

A Chemical/Thermal Pretreatment Process for the Dewatering
of Fuel-Grade Peat

by
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Abstract

Raw fuel-grade peat was treated with a surfactant, one of three inorganic salts or one of three cationic polymers. The pretreated peat was then pressed to determine the effect of these additives on water removal. Pressed peat samples treated with these compounds were resuspended in water and the weight percent of particles settled after one hour was measured. This data was correlated with the water removal data and provided insight into the effect of each additive on peat dewatering.

Using a batch heating system, raw peat was heated to temperatures below 200C before mechanical dewatering. Variations were made in heat up time, residence time and holding temperature. After heat treatment, dewatering could be further enhanced by the addition of one of the cationic polymers, chitosan hydrochloride.

The effect of several pretreatment processes on the quality of the expressed peat water was monitored. Toxicity tests showed that growth of microorganisms was uninhibited by the peat waters.

Resume

La tourbe energetique a ete traitee avec un surfactant, un des trois sels inorganiques ou des trois polymeres de charge positive. La tourbe, preablement traitee, a ete comprimee pour ensuite determiner l'effect des additifs sur l'extraction d'eau. Des echantillons de tourbe comprimes, traites avec ces additifs, ont ete suspendus dans l'eau.

Apres une heure, le pourcentage du poids des particules deposees a ete mesure. Les resultats obtenus ont ete compares avec les resultats d'extraction d'eau donnant plus d'information sur l'effet de chaque additif sur la deshumification de la tourbe.

Utilisant un systeme de bain chaud, la tourbe est chauffee a une temperature inferieure a 200C avant la deshumification mecanique. Des variations ont ete faites avec des parametres thermiques.

Apres traitement thermique, la deshumification peut etre encore plus augmente par l'addition de chitosan hydrochlorique, un des trois polymeres de charge positive.

L'effet de plusieurs traitement sur la qualite de l'eau de tourbe a ete observee. Des test pour contamination ont montre que le developpement de microorganismes n'a pas ete affecte par l'eau de tourbe.

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1. Introduction

Peat is a heterogeneous substance derived from plant matter in aquatic environments called bogs. Peat is composed of inorganic and organic solids and water. The aqueous fraction typically comprises 90% of its weight. When a large portion of the water is removed the solids remaining are a potential fuel.

Peat has been burned to generate heat for centuries in Northern Europe. Peat derived fuel (PDF) is used in the USSR, Ireland and Finland as a boiler feed to generate steam for electric power plants and for district heating. It can also be converted to synthetic fuels by thermal and/or biological processes. However, all of these applications must be preceded by a dewatering process.

1.1. Peat resources and classification

Known world peat resources are approximately 420.8 million hectares. Peat resources in North America are vast and mostly untapped. In fact, at present there is absolutely no commercial use of peat for fuel in either the United States or Canada (Punwani, 1981). Estimates of peatland rank Canada first with 170 million hectares. Peat has been identified as the second largest recoverable energy source after coal in the United States (Bettinger et al. 1983).

Chemical and physical characteristics of peat deposits vary widely. Properties depend on the type of plant matter which has settled in the bog, the organisms which have aided in decomposition and the climate.

The most widely used criterion for classifying peat in a natural deposit is degree of decomposition. One universally accepted method based on humification was developed by the Swedish scientist L. von Post in 1926. Useful in the field, the von Post scale ranks peat according to the color of water it releases when squeezed and the degree to which the plant remains are identifiable. Peat is ranked in ten categories. H1 peat releases almost clear water and the plant remains are easily identifiable. H10 peat has no discernable plant structure and the wet peat escapes between the fingers when squeezed (Cooper et al., 1984). Table 1.1 shows how the elemental analysis of peat samples varies with degree of decomposition (Fuschman, 1980).

The U.S. department of agriculture has developed a classification system (1975) which is also based on the degree of decomposition. It is actually a simplified version of the von Post scale. There are only three categories: Fibric which corresponds to von Post H1 to H3, hemic, von Post H4 to H6 and sapric.

According to Farnham (1981), for energy applications, classification should include caloric value, bulk density, ash content, sulfur content and percent of fines including colloids in the peat material. Kopstein (1980) in discussing the U.S.-D.O.E. peat program list the following requirements for fuel grade peat:

1. contain at least 18500 kJ/kg (dry basis)
2. must be 1.5 meters or more in thickness
3. contain less than 25% ash by weight (dry)
4. contain at least 80 acres of contiguous peat per square mile

Table 1.1. Variation in elemental composition with degree of humification.

Organic Element	Humification H1-H3	Humification H6-H7	Humification H9-H10
Carbon (%)	48-53	56-58	59-63
Hydrogen (%)	5.0-6.1	5.5-6.1	5.1-6.1
Oxygen (%)	40-46	34-39	31-34
Nitrogen (%)	0.5-1.0	0.8-1.1	0.9-1.9
Sulfur (%)	0.1-0.2	0.1-0.3	0.2-0.5

If the fuel grade peat is to be dewatered mechanically, water retention properties are also important.

Peat may also be classified on a chronological scale with other solid fuels. There is a continuum of coal grades, ranging from the oldest, anthracite, which has high fixed carbon, low volatiles and low oxygen content, to peat (often referred to as a "young coal") which is typified by high volatiles and oxygen content (Rohrer, 1985). In between peat and anthracite lie sub-bituminous coal, lignites and brown coals. Peat is compared to other fuels in table 1.2 (Monenco, 1981).

There are many properties which distinguish peat as a fuel from coal. Being 90% water and found in surface deposits, peat may be extracted, nondestructively, with floating harvesting equipment. Once extracted, post excavation reclamation is relatively inexpensive and effective. Many peats have extremely low ash content making them more attractive for direct combustion in oil fired boiler plants not readily convertible to coal (Rohrer, 1981). Peat is typically lower in sulfur than coal. This eliminates the need for expensive fuel desulfurization processes or pollution controls. Furthermore, the higher volatile content of peat makes it a more attractive feedstock for gasification processes.

1.2. Peat dewatering

Current peat dewatering process designs include three stages. The pretreatment stage entails either heat addition or chemical addition. The pressing stage removes between 55% and 85% of the water depending on the nature of the peat and the

Table 1.2. Properties of Peat and Related Fuels

Property	Fuel			
	Wood	Peat	Lignite	Bitumi- nous
Carbon (%)	48-50	50-60	65-75	76-78
Hydrogen (%)	6.0-6.5	5.0-6.5	4.5-5.5	3.3-5.0
Oxygen (%)	38-42	30-40	20-30	2.8-11.3
Nitrogen (%)	0.5-2.3	1.0-2.5	1-20	2.8-1.2
Sulfur (%)	-	0.1-0.2	1-3	1-3
Ash (%)	0.4-0.6	2-10	6-10	4-10
Volatile (%)	75-85	60-70	50-60	10-50
Moisture (%)	30-55	40-60	40-60	3-8
Heating Value* (kJ/kg)	18400- 19300	19700- 21300	20100- 24300	28500- 33100

* On a dry basis.

effectiveness of the pretreatment. Finally, the desired moisture content, usually less than 50% for peat fuel, is obtained by thermal drying.

The most energy intensive step is the last, thermal drying. It has been variously estimated that, depending on the system used, the heat required for thermal dewatering can range from 500kJ to 4000kJ per kilogram of water removed (Monenco, 1983). Rotary drum, flash or fluid bed dryers may be used to dry peat and are available commercially. These dryers are fueled by peat produced on site.

Rotary drum dryers used for peat drying are supplied by the M.E.C. company and consume 3552 kJ/kg of water evaporated. Flash dryers are available from Ahlstrom Company. They are rated at 3128 kJ/kg. Bell Engineering Works manufactures fluid bed dryers for peat which use 3340 kJ/kg of water evaporated.

To appreciate the energy cost of thermal drying consider a dryer consuming 3000 kJ/kg of water evaporated. To yield a product containing, say, 10% moisture content from a peat pressed to 70% moisture content, 2.2 kilograms of water must be evaporated per kilogram of dry peat. The dryer would consume 6600 kilojoules of energy. Producing a fuel grade product, heat content 22,000 kJ/kg of dry peat, more than 30% of the dry product would be burned.

Mechanical dewatering requires far less energy than thermal drying. Powered by electrical energy, presses consume in the order of 20 kJ per kg of water removed (Monenco, 1983). However, mechanical means will not bring the moisture content of untreated fuel grade peat below 55% (Punwani, 1983). This

barrier is usually attributed to the colloidal nature of peat which is considerable in highly decomposed fuel grade peat.

Conventional presses dewater peat by compressing it between either belts or rollers. Ingersoll-Rand manufactures a twin roller "Vari-Nip" filter press which can increase the solid content of raw peat to 30% (Monenco, 1983). Sulzer Brothers' "Multi-Nip" belt press produces 63% moisture content filter cakes of fuel grade peat. Centre de Recherche Industriel du Quebec has developed a rotary dewatering press which produces a product containing 32% dry solids (Smith, 1984).

1.3. Pretreatment

The objective of pretreatment is to reduce the cost of dewatering. Pretreatment processes reduce the hydrophilicity of peat and, therefore, facilitate mechanical dewatering. Increasing the amount of water removed during pressing decreases the burden of the more expensive thermal drying step. Rohrer (1981) performed an economic analysis on two dewatering processes, which differed only in one unit operation, the inclusion of thermal pretreatment. He concluded that thermal dryer costs (capital investment) and fuel consumption (of peat product) were two to three times higher with un-pretreated peat materials.

Two types of pretreatment have been used in commercial dewatering processes: chemical pretreatment and thermal pretreatment. Chemical pretreatment is, simply, the addition of "dewatering agents" to the raw peat before pressing. Thermal pretreatment, either wet oxidation or wet carbonization, is heating the raw peat before pressing.

Chemical pretreatment methods aim at destabilizing the colloidal nature of peat. A Finnish company reports that peat slurry can be mechanically dewatered to 55% moisture content in a belt press if electrolytes are added to the slurry (Punwani, 1983). However, most investigations into chemical pretreatment have only on a laboratory scale.

In a feasibility study by Cooper et al. (1985) a unique, multi-step dewatering process was developed. This process is represented in figure 1.1. In the process organic material dissolved in the expressed water is used as the substrate for a fermentation. The fermentation step removes dissolved organic components from the water and yields biological surface active agents (Mulligan and Cooper, 1985). These compounds, when added to the peat before pressing increase the amount of water removed. The chloride salt of chitosan, a polymer of glucose-amine found in the cell wall of many fungi, when converted to a cationic molecule, figure 1.2, proved to be the most effective dewatering agent. Using fuel grade peat from Barrington, Nova Scotia, more than 61% of the water was removed under optimum conditions.

Water removal data for several dewatering agents tested by Cooper et al. (1984) is shown in table 1.3. All of these compounds can be obtained from microorganisms. Lipase and esterase are enzymes. Samples treated with the two enzymes were incubated at 23C for 17 hours before pressing. The last compound shown in table 1.3 is a surfactant produced by the organism *Bacillus Subtilis*.

Synthetic surfactants and inorganic salts have also significantly enhanced the mechanical dewatering of fuel grade

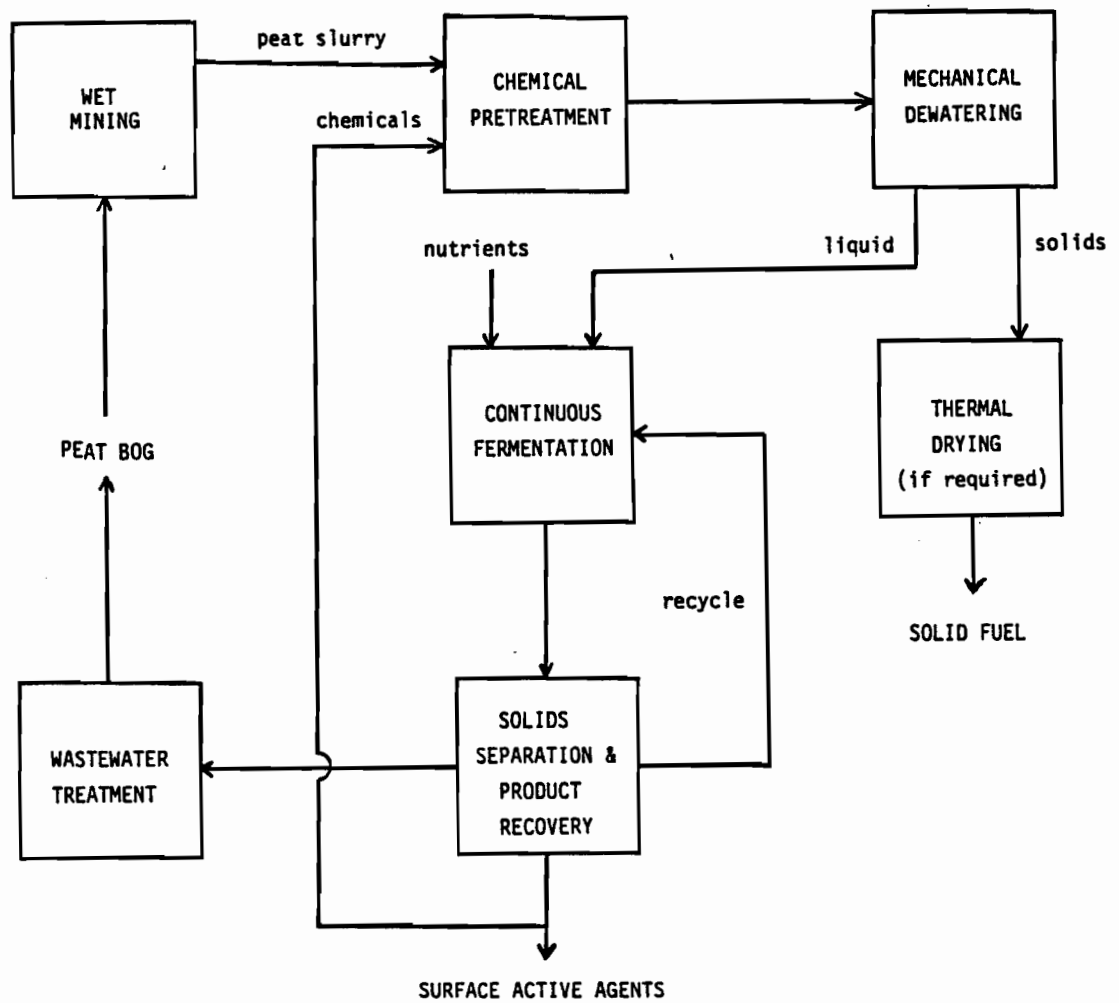


figure 1.1. Proposed dewatering process. (Cooper et al., 1985).

