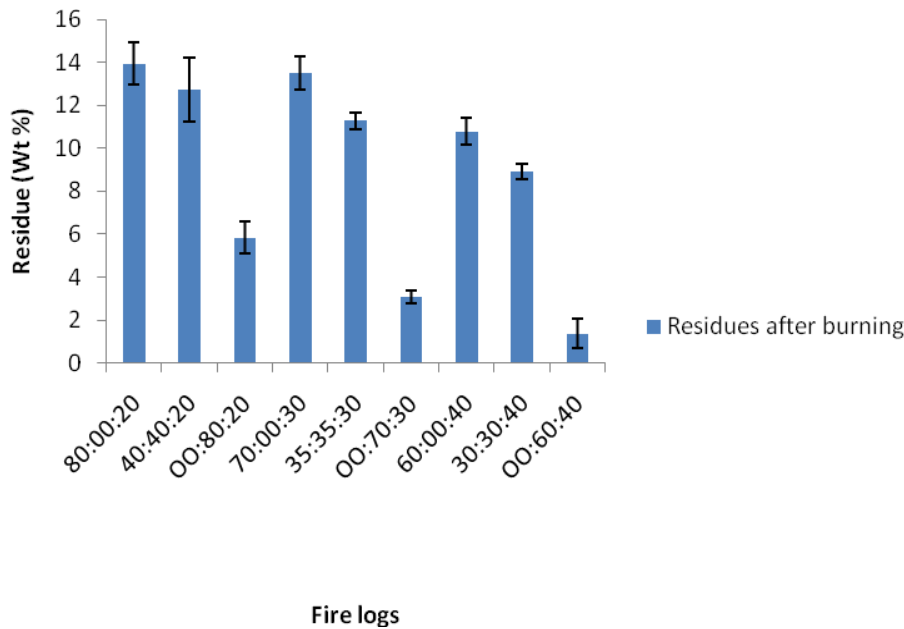


Figure 5.11 Weight of residues after burning

5.3.6. Puncture test on the fire logs

5.3.6.1 Maximum load on the samples

The maximum load applied to firelogs before failure/penetration (Figure 5.12) was greatest for flax straw only firelogs, followed by samples with 50% flax straw and 50% sawdust, followed in turn by 100% sawdust firelogs. The maximum load, 358.2 N was recorded on 70:00:30 firelogs, followed by 356 N, on 60:00:40 firelogs.

Thus the maximum load applied to firelogs with 100% flax straw indicated greater strength than in 100 % saw dust firelogs. Firelogs with 50% flax straw and 50% sawdust fell between the single material firelogs for all the three proportions of wax binder (Wamukonya and Jenkins, 1995). The fibre and lignin presented in the flax straw improved the strength of the samples when compared to sawdust (Day, 2005).

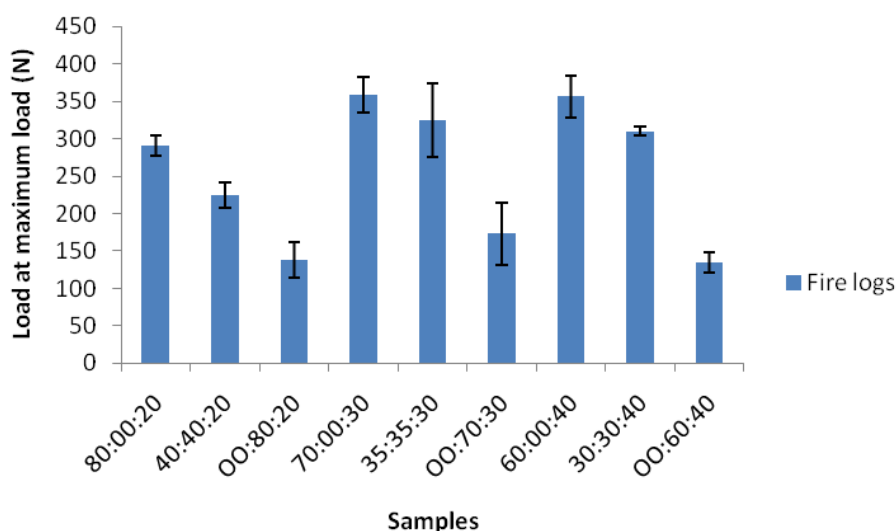


Figure 5.12 Maximum load on the fire log samples

5.3.6.2. Energy at breaking point

The energy in Joules at the breaking point of the fire log samples (Figure 5.13) was greatest in firelogs with 100% flax straw. This was a result of the better bonding of fibres and the presence of lignin in the flax stem. Among the firelogs, the maximum energy was recorded for 70:00:30 firelogs. Thus, samples with 30% wax and 70% flax straw was suitable in terms of strength. The energy used in breaking 60:00:40 firelogs was less than that of 70:00:30 due to the lesser amount of pressure applied to the 60:00:40 fire log samples during the production. The pressure applied was reduced to avoid the wax to be squeezed out of the mold.