Figure 1.2. The Transformation of Chitosan to a Charged Polymer

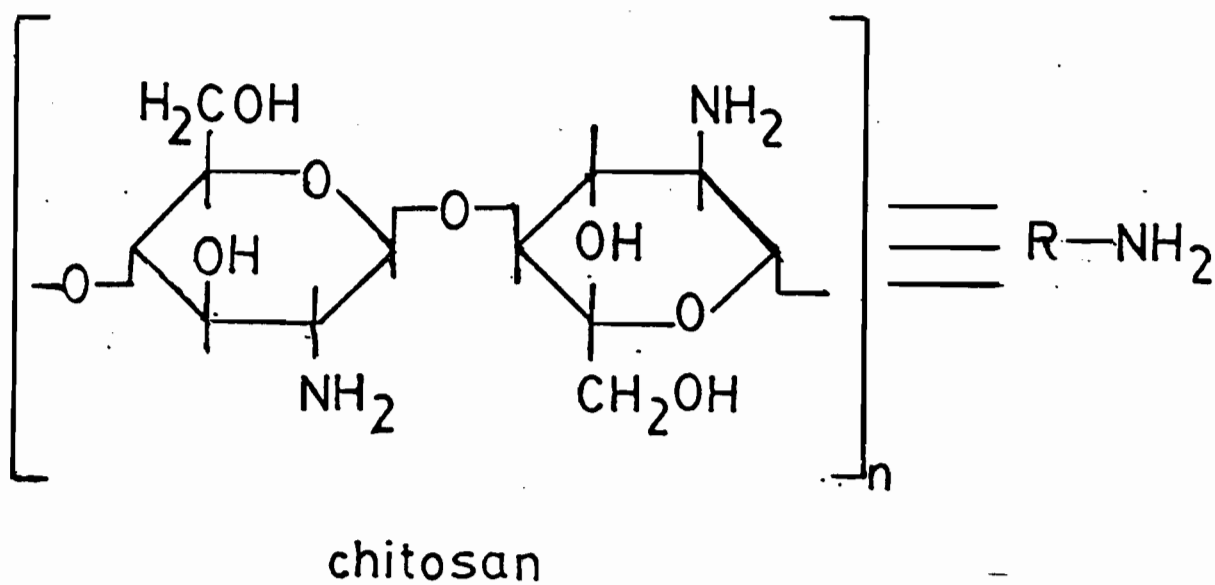
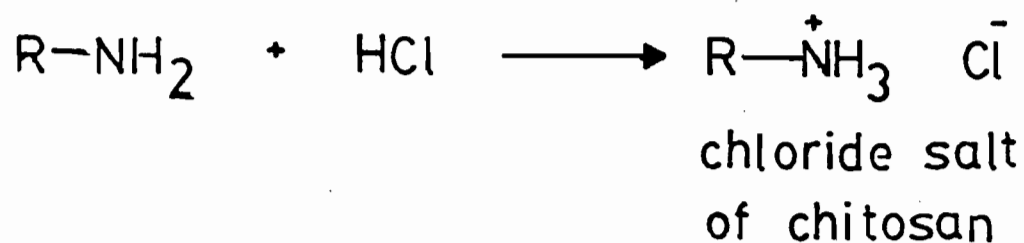


Table 1.3. Data for Several Dewatering Agents*

Dewatering Agent	Water Removed (%)	Initial Moisture (%)	Final Moisture (%)
Control	52.7	87.3	76.5
Chloride salt of chitosan	61.7	85.9	70.0
Lipase	58.8	87.3	73.9
Esterase	60.1	87.3	73.3
B. Subtilis precipitate	60.0	87.3	73.3

* An peat from Barrinton, Nova Scotia was used. The peat was pressed for 2.5 minutes at 19.6 atm.

peat. Exactly how these compounds affect the water retention properties of peat is not clear. However, many authors explain the results of chemical pretreatment in the frame of colloid chemistry.

Small negatively charged particles of peat possessing a large surface area to volume ratio are dispersed throughout the aqueous phase (Ayub and Sheppard, 1985). Destabilizing the dispersion by coagulation and/or flocculation aids the solid-liquid separation during pressing. Flocculation defines a bridging mechanism that enmeshes particles by adsorption into a floc that often has a higher settling velocity than the original unflocculated particle (The American Water Works Association, 1971). Flocculation can be enhanced by both cationic- and anionic-charged polymers. Coagulation describes a process whose primary force is electrostatic (Gilman et al., 1979). Addition of cationic material reduces electrostatic repulsions between colloids and allows the particles to aggregate.

Cooper (1985) has pointed out that (1) only cationic polyelectrolytes are effective, neutral and anionic polymers will actually inhibit dewatering, and (2) dewatering of decomposed peat, which is more colloidal in nature, is improved more dramatically when chemically pretreated than dewatering of less humified peat. Cooper (1985) also noted that pressed peat pretreated with the cationic polyelectrolyte derived from chitosan settled quicker when resuspended in water after pressing than untreated peat.

Sheppard and Ayub (1985) have shown that surface charge plays an important role in the dewatering of peat. Pretreating peat samples with a cationic surfactant, dodecyltrimethylammonium bromide, lowered the zeta-potential of peat particles and produced a corresponding increase in water released during pressing. In fact, when the concentration of surfactant added neutralized the surface charge of peat, i.e. at zero zeta-potential, maximum water removal occurred.

As early as 1850 it was known that the water-bonding structure of peat could be broken down by heating it (Chornet et al., 1981). Since the mid-nineteenth century peat has been thermally treated in a variety of ways. These include both batch and continuous processes, with and without oxygen and under varying degrees of heat and pressure. Current pretreatment processes fall into one of two generic categories, wet carbonization and wet oxidation.

In the wet carbonization process raw peat (solids content less than 15%) is heated at relatively low temperatures (180-350°C) in a pressurized reactor. Residence times are usually less than one hour. Heat is provided to the reactor from an external source, frequently a steam generator using a part of the product peat for fuel. Wet carbonization provides two benefits. It (1) promotes dewatering and (2) enhances the heating value of the peat by improving the oxygen to carbon ratio. Before entering the reactor the peat slurry is often preheated in an extensive and sometimes complex heat exchanger.

Wet oxidation processes attempt to reduce the external heating requirements by oxidizing (burning) a fraction of the

peat as a slurry. Burning can be initiated by preheating to the ignition point and then supplying a source of oxygen to the slurry. Once the reaction is started, the rate and extent of oxidation will determine the pretreatment temperature (Gallo and Sheppard, 1981). Overall thermal efficiency for wet oxidation processes is lower than for wet carbonization processes largely due to combustion of a large fraction of the feed during the former (Sheppard et al., 1985).

Table 1.4 (Monenco, 1983) highlights several parameters important to three commercial dewatering processes which apply heat pretreatment. Plans for construction in Sweden and the U.S.A. of plants using the JP Energy process, for which the North American licensee is Wheelabrator-Frye, have been drawn up. The Zimpro process, developed by Zimpro Inc. of Wisconsin, has been tested successfully on a laboratory scale. The same is true for the NRC/Sherbrooke work which uses high speed heating, compression and pressure release in a relatively low severity process.

Although the benefits of heat treatment have been known for over a century accepted mechanisms of how the peat is altered, chemically and/or structurally, have yet to be developed. This research is difficult because peat is a very heterogeneous matter. This heterogeneity is due to the wide range of plant matter that collects in a single bog and the large number of routes of decomposition which yield the formation of peat.

Chemically, peats are composed mostly of organic material. The small percentage of inorganic materials (2-10%)

Table 1.4. Comparison of Dewatering Processes

Process	Module Size	Reactor Conditions			Moisture Content	
	Size (k. tonnes per year)	Temp (C)	Press (kPa)	Time (min)	Filter Cake (%)	Product (%)
JP-Energy	100-300	210	2500	30	50	25.84
Zimpro	23-27	150	2170	30	50	20.39
NRC/ Sherbrooke	—	190	4140	1	50	—

usually present does not have a significant effect on dewatering characteristics. The organic material exists as an enormously complex mixture derived from chemically stable residues of plant tissue.

A number of methods have been used to chemically classify peat substances. One system uses several broad categories to group the organic components in peat. Each category is characterized by its solubility in a standard solvent (Fuchsman, 1980). Those substances which can be dissolved by suitable organic solvents are bitumens. Bitumens encompass waxes and "resinous" materials. A second category, humic acids, are soluble in aqueous base and precipitated by acidification of the alkaline extract. A third group which is characterized by its solubility in acid includes carbohydrates related to cellulose and some protein like substances. The fourth group consists of lignins which are soluble in strong base and are characteristically phenolic in their reactions.

In the low temperature processes studied here only three of the above classes of compounds are of interest: the humic acid fraction, the carbohydrates and the lignins. It is these three classes of peat constituents which are most likely to undergo chemical changes during heating. The bitumens are among the last distinguishable components to break down or volatilize when peat is exposed to heat. Generally, they begin to undergo chemical changes at 350C, however, physical changes may occur at lower temperatures (Fuchsman, 1980).

Because there exist no accepted conventions on procedures for separating peat compounds, and, furthermore, because each

peat sample is different in its chemical make up, the distinction between classes and even the characterization of the classes is often very subjective. Many authors tend to define the categories in their own way. However, an attempt to define the humic acids, lignins and carbohydrates commonly found in peat follows.

Humic acids which are extracted from peat by dilute alkaline media, are amorphous, brown or black, hydrophilic, acidic, polydisperse substances of molecular weights ranging from several hundreds to tens of thousands (Schnitzer and Kahn, 1972). This fraction can represent up to 40% of the dry weight of peat. After alkaline extraction of peat, acidification of the solution causes precipitation of the humic acids, however, another class of compounds, fulvic acids, remain in solutions. Fulvic acids are closely related to humic acids. They differ principally in molecular weight and functional group content, with the fulvic acid fraction having a lower molecular weight but a higher content of oxygen containing functional groups per unit weight. A wide variety of chemical degradation experiments on humic acids and fulvic acids have shown that the major degradation products are phenolic and benzenecarboxylic acids. Schnitzer (1985) has suggested that these benzene carboxylic acids and phenolic acids are the "building blocks" of humic and fulvic acid. He believes that the molecular forces holding the "building blocks" together are similar in both fractions, consisting mainly of hydrogen bonds, van der Waal's forces and pi-bonding.

Lignins are closely related to humic acids chemically; both substances are polyphenolic in character (Fuschman, 1980). The lignin fraction can be extracted from peat with a strong base. Between 10 and 15 percent of the dry weight of peat is lignin. The fundamental structural unit of lignin is a phenol substituted in the para-position by an n-propyl chain and in one or both ortho-positions by methoxyl groups. These units can be linked to each other in many ways, usually through oxygens bonded to the carbons of the propyl group.

Because both the lignin fraction and the humic acid fraction can be extracted from peat with base, some authors do not distinguish between the two. The principal differences are (1) the predominant functional group in humic acid is the carboxyl group (about 8 mmoles/gram) which is not found in lignin's structure, (2) the solubility in dilute alkali of humic acid but not of lignin and (3) the high concentration of methoxyl groups in lignin; in humic acid the methoxyl group concentration is about 0.2 mmol/gram.

Carbohydrates are the third group of peat compounds to be considered. The most abundant carbohydrates found in peat can be further separated into four groups. They are hemicellulose, chitin, glycoside and cellulose. Hemicelluloses are highly complex substances. Each molecule consists typically of 200-300 sugar monomer units. The hydrolysis of peat hemicelluloses yields glucose, fructose, mannose, galactose and xylose among others. Chitin, which is commonly found in the cell walls of microorganisms, is a linear polymer of N-acetyl glucoseamine. The third type of carbohydrate found in peat,

glucosides, constitute a varied group of substances consisting of sugars bonded to noncarbohydrate molecules. Finally, cellulose, which is a polymer of glucose molecules, is the principle material found in the fibrous parts of plants.

In typical wet carbonization processes (temperatures above 180C) it is known that the chemical structure of peat undergoes significant change. It is these chemical changes which are believed to facilitate the de-watering of heat treated peat. Less work has been done on heat treatment processes below 200C. However, even at 75C peat de-watering is enhanced significantly (Cooper et al., 1985). Only a small amount of research has been devoted to characterizing the chemical reactions peat undergoes when heated under these low severity conditions.

Chornet et al. (1981) studied a low severity wet carbonization process in which peat was heated to 90C and 190C for residence times of approximately one minute. Their main goal was to use shorter residence times to achieve breakdown of the hydrophilic nature of the peat substrate. Specifically, they believe that short residence times are sufficient for hydrolytic reactions to occur. These reactions are reported to be acid catalyzed and cause the destruction of the hydrogen bonding which exists in the carbohydrate fractions, hemicellulose and cellulose. The principle chemical changes recorded in this process where: (1) a fifty percent reduction in the base soluble-acid soluble material (this corresponds to the fulvic acid fraction described earlier) and (2) an increase in carbon content and a decrease in oxygen content in the processed peat.

Fuchsman (1980) describes the chemical processes peat undergoes during heating in considerable detail. The effect of wet carbonization (temperatures of 180 - 220C) on the dewatering properties of peat are again attributed to the breakdown of the highly hydrogen bonded molecular structure of peat. The principle chemical changes which occur during heating of peat at low temperatures are the decarboxylation of the carboxylic acid groups in the hemicelluloses and humic acids. The reactions up to 300C, he explains, are easily idealized by low-temperature reaction mechanisms, e.g. loss of carbon dioxide by decarboxylation of acid groups, dehydration by splitting off water from hydroxylated aliphatic structures and generation of low-molecular-weight alcohols by simple rearrangement of esters.

Schnitzer and Kahn (1972) have done extensive work with humic acid and fulvic acid. Thermal degradation studies were done with both substances. Analysis of DTG curves lead them to attribute low-temperature peaks to the elimination of functional groups. The most dramatic change in functional group concentration observed below 200C was loss of approximately 10% of the carboxylic acid groups in fulvic acid. The main reactions governing the pyrolysis of humic acid are believed to be (1) dehydrogenation (up to 200C); (2) a combination of decarboxylation and dehydration (between 200 and 250C); (3) continuous dehydration. The main reaction determining the pyrolysis of fulvic acid is dehydration.

1.4. Expressed water quality

In developing a peat dewatering process a parameter which must be assessed is the quality of the expressed water. The

quantity of effluent released in a process designed to de-water fuel grade peat is generally 8 to 9.5 times the weight of the dried product. A medium size plant of 300,000 dry T/a would annually discharge 2.7 million cubic meters at a rate of 360 cubic meters per hour (Monenco, 1983). Two types of peat extract present in the effluent must be considered: the suspended solids and the dissolved organics.

In de-watering processes using strictly mechanical and/or thermal evaporation means, the liquid effluent is considered to be chemically and biochemically unchanged. In such cases only the suspended solid concentration is important. However, the typical wet carbonization process subjects peat to high temperatures and pressures. Decarboxylation occurs and peat is broken down into lower molecular weight compounds. A good amount of data has been compiled showing that many of these organic molecules are dissolved in the water released by thermal pretreatment de-watering processes (Washburn & Gillis Associates Ltd., 1983). The concentration of organic molecules must be monitored to insure that toxic effluent is not returned to the environment.

2. Objectives

In order to produce an economical fuel from peat an energy efficient dewatering method is essential. Mechanical pressing consumes relatively low quantities of energy. Pretreating the peat either mechanically or chemically facilitates mechanical pressing. Therefore seven compounds possessing a range of properties were used in pretreatment studies with the intent to determine their optimum effectiveness and how they alter the colloidal properties of peat.

The second objective was to determine the effects of low temperature heat pretreatment on the dewatering of peat and to gain some insight into how heat effects the water retaining structure of peat. Combined heat and chemical pretreatment was to be studied.

Peat dewatering processes yield large quantities of expressed water. Often, depending on the form of pretreatment, the water is toxic. Therefore, the third objective was to monitor the quality of peat water removed from pretreated peat and assess, to some extent, the possibility of growing microorganisms on this water.

3. Materials and methods

3.1. Mechanical press

The blue print for a peat press die was obtained from the Ontario Research Foundation. The die (figure 3.1) consists of a base with a shallow excavation in which a cylinder is rested. A sintered steel filter and a support disc are inserted into the cylinder and a piston is used to apply pressure to the peat samples.

In order to apply a maximum of 30 atm of pressure on the samples, a pneumatic system, with a pneumatic cylinder of 4.5 inches, connected to a 96 psi air line was employed. A double acting cylinder was chosen so that it could be used to insert as well as retract the piston of the press.

3.2. Standard pressing procedure

The peat used in all experiments was taken from a bog in Barrington, Nova Scotia. An analysis is presented in table 3.1. To a peat sample of approximately 25 grams was added the stated amount of dewatering agent. This mixture was stirred in a ball mill for 5 minutes. The sample was then transferred to the peat press where it was pressed for 3.0 minutes at 19.3 atm. (For the determination of these parameters please see Cooper et al., 1984). After pressing, the sample was immediately weighed. Studies on the reproducibility of pressing experiments proved % moisture content values to be accurate to within $\pm 2\%$ absolute, using the 95% confidence interval.

3.3. Control pressing procedure

The control procedure was the same as the standard

Figure 3.1. Diagram of Peat Press

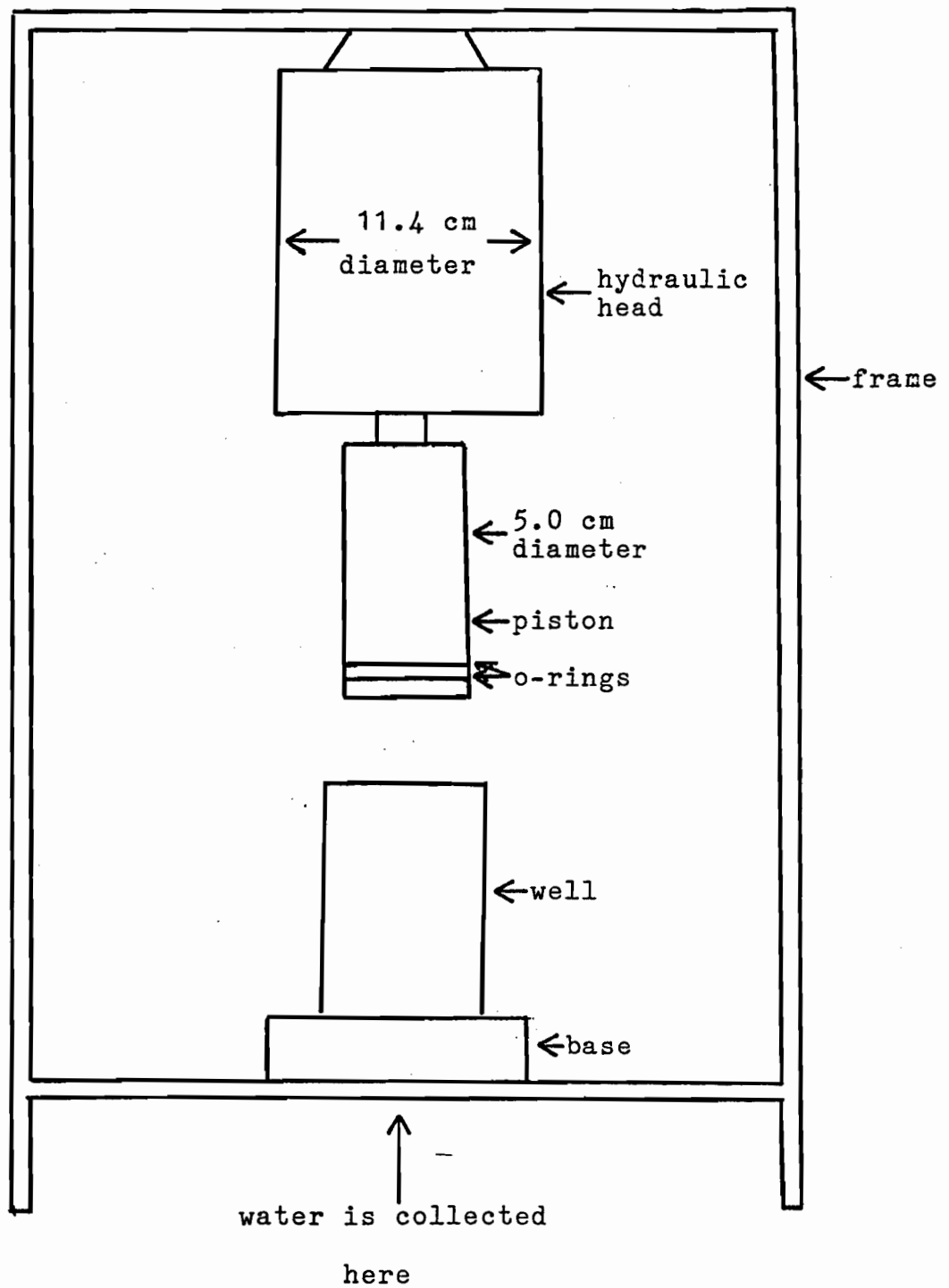


Table 3.1. Analysis of the Barrington, N.S. peat

Component Analyzed	
Ash content (%)	11.8
Carbon (%)	54.9
Hydrogen (%)	5.0
Oxygen (%)	25.8
Sulfur (%)	0.5
Nitrogen (%)	2.0
Initial Moisture (%)	86-92
Degree of Decomposition (von Post Scale)	H9
Caloric value (MJ/kg) (moisture free basis)	23.0

pressing procedure except the peat was pressed without pretreatment.

3.4. Calculations of the water content of peat

All pressing studies were done in triplicate. The values are an average of the results from three pressings.

Water Content of the Unpressed Peat

$$\text{water content} = \frac{\text{wt of water in sample}}{\text{wt of dry peat in the sample}}$$

Water Content of Pressed Peat Samples

$$\text{weight of water in pressed peat} = \text{wt of pressed sample} - \text{wt of pressed sample after drying}$$

$$\% \text{ moisture content of pressed peat} = \frac{\text{wt of water in pressed sample}}{\text{wt of pressed sample}} \cdot 100$$

$$\text{water content after pressing} = \frac{\text{wt of water in pressed sample}}{\text{wt of dry peat in pressed sample}}$$

$$\text{weight of water removed by pressing} = \text{water content of unpressed peat} - \text{water content of pressed peat}$$

$$\% \text{ water removed} = \frac{\text{wt of water removed by pressing}}{\text{wt of water in unpressed sample}} \cdot 100$$

3.5. Preparation of the chloride salt of chitosan

The chitosan starting material was purchased from the Sigma Chemical Company, St. Louis, Mo. 5 g of chitosan salt were added to 50 mL of hydrochloric acid. The mixture was stirred and heated at 70°C for 2h. The solution was then cooled by means of an ice bath and acetone was added until a white precipitate appeared. The precipitate was collected by vacuum filtration and washed twice with 100mL portions of acetone.

The white solid was air dried.

3.6. Other dewatering agents

The two synthetic flocculating agents, Praestol 444K (high charge cationic) and Praestol 411K (low charge cationic), and the salts, dodecyltrimethylammonium bromide, ferrous chloride, aluminum chloride and ammonium chloride were purchased from the Sigma Chemical Company.

3.7. Determination of concentration of suspended peat solids

Expressed water from an untreated peat sample containing suspended peat particles was centrifuged to separate out the solids. Five solutions of various concentrations of the particles were then prepared. Immediately after vortexing the solutions for 30 seconds, to insure uniformity, several milliliters were transferred to a cuvette. Absorbance measurements were taken on the Hitachi Perkin-Elmer double beam spectrophotometer at 600nm. Using linear interpolation and this data (Figure 3.2) provides an easy method for determining suspended solids concentrations in peat water samples.

3.8. Particle settling measurement on pressed peat

After a peat sample had been pressed, a 50 mg section was taken and added to 10 ml of water. The solution was vortexed for 30 s in order to resuspend the peat particles and then transferred to a cuvette. A reading was taken immediately and again after one hour, on the spectrophotometer. The weight percent of particles settled was then calculated as follows:

I = initial reading

F = final reading (after 1 h)

% settled, 60 min = $(F - I) / I$

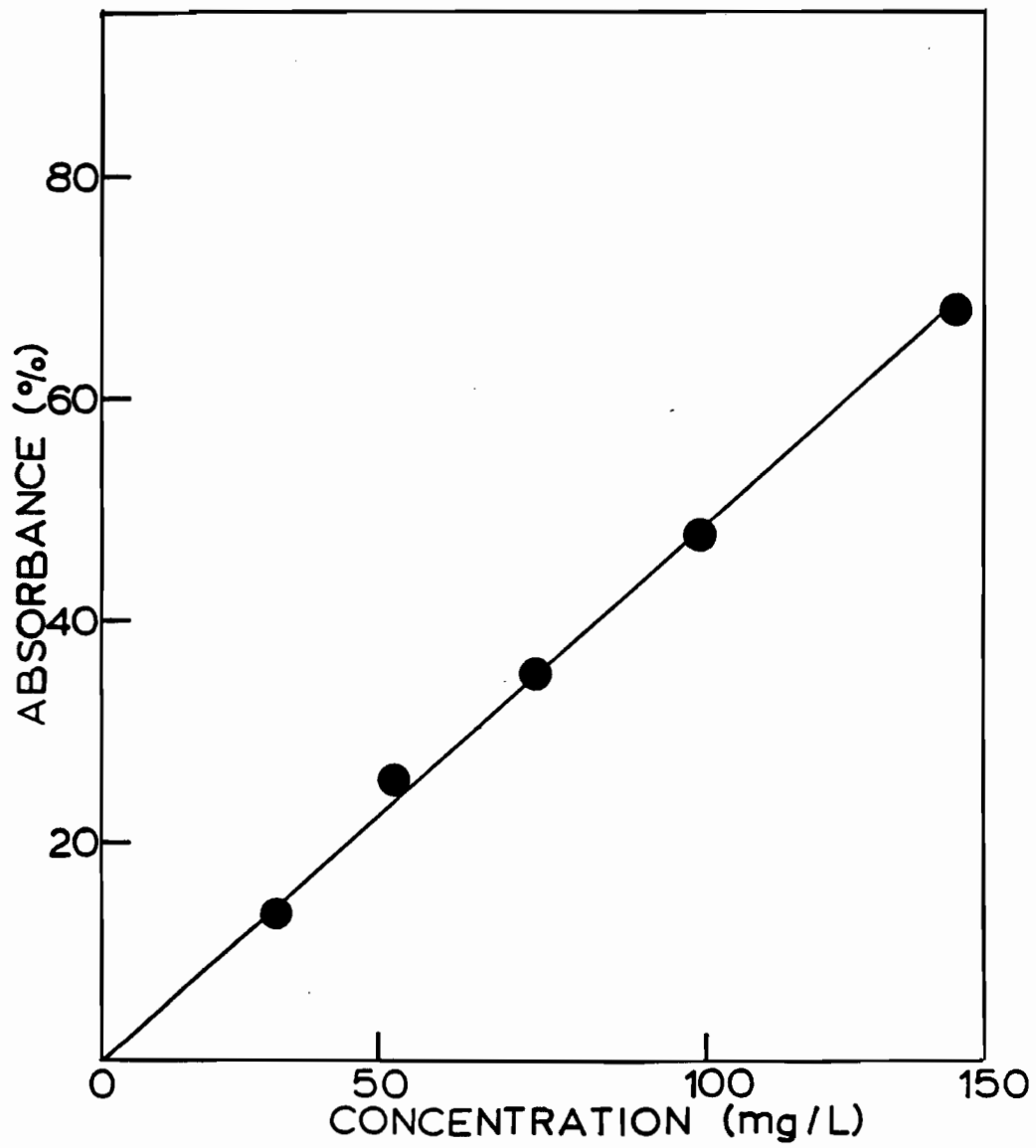


Figure 3.2. Percent absorbance (600nm) of suspension of peat particles in water versus concentration.

Approximately 20 milliliters of expressed water were recovered from pressing after pretreatment. One milliliter was diluted with 9 ml of distilled water. After vortexing for 30 s part of the solution was transferred to a cuvette. An immediate reading was taken on the spectrophotometer and the initial concentration of suspended solids was calculated. One hour later a second reading was taken. The percent settled after one hour was then calculated as described above.

3.10. Standard heating procedure

A Parr 4501 two-liter pressure reactor was used to heat peat to temperatures between 25C and 200C. The jacket was first preheated. After a determined amount of time the pressure vessel was placed into the jacket and allowed to reach the desired temperature. After the heat exposure, cooling to room temperature was performed rapidly by immersing the vessel in cold water. The temperature was monitored by a Truetest dial thermometer placed in the thermowell which was immersed in the peat. After removal from the vessel the peat was pressed and its moisture content determined.

3.11. Heating with an inert atmosphere

Before being placed into the heating jacket the pressure vessel was flushed with nitrogen gas for fifteen minutes and then sealed. The nitrogen was bubbled through distilled water to prevent it from dehydrating the peat. The standard heating procedure was then followed.

3.12. Heating with stirring

During the standard heating procedure the peat was stirred at 100 rpm with the propeller stirrer attached to the head of the pressure vessel.

3.13. Growth inhibition studies

A study of the inhibitory effects of peat run-off water and pressed peat water was preformed. A variation of the Kirby-Bauer method for determining bacterial sensitivity to antibiotics was employed to determine the inhibitory effects of the peat waters on various microorganisms. The revised method involved growing pure colonies of the various organisms in shake flasks and then innoculating standard agar plates with the organism to be tested. Then, sterile filters, which had been pre-soaked in the pressed peat water or the peat run-off water, were placed on the plates in various locations. The degree of inhibition to growth was judged as follows. First, the area of clearing was calculated after 24 hours. This gave a rough initial indication of how sensitive the organism was to the peat water. The larger the area of clearing, the more the organism was inhibited by the peat water. Then, the area of clearing was recalculated after 48-72 hours. This measurement determined if an organism could adapt to the new environment and begin to grow again, normally.

3.14. Toxicity of expressed water and heat content of heat treated peat

Dried peat samples which had been exposed to a variety of pretreatment processes were sent to the NRC Atlantic Research Laboratory (Peat Program) for determination of calorific value. Expressed peat water samples were sent to the Ontario Research Foundation for "Microtox" tests.

Chapter 4. Results

4.1. Chemical pretreatment

Cationic polymers, inorganic electrolytes and cationic surfactants enhance the mechanical dewatering of fuel grade peat (Cooper et al., 1985). Several compounds, each from one of these categories, were investigated. To optimize the effectiveness of the dewatering agents each was added to raw peat samples at various weight ratios (weight of additive/-weight of dry peat) before pressing. The moisture content and particle settling rate of the pressed samples were measured.

4.1.1. Polyelectrolytes

Three polyelectrolytes were tested. Two of these, Praestol 411K and Praestol 444K, are commercially available synthetic polymers while the third is the chloride salt of protonated chitosan. Peat samples were treated with the polyelectrolytes and pressed under standard conditions. A 50 milligram section of the pressed peat was resuspended in water and the settling of the particles was monitored for one hour. The remainder was dried to a constant weight and the quantity of water removed was calculated.

Table 4.1 shows the results of additions of chitosan hydrochloride. When peat was mechanically pressed without any chemical addition only 32.1% of the water was removed. When an optimal ratio of chitosan salt was added (0.02 weight chitosan salt / weight of dry peat) the water removal improved to 63.9%. Increases in this ratio yielded steadily improved dewatering up to the maximum. Peat treated with a ratio greater than this optimum amount of chitosan produced increasingly worse results.

Table 4.1. The effect of chitosan hydrochloride on mechanical dewatering.

Ratio of chitosan to dry peat (wt/wt)	Amount of water removed (kg water/ kg dry peat)	Water removed (%)	Moisture content of pressed peat (%)
Control	2.34	32.1	83.2
0.004	3.25	44.7	80.1
0.019	4.68	64.3	72.1
0.030	4.65	63.9	72.4
0.042	4.49	61.7	73.6
0.050	4.43	60.9	74.0

In figure 4.1 the data from table 4.1 is plotted. Also plotted is the settling data from resuspended pressed samples which had been treated with chitosan salt. The maximum settling rate occurred at a chitosan salt ratio that was very close to the chitosan ratio where the maximum amount of water removal occurred. This figure shows that the settling rate curve and the water removal curve follow a similar pattern. The parameters increase at lower ratios of chitosan and each reaches a maximum. After this maximum, each begins to decrease.

The same investigation was undertaken with Praestol 411K and Praestol 444K to determine whether this relationship between water removal and particle settling rate was unique to chitosan or whether it was common to other polyelectrolytes. Results for the high charge density, cationic Praestol 444K are presented in table 4.2. As the amount of Praestol 444K added to a peat sample is increased, the water removed increased, reaching a maximum of 62.4% at a ratio of 0.015 wt additive / wt dry peat. When more than 0.015 grams of Praestol 444K per gram of dry peat were added less than 62.4% of the water was released. Thus following a pattern similar to the chitosan data. However, increasing the Praestol 444K ratio above the optimum yielded water removal data which drops off more dramatically than the chitosan water removal data.