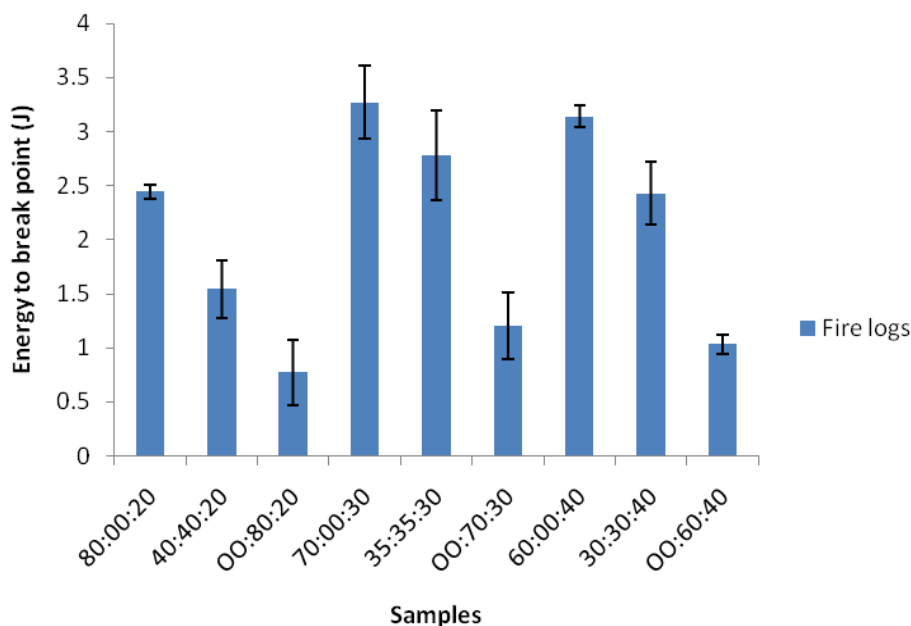


Figure 5.13 Energy at breaking point of the fire log samples

The energy at breaking point of the firelogs showed same trend as load at maximum load: for all the samples studied, the firelogs with 100% flax straw showed the maximum energy at breaking point for all the three wax binder percentages, followed by 50% flax straw- 50% sawdust and 100% sawdust firelog samples (Wamukonya and Jenkins, 1995; Day, 2005).

Conclusion

The properties of fire logs made from flax fibre were studied. The maximum and mean burning temperatures were found to be greater for 70:0:30 firelogs. At 3.18 g min^{-1} the minimum burning rate occurred with 35:35:30 firelogs, while the highest burning rate (4.14 g min^{-1}) was achieved with 70:00:30 firelogs. The percentage weight of residues was maximum in fire logs with only flax straw and minimum in firelogs with only sawdust. The strength of the fire log samples to withstand load and the energy necessary to break the samples was greatest for 70:00:30 firelogs. Considering all the results, a firelog made from 70% flax straw and 30% wax was best among all tested firelog compositions in terms of strength and burning properties.

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CHAPTER 6

SUMMARY AND CONCLUSION

Flax is abundant in Canada. Flax seeds were considered as the only useful part of a flax plant. But later, the significance of flax straw and its fibre was recognized by the modern civilization and now flax fibres are used in many areas like apparel industry, paper industry etc.

A study on microwave drying of flax fibre under controlled hot air drying temperatures was set up to reduce the time of heating and to increase product quality. The flax straw was also heated by using a microwave-convective method. Considering both drying methods at three different temperatures of 40° C, 60° C and 80° C, it was observed that the percentage reduction of drying time was 30.8%, 54.8% and 48.5 % respectively, for the microwave method. Drying curves were successfully fitted with various mathematical models.

The color change of microwave-convective dried flax fiber was greater than that of hot air dried samples within the range of temperatures tested. However flax straw did not show any significant colour change. The tensile strength and modulus of elasticity increased with an increase in the temperature for both flax fibre and straw; however, the tensile strength and moduli of elasticity of hot air dried flax fibre and straw were greater than those of microwave dried samples. The modulus of elasticity was found to decrease with an increase in diameter of both flax fibre and straw.

The burning and compressive properties of flax straw were studied by producing artificial firelogs with flax, sawdust and wax. The burning rate was increased with the increase in the binder (wax) percentage. The burning rate and residue content was high in firelogs containing 70% flax straw and 30% wax. The mean burning temperature, maximum burning temperature, as well as the mechanic properties of load and energy at breaking point was high in the 70% flax straw and 30% wax mixed firelog. So while considering all the factors discussed, the firelog with 70% flax straw and 30% wax appeared to be the best.

The microwave drying of flax straw was very fast when compared to hot air drying. But the size of flax straw bale is so large that it is not possible to dry one in a normal small microwave cavity. So, the design of a bigger system suitable for the efficient drying of the big bales is necessary.

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