The data in table 4.2 along with settling data for pressed samples which had been treated with Praestol 444K is plotted in figure 4.2. The settling curve again has a shape similar to the water removal curve. Resuspended pressed peat treated to

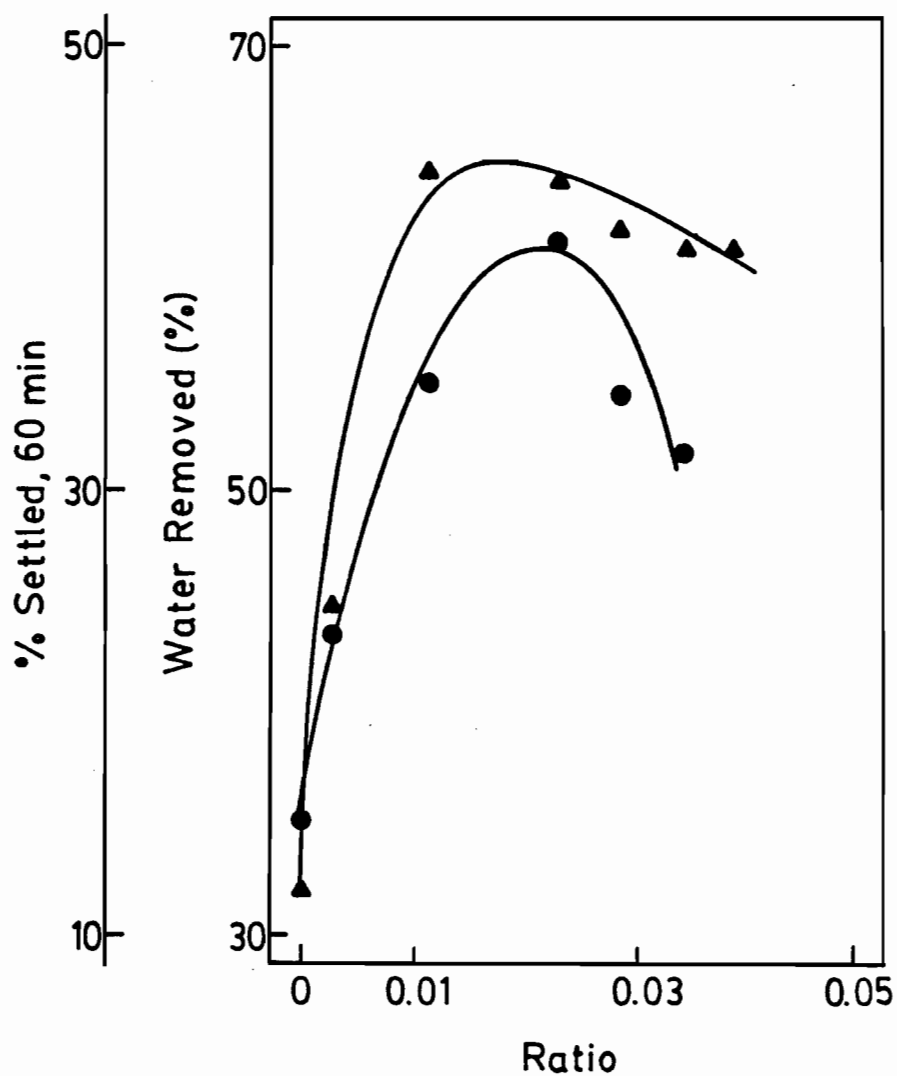


Figure 4.1. Enhancement of water removal from peat treated with chitosan. Water removed (▲) and percent settled after 60 min (●) versus the ratio of chitosan to dry peat in the sample (wt/wt).

Table 4.2. The effect of Praestol 444K on mechanical dewatering.

Ratio of Praestol 444K to dry peat (wt/wt)	Amount of water removed (kg water/ kg dry peat)	Water removed (%)	Moisture content of pressed peat (%)
Control	2.34	32.1	83.2
0.004	3.97	54.5	76.8
0.015	4.54	62.4	73.3
0.031	4.02	52.2	76.5
0.039	3.21	44.1	80.3
0.057	2.62	36.0	77.6

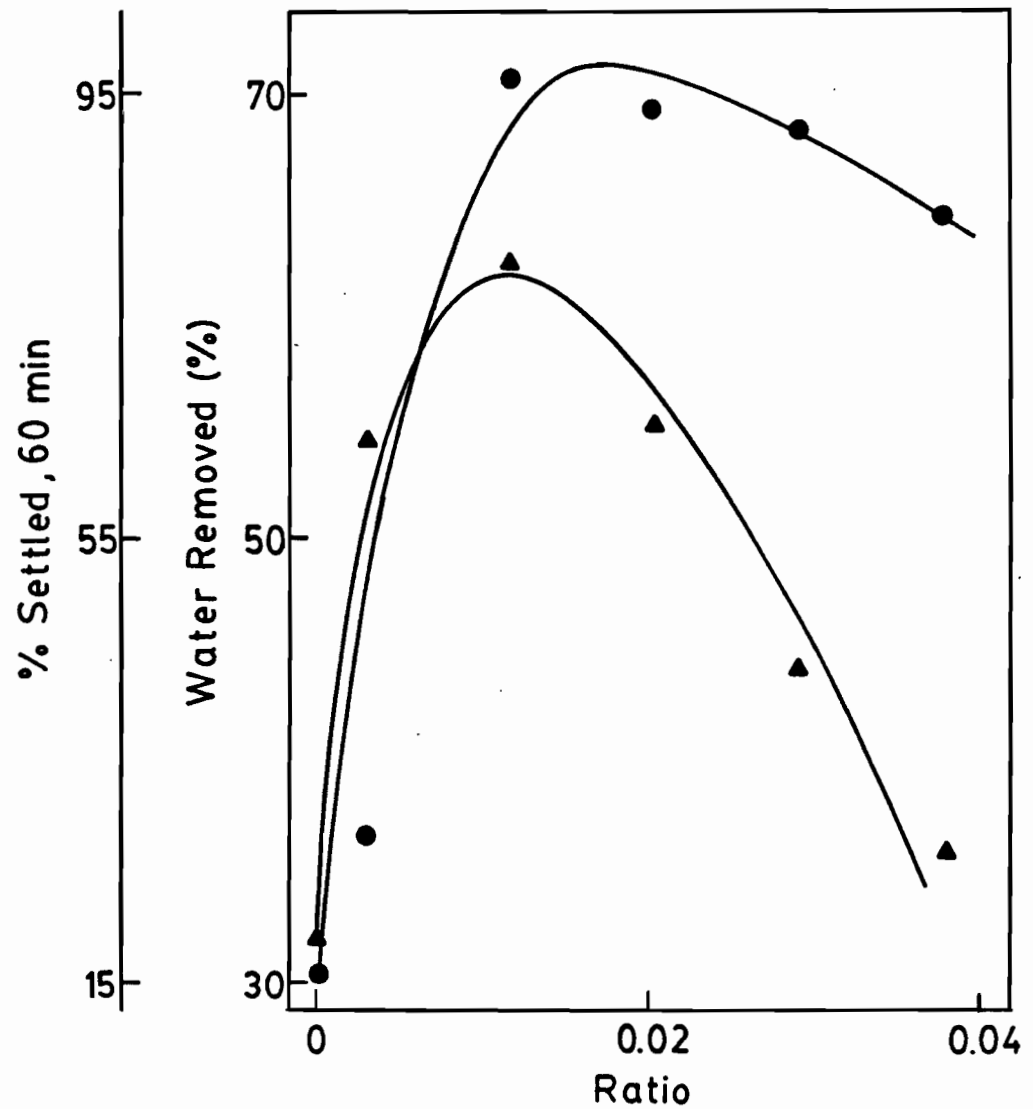


Figure 4.2. Enhancement of water removal from peat treated with Praestol 444K. Water removed (▲) and percent settled after 60 min (●) versus the ratio of Praestol 444K to dry peat in the sample (wt/wt).

a ratio of 0.015 settled fastest, 95.5% after one hour. This ratio is exactly the optimum Praestol 444K ratio for water removal. Further comparison of water removal and particle settling indicates a correlation between these two parameters.

Pressed samples treated with Praestol 444K were observed to be very "sticky" or "gummy". This was not observed for the chitosan samples.

Data for the low charge density cationic polyelectrolyte, Praestol 411K is shown in table 4.3 and plotted in figure 4.3. This polymer was not as effective at enhancing dewatering as Praestol 444K or the chloride salt of chitosan. The maximum water removal with mechanical pressing, 47.7%, occurred at a ratio of 0.040. The water removal curve has a shape similar to the water removal curves for the two other polyelectrolytes, however, it does not correlate well with the settling curve. An optimum settling ratio was not measured in the range of ratios tested.

4.1.2. Monomeric electrolytes.

Four salts were studied, three inorganic salts, ammonium chloride, ferric chloride and aluminum chloride, and one quaternary ammonium salt, dodecyltrimethylammonium bromide (DTAB), which has surfactant properties. Each compound was again added at various weight ratios (weight of additive / weight of dry peat) in order to characterize and optimize their effect on water removal and pressed peat particle settling.

The results for ammonium chloride are shown in table 4.4. This salt was not as effective a dewatering agent as chitosan or Praestol 444K. Maximum water removal was 55.2%. This is a

Table 4.3. The effect of Praestol 411K on mechanical dewatering.

Ratio of Praestol 411K to dry peat (wt/wt)	Amount of water removed (kg water/ kg dry peat)	Water removed (%)	Moisture content of pressed peat (%)
Control	2.45	33.7	82.9
0.004	2.53	34.8	82.6
0.010	3.04	41.8	80.9
0.016	3.38	46.4	79.6
0.026	3.29	45.2	80.0
0.040	3.47	47.7	79.2
0.052	3.41	46.8	79.5
0.056	3.43	47.1	79.4
0.063	2.82	38.7	81.7

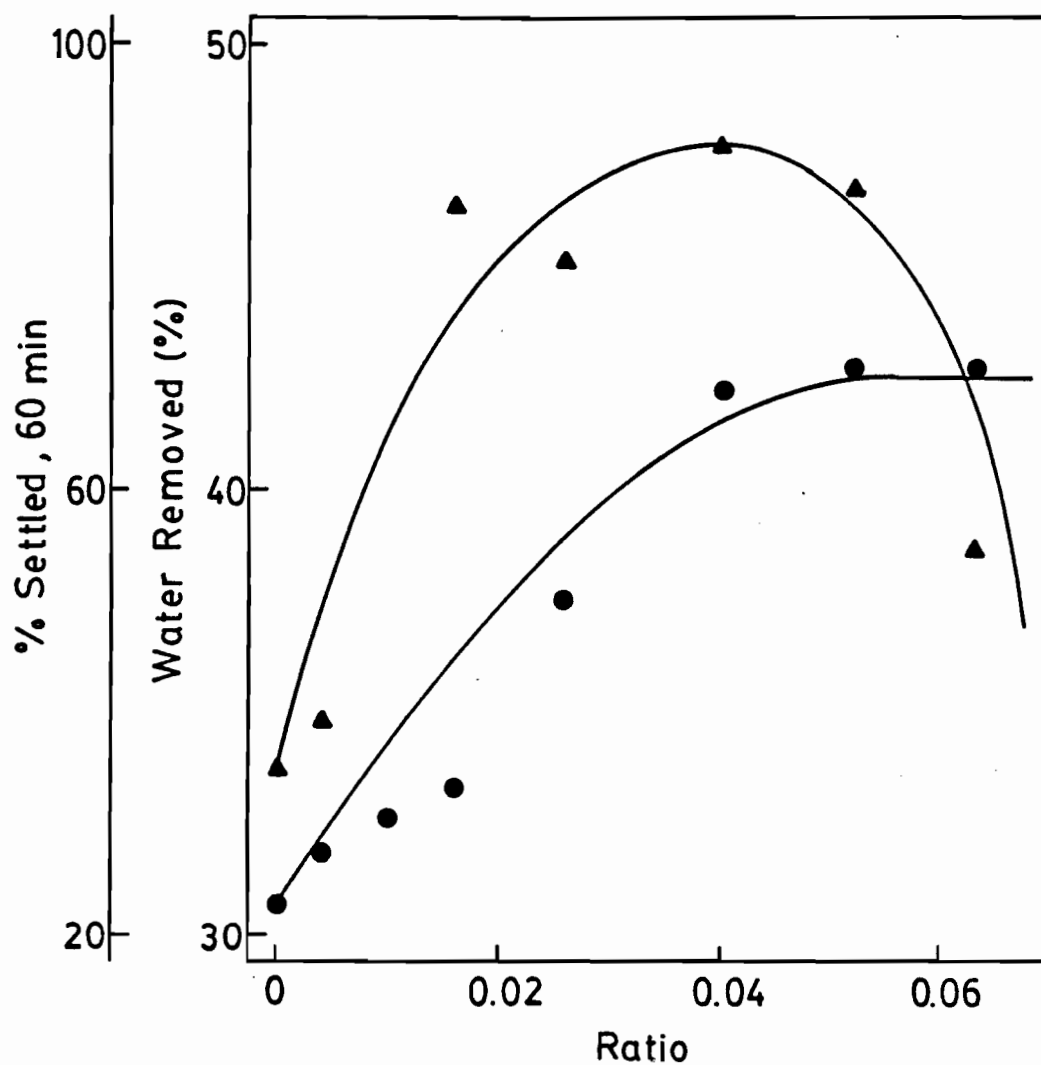


Figure 4.3. Enhancement of water removal from peat treated with Praestol 411K. Water removed ($\blacktriangle$) and percent settled after 60 min ($\bullet$) versus the ratio of Praestol 411K to dry peat in the sample (wt/wt).

Table 4.4. The effect of ammonium chloride on mechanical dewatering.

Ratio of ammonium chlo- ride to dry peat (wt/wt)	Amount of water removed (kg water/ kg dry peat)	Water removed (%)	Moisture content of pressed peat (%)
Control	2.78	38.2	81.8
0.004	3.80	52.2	77.7
0.010	4.02	55.2	76.5
0.017	3.80	52.2	77.7
0.030	3.85	52.8	77.4
0.043	3.80	52.2	77.7
0.048	3.77	51.8	77.8

significant improvement in dewatering when compared to the control, 38.2%. The settling rate of pressed peat particles treated with ammonium chloride was also much lower than for the polyelectrolytes (figure 4.4). This figure shows that both water removal and particle settling increase, reach a maximum and then drop off as the ratio is increased, similar to the pattern observed for chitosan and Praestol 444K. However, the drop off is much less pronounced and the water removal curve does not correlate with the particle settling curve.

When 0.062 grams of ferric chloride / gram of dry peat were added before pressing, 68.1% of the water was removed, 32.8% more than the control (table 4.5). This result was impressive but it was attained at a much higher concentration of additive than for previously studied molecules. Above this ratio ferric chloride's effectiveness leveled off (figure 4.5). At 0.062, 64% of the suspended particles settled after one hour, a significantly greater percent than the control. Particle settling continued to increase when a larger ratio was used, yielding a curve which does not correlate with the water removal curve.

Water removal data for aluminum chloride is presented in table 4.6. The water removal was enhanced (maximum of 63.6% at 0.018) and the particle settling was also excellent. However, figure 4.6 indicates that these parameters do not have their maximum at the same additive ratio. There is no correlation between the curves.

The surfactant DTAB proved to be the most effective dewatering agent of all those tested. When 0.055 grams of DTAB

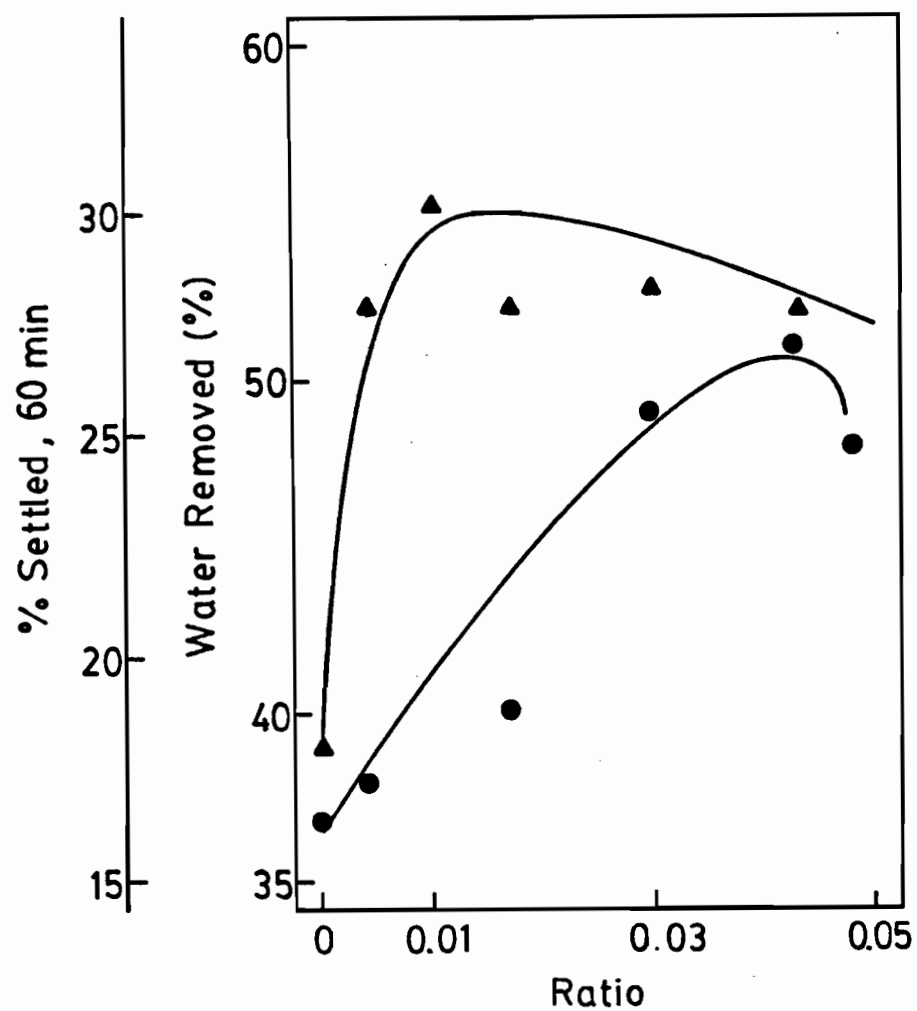


Figure 4.4. Enhancement of water removal from peat treated with ammonium chloride. Water removed (▲) and percent settled after 60 min (●) versus the ratio of ammonium chloride to dry peat in the sample (wt/wt).

Table 4.5. The effect of ferric chloride on mechanical dewatering.

Ratio of ferric chlo- ride to dry peat (wt/wt)	Amount of water removed (kg water/ kg dry peat)	Water removed (%)	Moisture content of pressed peat (%)
Control	2.64	36.3	82.3
0.004	3.78	51.9	77.8
0.018	4.25	58.4	75.2
0.032	4.32	59.3	74.8
0.043	4.58	62.9	72.9
0.058	4.58	62.9	72.9
0.062	4.96	68.1	69.9
0.070	4.62	63.5	72.7

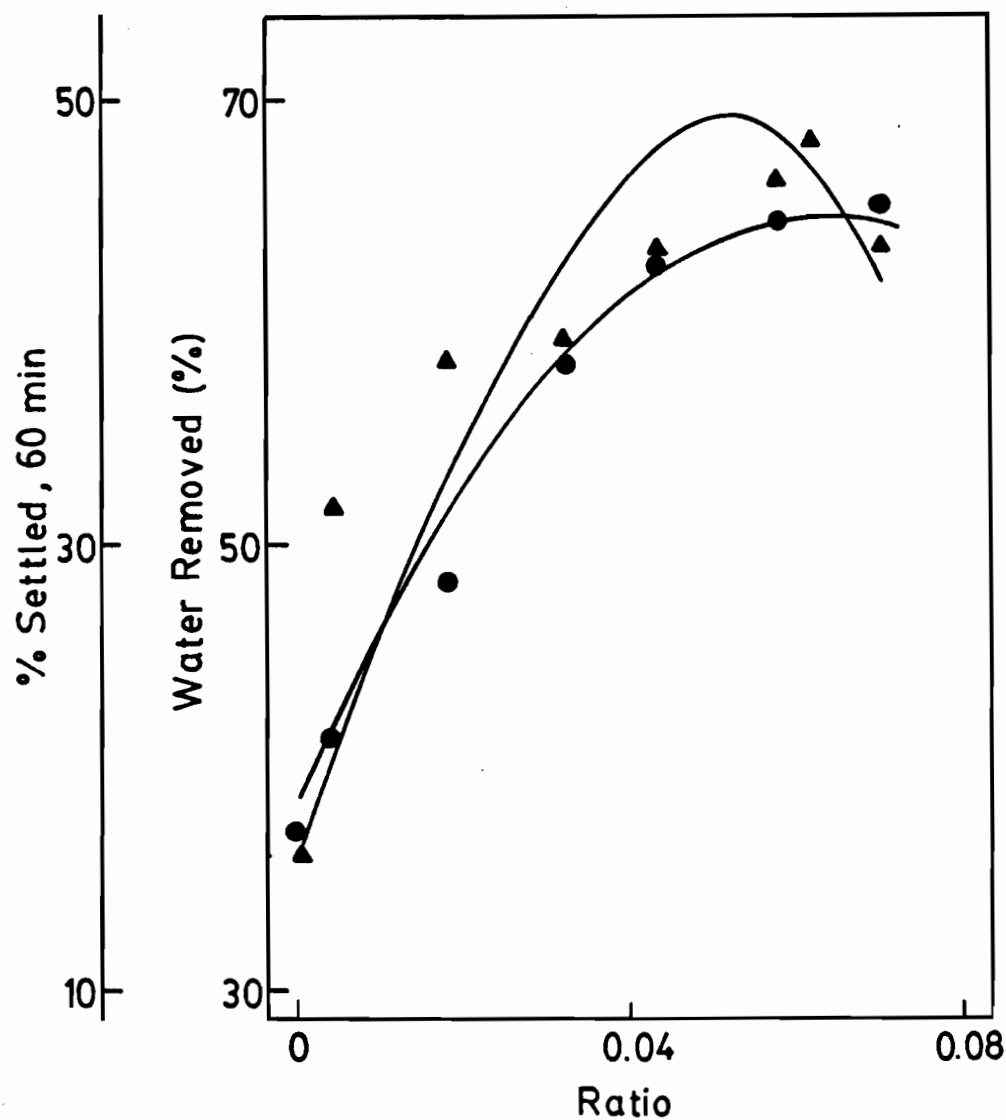


Figure 4.5. Enhancement of water removal from peat treated with ferric chloride. Water removed (▲) and percent settled after 60 min (●) versus the ratio of ferric chloride to dry peat in the sample (wt/wt).

Table 4.6. The effect of aluminum chloride on mechanical dewatering.

Ratio of aluminum chlo- ride to dry peat (wt/wt)	Amount of water removed (kg water/ kg dry peat)	Water removed (%)	Moisture content of pressed peat (%)
Control	2.62	36.0	82.3
0.005	4.44	61.0	74.0
0.018	4.63	63.6	72.6
0.032	4.59	63.0	72.9
0.048	4.47	61.4	73.8
0.054	4.31	59.2	74.8

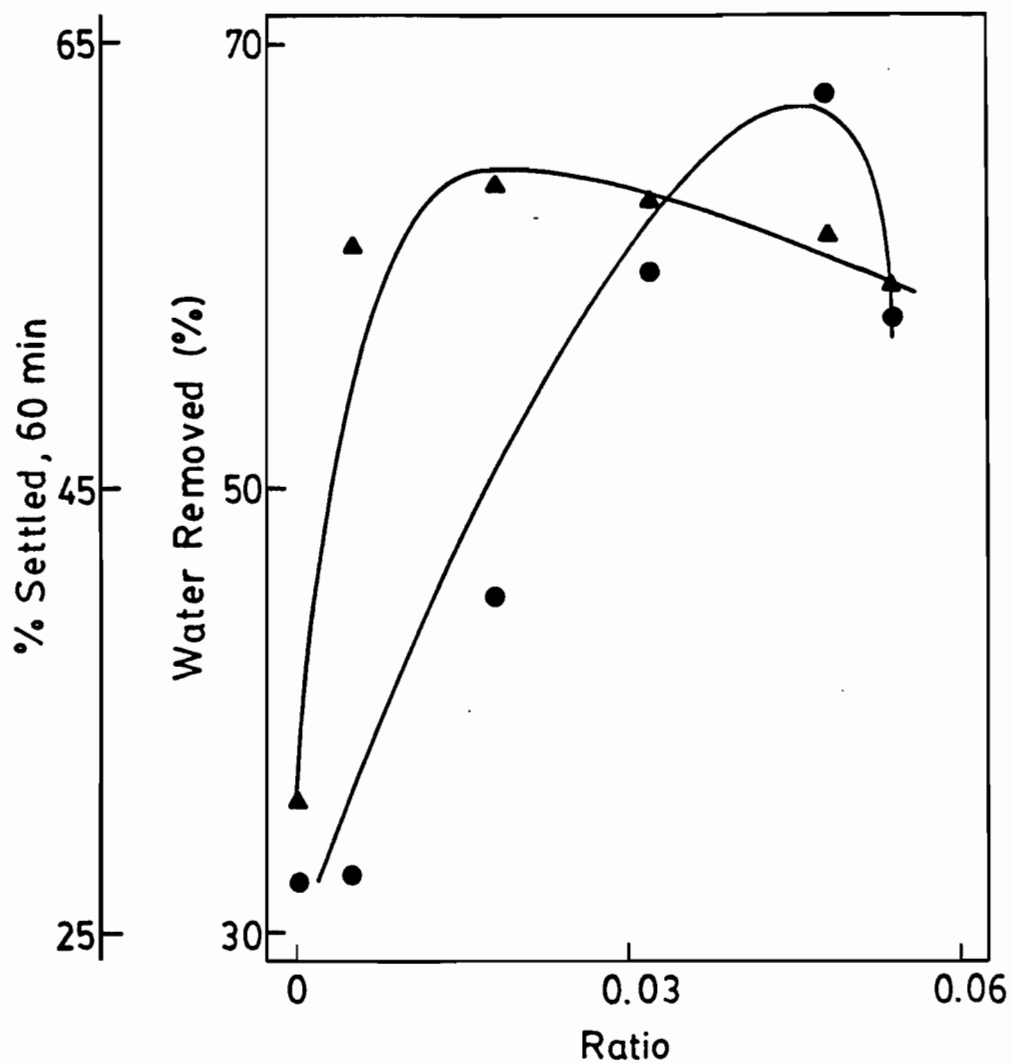


Figure 4.6. Enhancement of water removal from peat treated with aluminum chloride. Water removed ($\blacktriangle$) and percent settled after 60 min ($\bullet$) versus the ratio of aluminum chloride to dry peat in the sample (wt/wt).

were added per gram of dry peat, 68.8% of the water was released during pressing (table 4.7). Water removal and particle settling data are both plotted in figure 4.7. The percent of particles settled after one hour for peat treated with DTAB was higher than for the inorganic salts. The particle settling curve and the water removal curve show very good correlation. Each parameter increased and then decreased at almost the same ratios of DTAB.

4.2. Thermal pretreatment

The heat pretreatment experiments were performed in a simple Parr 4501 two-liter pressure vessel. With the batch heating system three parameters may be varied: heat up time, holding temperature and residence time. The heat up time is the time required to bring the peat from room temperature to the desired holding temperature. The holding temperature is the maximum temperature and the residence time is how long the peat is held at this maximum temperature; after which the peat is immediately cooled back to room temperature. Each of these parameters was varied in separate experiments while the other two were kept constant.

With this equipment it was first necessary to establish procedures for heating peat to desired temperatures. After trial and error experimentation the rheostat was calibrated. The data is shown in table 4.8.

During the heating experiments the temperature was recorded at approximately 5 minute intervals. An example of this data is shown in figure 4.8. In this experiment the heat up time was 30 minutes, the residence time was 15 minutes and

Table 4.7. The effect of dodecyltrimethylammonium bromide on mechanical dewatering.

Ratio of DTAB to dry peat (wt/wt)	Amount of water removed (kg water/ kg dry peat)	Water removed (%)	Moisture content of pressed peat (%)
Control	2.41	33.1	82.9
0.004	3.15	43.3	80.5
0.018	3.98	54.7	76.7
0.030	4.26	58.5	75.1
0.042	4.65	63.9	72.5
0.055	5.01	68.8	69.4
0.060	4.31	59.2	74.8
0.068	4.36	59.9	74.5

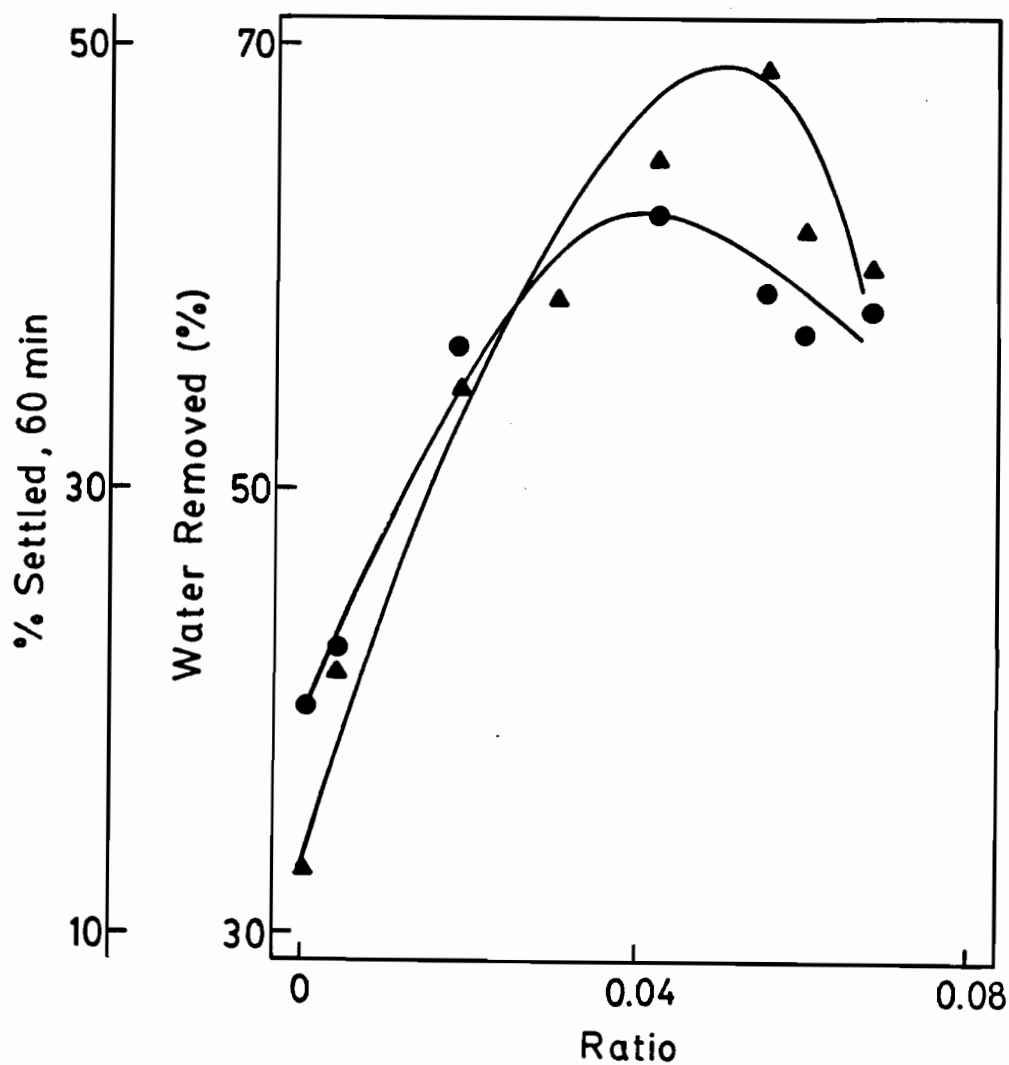


Figure 4.7. Enhancement of water removal from peat treated with dodecyltrimethylammonium bromide. Water removed (▲) and percent settled after 60 min (●) versus the ratio of dodecyltrimethylammonium bromide to dry peat in the sample (wt/wt).

Table 4.8. Calibration of heating apparatus rheostat. Thirty min heat up time.

Holding temperature (C)	Procedure
50	100 for 10 min. Cut to zero and place in jacket.
75	110 for 15 min. Cut to zero and place in jacket.
100	120 for 20 min. Place in jacket. Cut to 15 after 1 min.
125	120 for 30 min. Place in jacket. Cut to 45 after 1 min.
150	120 for 1 hr. Place in jacket. Cut to 30 after 3 min.
175	120 for 1 hr. Place in jacket. Cut to 50 after 8 min.
200	120 for 80 min. Place in jacket. Cut to 70 after 12 min.

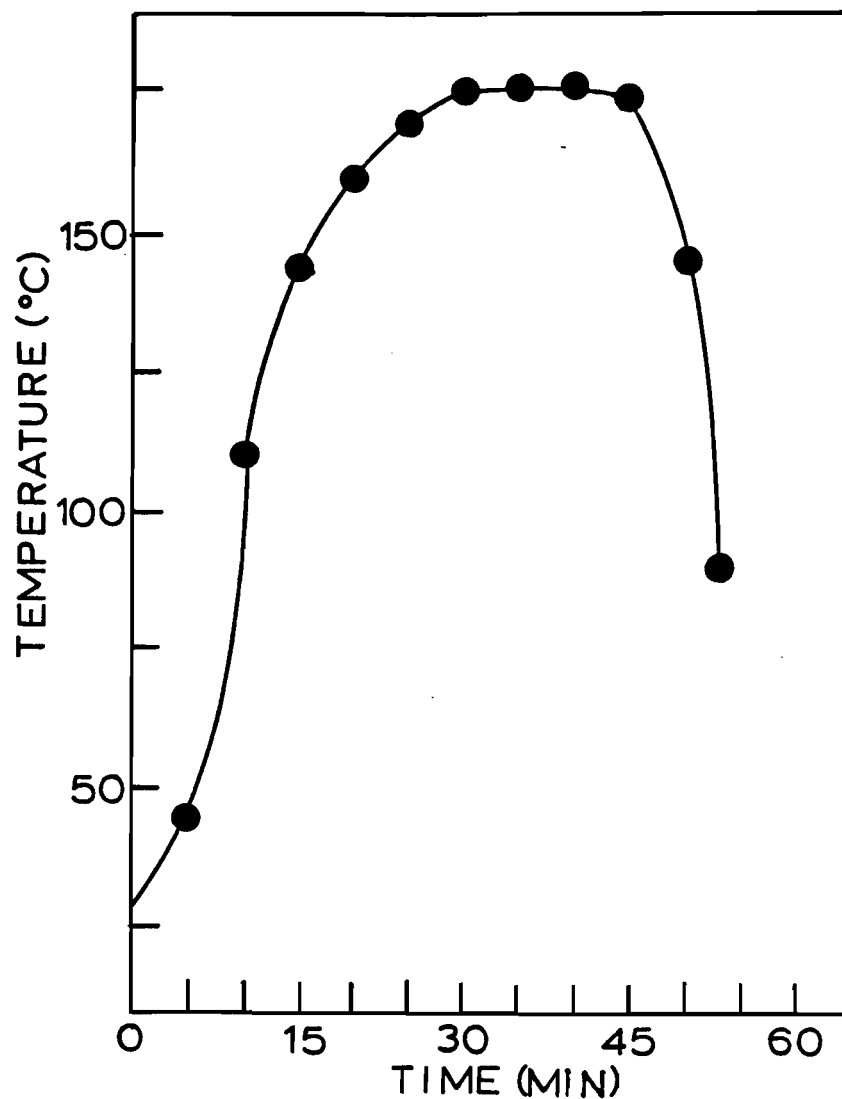


Figure 4.8. Temperature of peat in vessel during thermal pretreatment vs. time. The figure shows the heat up time, first 30 min and the 15 min holding time. The holding temperature is 177°C.

the holding temperature was 177°C. Heating experiments were done over a range of holding temperatures, from 50 to 200°C. Two heat up times, either 15 or 30 minutes, were studied. The residence times were varied from 0 to 90 minutes.

4.2.1. Fifteen minute and zero residence time experiments

Eleven heat pretreatment runs were done with a 15 minute holding time. Each run had a heat up curve similar to figure 4.8. After heat pretreatment, 3 samples at each holding temperature were cooled, pressed and the amount of water removed was calculated. The average amount of water removed by pressing for each temperature tested is shown in figure 4.9. As expected, as the temperature increases the amount of water removed by pressing increases. However, even relatively low temperatures were effective. For example, heating the peat to just 75°C improved the amount of water removed during pressing from 50.2% the control value, to 56.1%, a 6.1% increase. The improvement with increase in temperature continues steadily up to 175°C, where the effect begins to level off, and about 85% of the water is removed.

Also shown in figure 4.9 are the results of six heat pretreatment runs done with zero holding time. Again there is significant improvement in the amount of water removed at only 68°C, where 58.6% is removed.

The data, using either residence time, falls close to the same curve at low holding temperatures. However, at higher holding temperatures the additional heat added during the 15 min residence time caused small increases in the percent water removed compared to the zero residence time.

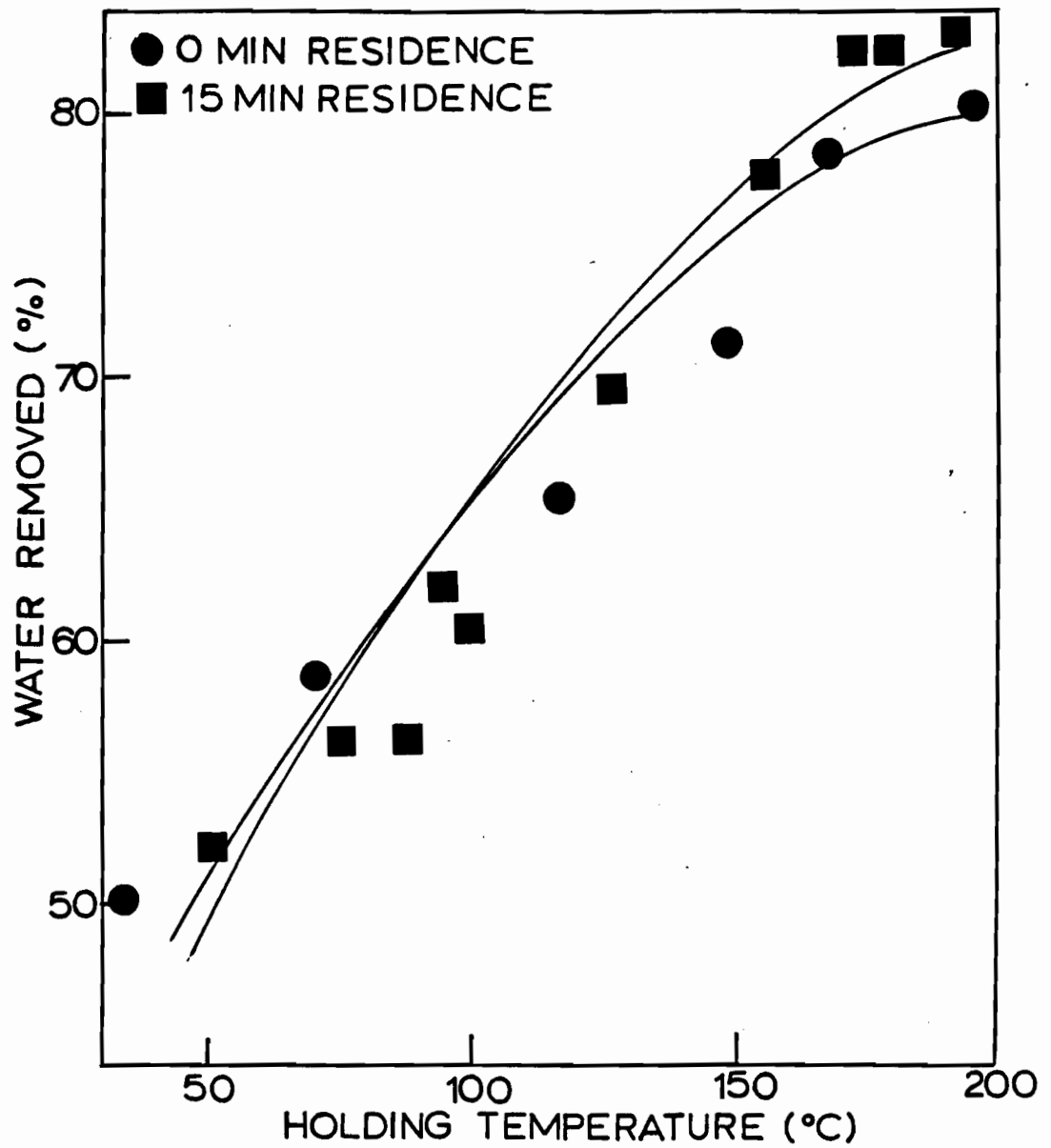


Figure 4.9. The percent water removed after thermal pretreatment vs. the holding temperature. The heat up time was 30 minutes.

4.2.2. Residence time experiments

In the section above, data using two residence times, 15 minutes and zero minutes, is presented. Although almost 20 data points were obtained the holding temperature is the principal variable. Therefore, further residence time experiments were conducted in which the holding temperature was constant.

An improvement in the percent water removed occurred when residence times were longer than 15 minutes and the holding temperature was 165C or 105C. The data are plotted in figure 4.10. The improvement with longer residence time was more pronounced when the holding temperature was 165C. Holding at 165C for 60 minutes, 83.2% of the water was removed, 16.7% more than the zero residence time run. The majority of experiments which follow was done with zero holding time.

4.2.3. Effect of changes in heat up rate

Changing the length of the heat up period also altered the effectiveness of thermal pretreatment. Figure 4.11 shows a heating curve for a run in which the heat up time is 15 minutes, the residence time is zero, and the final temperature is 150C. Four runs were done with 15 minute heat up time; the results are plotted in figure 4.12.

Heating the peat up to the holding temperature more rapidly decreases the effectiveness of thermal pretreatment. The curve drawn through the squares in figure 4.12 shows the relationship between percent water removed during pressing and the holding temperature when the heat up time is 30 minutes and the residence time is zero. The curve drawn through the

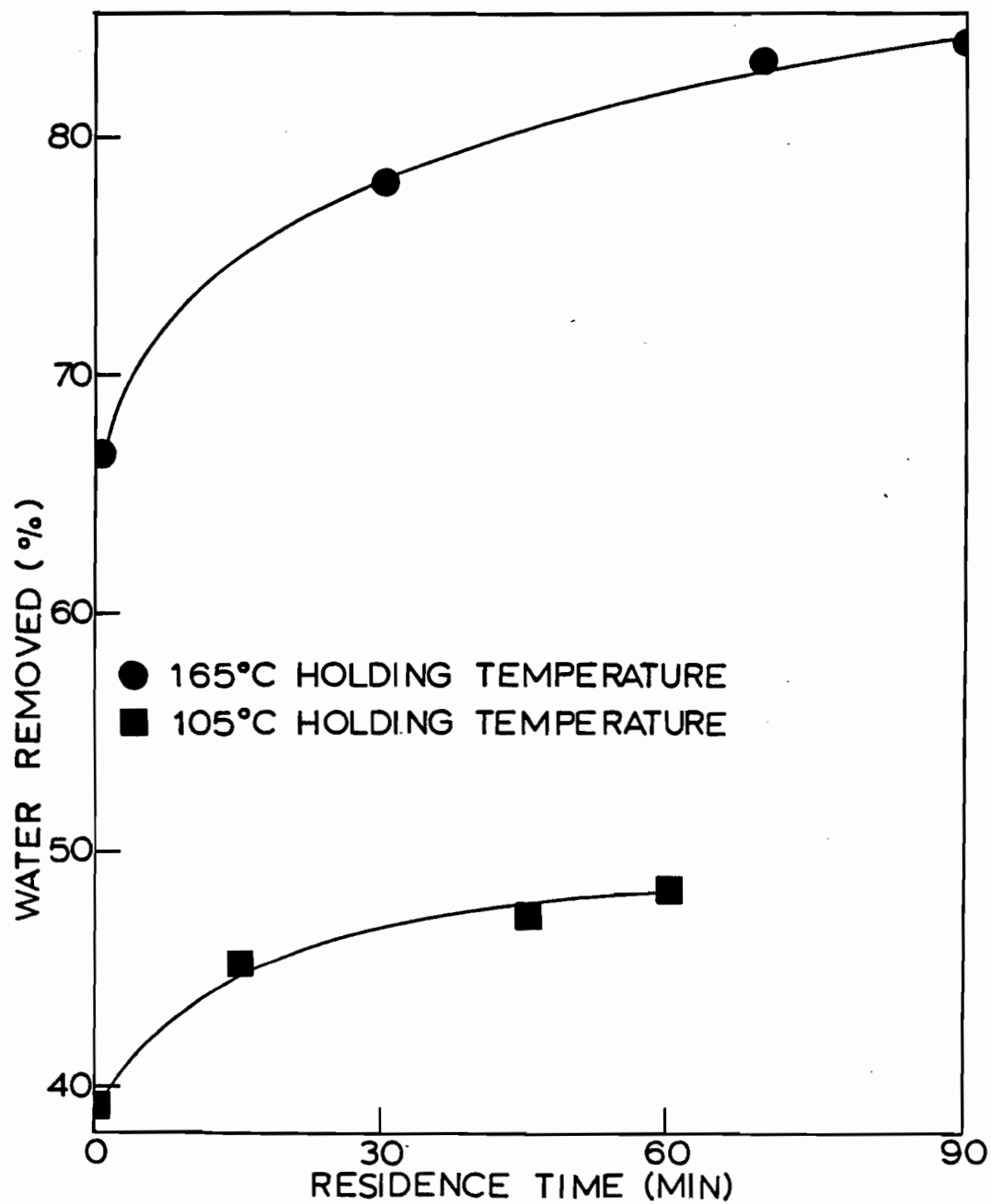


Figure 4.10. The percent water removed after thermal pretreatment vs. the holding time. The heat up time was 30 min.

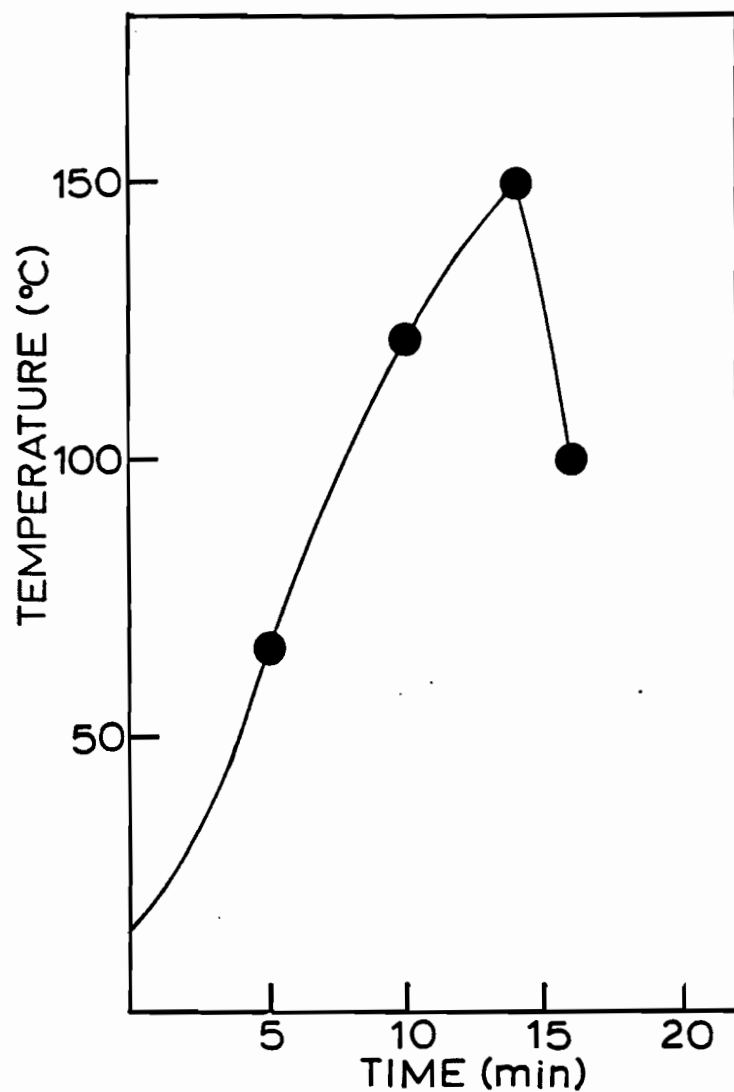


Figure 4.11. Temperature of peat in vessel during thermal pretreatment vs. time. The heat up time was 15 min, the holding time 0, and the holding temperature 150C.

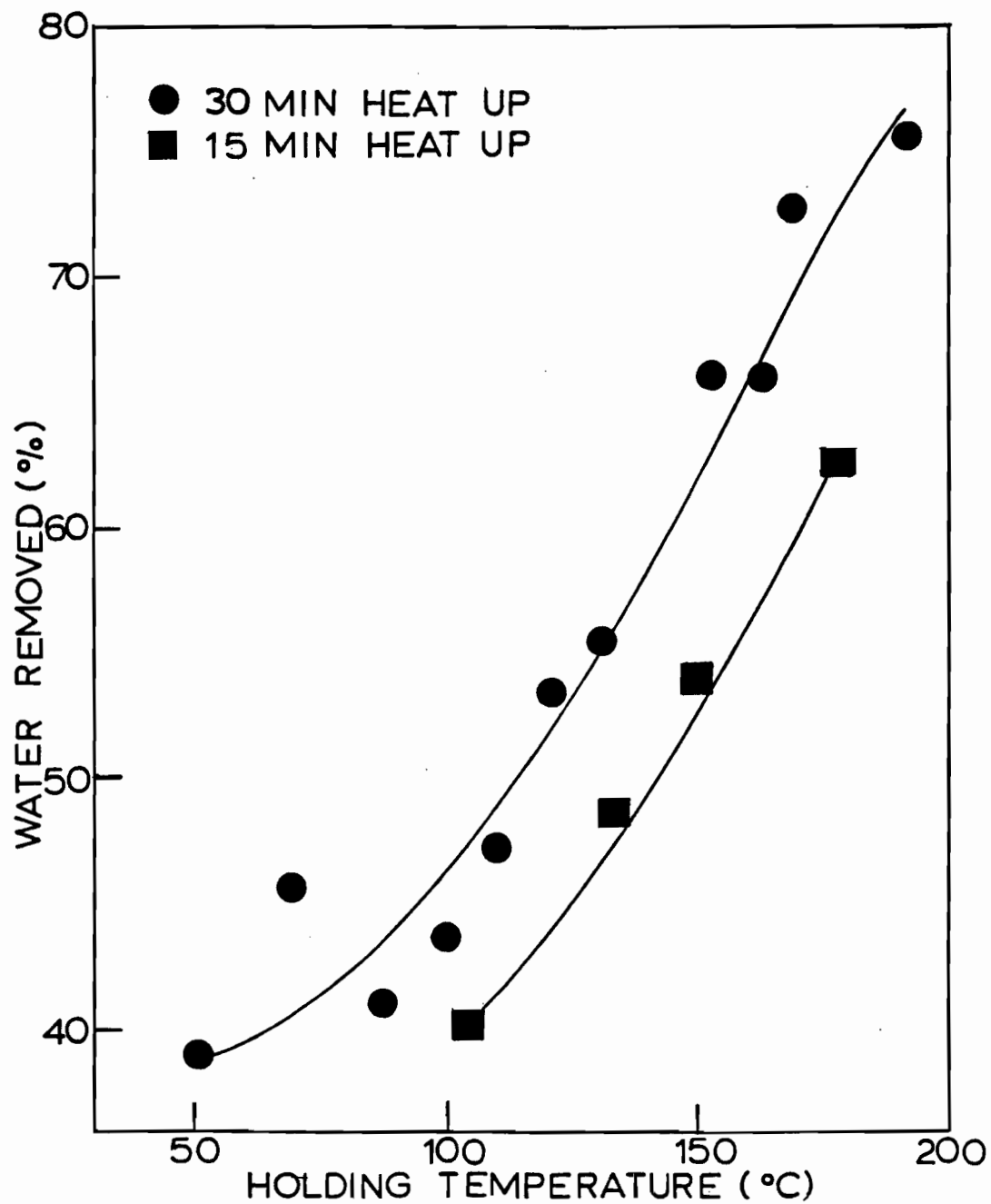


Figure 4.12. The percent water removed after thermal pretreatment vs. the holding temperature. The holding time was 0 min.

circles differs only in that the heat up time is 15 minutes. The fifteen minute heat up time data lies consistently below the 30 minute heat up time data. The difference between the curves at any temperature is approximately 6%.

4.2.4. Combined chemical and thermal pretreatment.

Further experiments were done to study the effect of chitosan salt on heat treated peat. Using a heat up time of 30 minutes and 0 minute residence time, 300 gram batches of raw peat were heated to ten temperatures between 50 and 200C. After heating, three 25 gram samples were pressed and the average amount of water removed at each temperature is plotted as a square in figure 4.13. The remaining heat treated peat was mixed with the optimum ratio of chitosan salt and again, 3 samples were pressed; the average amount of water removed is plotted as a circle in figure 4.13.

The graph shows that heat treated samples to which chitosan salt was added consistently released more water when pressed than the samples heated by the same procedure and pressed without chemical pretreatment. However, as the temperature increases the distance between the two curves lessens. At 100C there is difference of 10% and at 175C the difference is 4%.

4.2.5. Heat treatment with an inert atmosphere.

In order to further characterize the low temperature heating process the standard heating procedure was run with an inert atmosphere. In the standard heating procedure the two liter pressure vessel contains air which is sealed in with the peat during heating. In the inert atmosphere experiments,

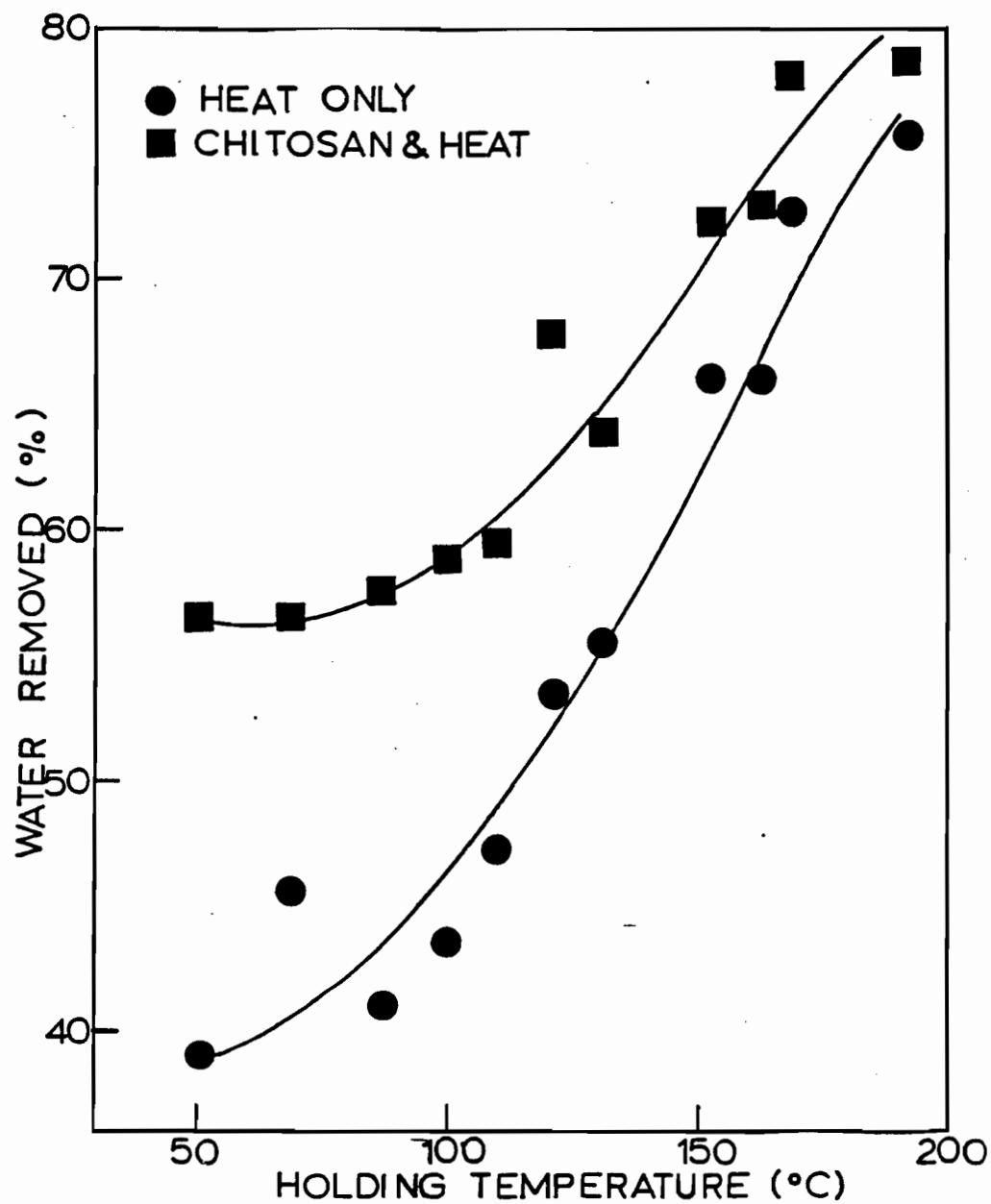


Figure 4.13. The percent water removed after thermal or thermal/chemical pretreatment vs. holding temperature. The heat up time was 30 min; the holding time was 0 min.

after the peat was placed in the reactor nitrogen gas was used to displace the air. Then the peat was heated in the usual manner.

In figure 4.14 the two curves from figure 4.13 have been reproduced and the data from the inert atmosphere heating experiments is plotted. The nitrogen gas procedure was done for a 100 and 150C run; 30 minute heat up time, zero minute residence time. The figure allows for comparison of inert atmosphere data and the data from heating under air. Both sets of points (samples treated with and without chitosan salt) fall very close to the curves. The presence of oxygen during the heat pretreatment has no detectible effect on the amount of water removed during pressing.

4.2.6. Stirring during heating.

Heat pretreatment experiments were run at two temperatures to determine the effect of stirring during heating on the dewatering of peat. In the standard heating experiments the peat is static during heating. Stirring might improve heat transfer or alter the colloidal properties of peat and, therefore, increase or decrease the percent water removed.

The Parr 4501 pressure vessel is equipped with a belt driven propeller type stirrer. The propeller was rotated approximately 100 times a minute during both heat pretreatment runs. The results obtained after pressing are plotted in figure 4.15. Again the curves from figure 4.13 are shown with the stirring data for comparison. The points fall very close to the curves. Stirring during heating does not alter the dewatering of heat treated peat in this temperature range.

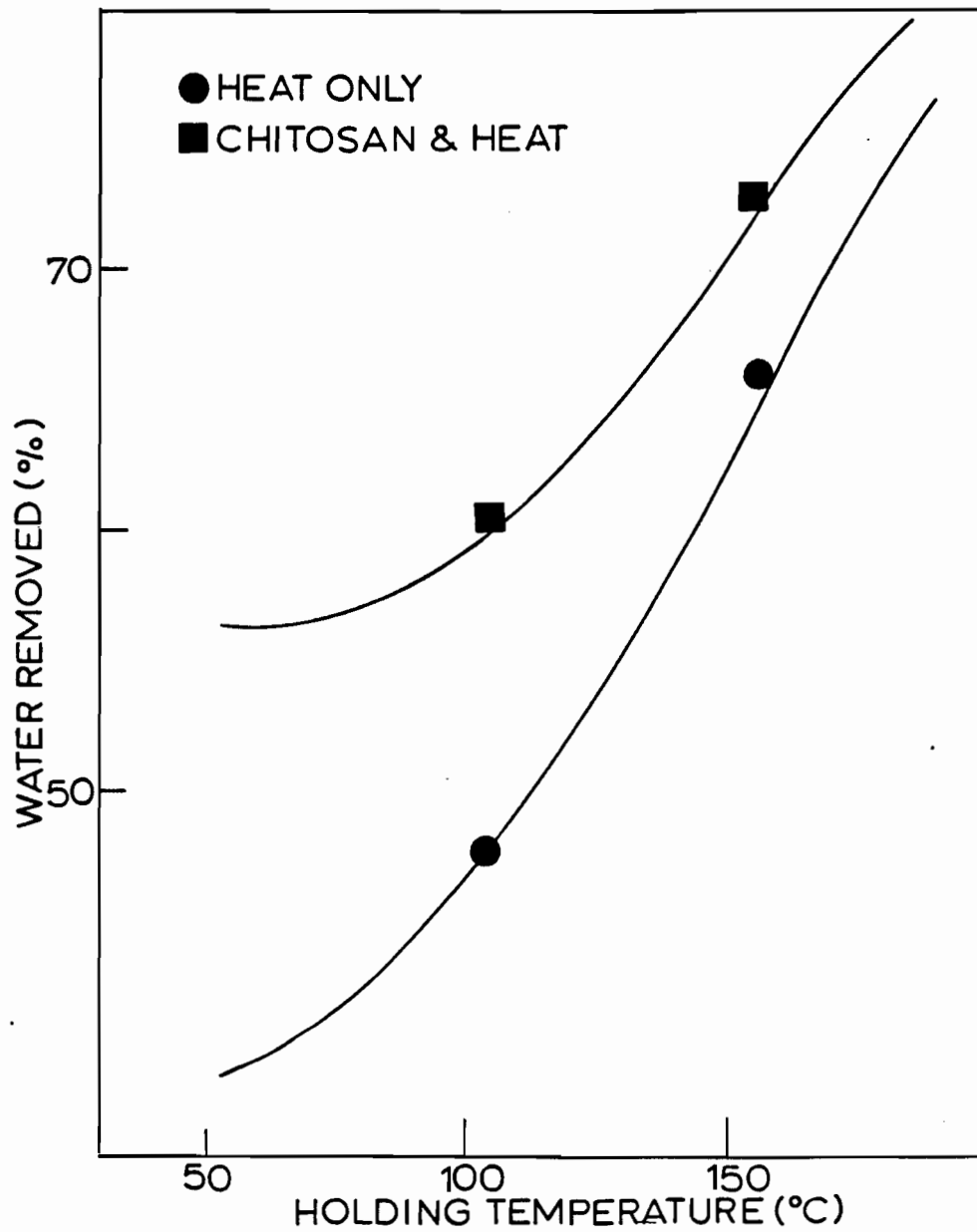


Figure 4.14. The percent water removed after thermal pretreatment under an inert atmosphere vs. holding temperature. The heat up time was 30 min; the holding time was 0 min.

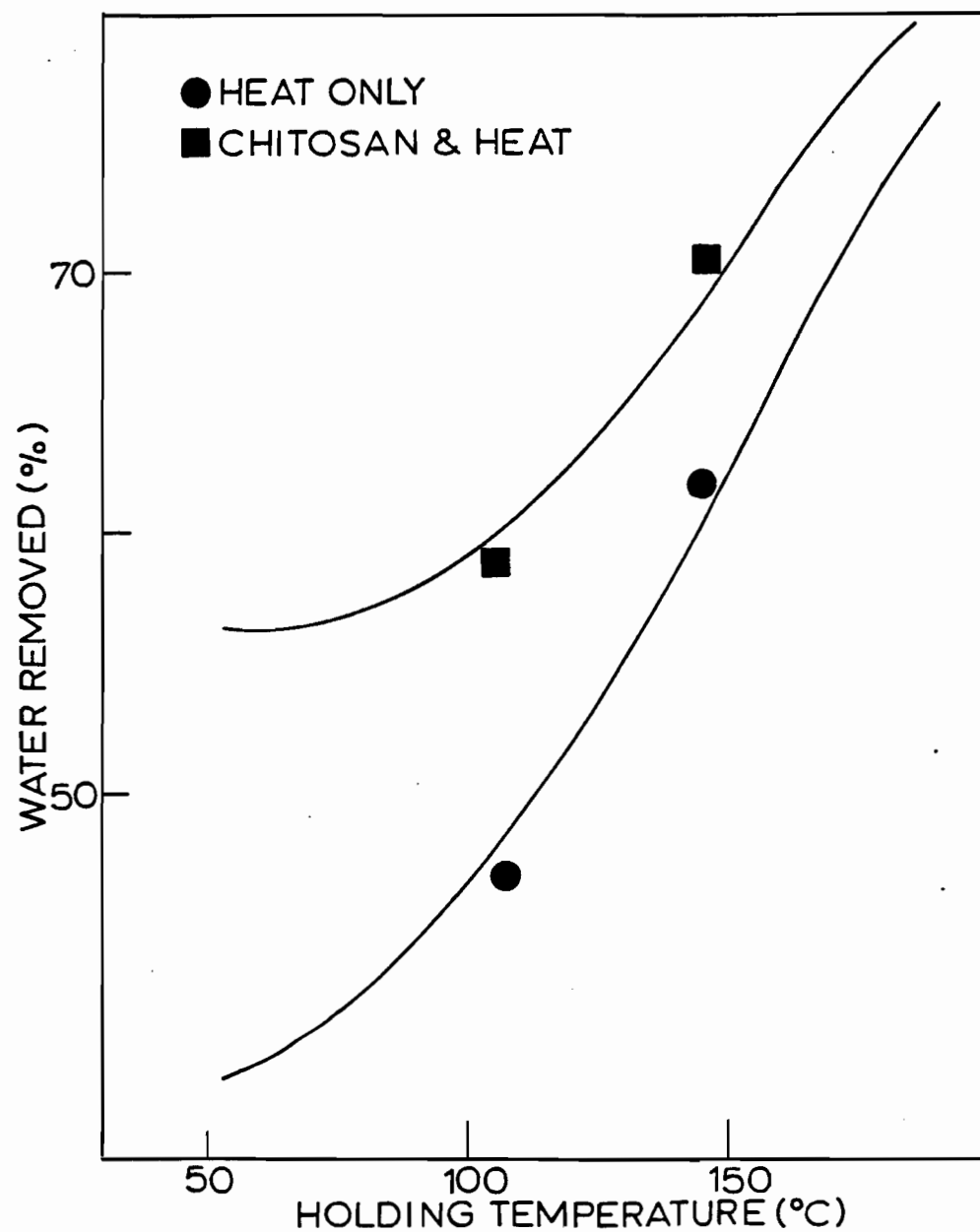


Figure 4.15. The percent water removed after thermal pretreatment with stirring vs. holding temperature. The heat up time was 30 min; the holding time was 0 min.

4.2.7. Heat content of pretreated peat

Samples which had been pretreated, pressed and oven dried, along with one control sample (no pretreatment) were sent to the National Research Laboratory in Halifax. There, the calorific value of each sample was measured to determine the effect of our pretreatment process on this parameter. The results are shown in table 4.9. The numbers show small, random variations and no trend could be detected. The variations are probably due to the heterogeneity of the raw peat used in the pretreatment experiments. Neither heating nor the addition of chitosan salt changes the calorific value of the dry peat.

4.2.8. Particle settling

Particle settling measurements were performed on peat heated to four different holding temperatures. The pressed chitosan and/or heat treated samples were resuspended and used in settling tests. The heat up time was thirty minutes and the residence time was zero. The data is graphed in figure 4.16.

The amount of suspended solids which settled after 1h, from peat which was only heat treated increased smoothly with holding temperature. The heat and chitosan treated samples consistently settled faster. The percent solids, which settled, from the heat and chitosan treated peat remained relatively constant at the four holding temperatures investigated.

4.3. The quality of expressed peat water.

4.3.1. Toxicity tests.

The growth inhibition study (table 4.10) indicated that

Table 4.9. Gross calorific value on a dried basis (kJ/kg)*

Heat pretreatment with zero holding time

Holding temperature (C)	No chemical pretreatment	Chitosan salt added
Control	22.98	22.76
87	22.14	22.66
100	21.60	23.11
131	22.55	22.83
139	22.99	23.37
169	23.12	22.71
192	22.69	22.87

Heat pretreatment with 15 minute holding time

Holding temperature (C)	No chemical pretreatment
42	22.43
75	22.82
100	23.02
125	22.69
146	22.94
165	22.89
197	22.91

* The values presented here were determined by the NRC Atlantic Research Laboratory and were reported to be accurate within +/- 0.001 kJ/kg. They have been rounded off here.

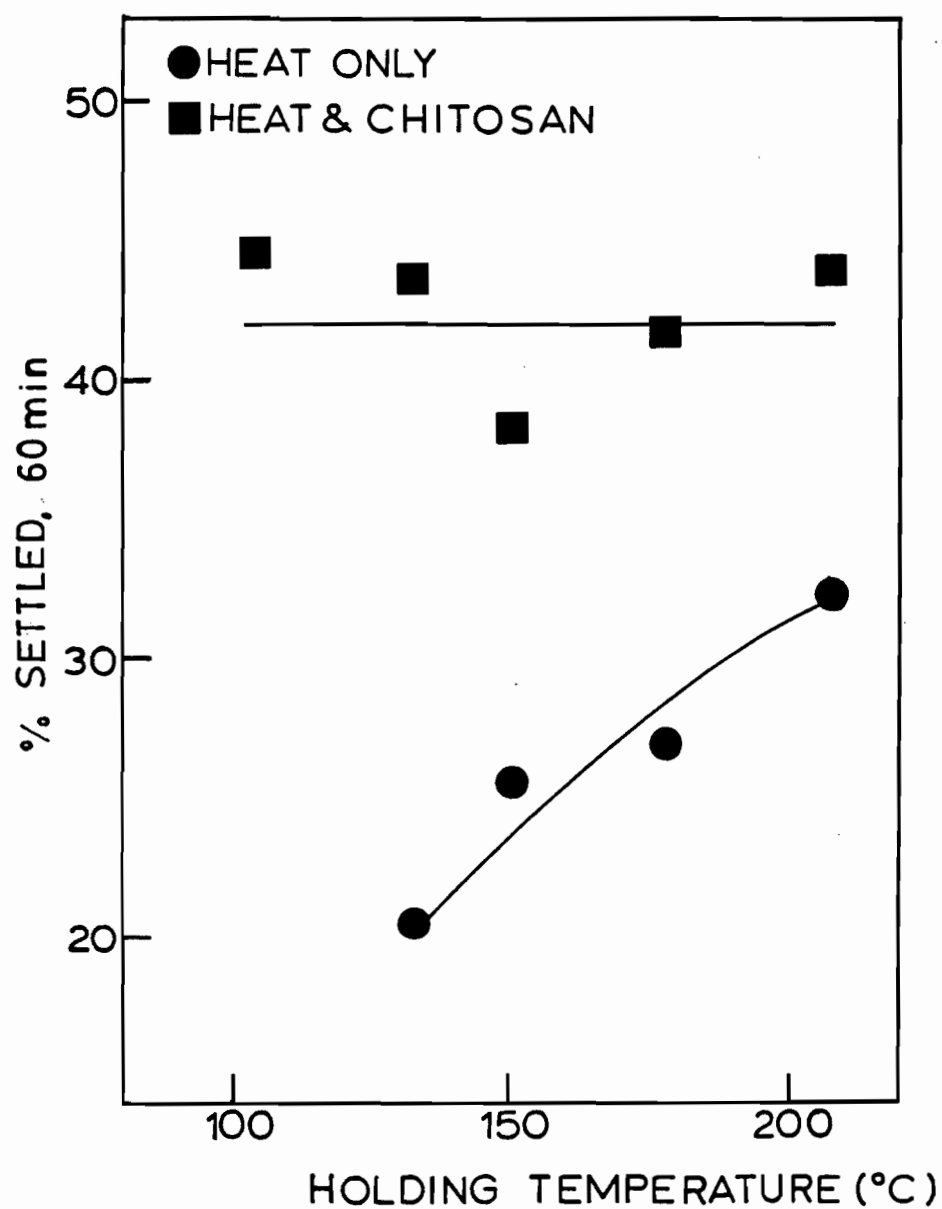


Figure 4.16. The percent settled after 60 min vs. holding temperature. The heat up time was 30 min; the holding time was 0 min.

peat water did not have a dramatic effect on the growth of the organisms. The greatest inhibitory effects were found with the Gram positive bacteria Bacillus subtilis and Micrococcus luteus and to a lesser extent Staphylococcus epidermidis. The gram negative bacteria Psuedomonas acidovorans, Escherichia coli and Proteus rettgeri tended to be initially inhibited but quickly adapted. The yeast Saccharomyces cerevesiae showed no signs of growth inhibition by peat water. Most microorganisms showed only an initial area of clearing which was subsequently totally overgrown. The expressed water used in these tests was derived exclusively from peat which had not undergone any pretreatment process.

A second test, the Microtox test, was used to determine the effect of both chemical and thermal pretreatment on the toxicity of the expressed peat water. The Microtox tests were performed at the Ontario Research Foundation. The test employs bacteria which emit light. When they are exposed to toxic solutions the amount of light emitted decreases. Basically, the percent of toxic solution which reduces the amount of light emitted by 50% is called the EC50 value for that sample (actually the EC50 value is estimated by graphically interpolating between values obtained from a dilution series). The higher the EC50 value the less toxic the sample.

Eight samples were sent to the Ontario Research Foundation for this analysis. The results are shown in table 4.11. Measurements were taken after both five and fifteen minutes of exposure. In all cases the toxicity was very low and EC50 values could only be obtained for the expressed water from

Table 4.10. Inhibitory effects of peat run-off water and pressed peat water on organism growth.

Organism	Peat run-off water		Pressed peat water	
	Area of clearing (square mm)		Area of clearing (square mm)	
	Initial	Final	Initial	Final
<u>Bacillus subtilis</u>	21.65	5.70	6.88	2.23
<u>Pseudomonas acidovorans</u>	11.78	0	14.32	0
<u>Escherichia coli</u>	8.69	0	3.37	0
<u>Saccharomyces cerevisiae</u>	0	0	0	0
<u>Staphylococcus epidermidis</u>	5.70	0	5.70	0
<u>Micrococcus luteus</u>	5.00	3.37	15.62	11.78
<u>Proteus rettgeri</u>	0	0	16.28	0

Table 4.11. Results of Microtox test.

Expressed water from heat treated samples

Holding temperature (C)	5 min EC50 (% sample)	15 min EC50 (% sample)
100	VLT	VLT
131	VLT	VLT
163	VLT	VLT
192	28	28

Expressed water from heat and chitosan treated samples

Holding temperature (C)	5 min EC50 (% sample)	15 min EC50 (% sample)
100	VLT	VLT
131	VLT	VLT
163	VLT	VLT
192	28	28

VLT = very low toxicity

samples which had been heated to 192C in pretreatment. It would have required extrapolation beyond the data points to estimate EC50 values for the lower temperature samples.

4.3.2. Initial concentration of suspended solids.

When peat is pressed mechanically the water which is released contains suspended particles. Generally, the severity and type of pretreatment process used before pressing effects the concentration of suspended peat solids in the expressed water. Therefore, this parameter was measured for peat pretreated at eleven different temperatures, thirty minute heat up time and zero minute residence time, with and without chitosan salt. The results are shown in table 4.12.

In table 4.12 the lowest initial concentration of suspended peat solids is 430 mg/liter, for peat which was not heated but was pretreated with chitosan salt. The highest initial concentration is 3220 mg/liter from peat which was heated to 192C. For each pretreatment temperature studied the addition of chitosan salt does not have a significant effect on the concentration of suspended solids. However, the data clearly shows that an increase in pretreatment temperature results in an increase in suspended solids concentration.

4.3.3. Settling tests.

One hour after the initial concentration readings were taken a second reading was made on the expressed water samples. These two measurements allowed calculation of the percent of solids settled after one hour. The results are shown in table 4.13. There is no correlation between the percent settled and the pretreatment temperature. At each

Table 4.12. Initial concentration of suspended solids in expressed water.

Holding Temperature* (C)	Initial Solids Concentration (mg/L)	
	Heat Only	Heat and Chitosan
Control	550	430
51	590	550
87	460	470
100	530	610
110	670	450
121	840	600
131	1410	1320
139	2750	2930
169	2880	2670
192	3220	3000

* Heat up time was thirty minutes. Residence time was zero.

Table 4.13. Settling tests on expressed water.

Holding temperature* (C)	% Settled, 60 min	
	Heat Only	Heat and Chitosan
Control	5.6	14.2
51	16.9	24.5
87	22.8	30.6
100	22.5	44.1
110	10.2	23.1
121	15.1	27.0
131	15.1	29.9
139	18.0	28.1
169	15.8	29.4
192	18.0	22.9

* Heat up time was 30 minutes. Residence time was zero.

temperature a greater percent of suspended solids settled out after one hour from water samples pressed out of peat which was treated with chitosan salt. The average difference after one hour is 14.4%, a significant improvement for chitosan treated peat.

A third reading was taken after 5 days had passed. For heat treated samples the concentration of suspended solids was consistently 100 ± 10 mg/liter. For heat and chitosan treated samples the concentration was always less than 10 mg/liter. Visually, the expressed water from samples which had not been treated with chitosan was still murky after five days and the water from samples which had been treated with chitosan salt was clear.

The rate at which the suspended peat particles settle is determined by their size. Larger particles settle faster. Thus, peat treated with chitosan salt yields expressed water with suspended solid particles which are on the average larger in size than peat which is not treated with chitosan salt.

5. Discussion

Both chemical and thermal pretreatment alter the water retention properties of peat. The study of seven compounds which improved peat dewatering and of a wide range of heating conditions provided some insight into the solid-water interactions which limit the effectiveness of mechanical dewatering.

Several authors have attempted to categorize the ways in which the aqueous phase is bound to the peat. Chornet (1981) stated that there are four types of peat water. Ordered from easiest to remove to hardest, these are: (1) water in macropores, (2) water in micropores, (3) colloiddally bound water and (4) chemisorbed water. For a raw peat (92% moisture content), on average, 15% of the water is found in pores, 30% is colloiddally bound and the remainder is chemisorbed. According to Sheppard and Ayub (1985) water in peat can be broadly classified as (1) mechanically entrapped water; (2) capillary water and (3) chemically bound water. Sheppard and Ayub also quote Ljungkvist and Munteer who claim 25% is "loosely bound" water, 40% capillary water, 25% colloidal water and 10% chemically bound water.

It is profitable to compare peat dewatering to sludge dewatering. Sludge has been studied more thoroughly and the solid-water interactions are better understood. The phenomena which explain the dewatering characteristics of sludge are similar to those usually used to explain peat dewatering.

Biological sludge is composed mostly of organisms that contain water in several different forms. The particles generally exhibit a net negative charge in the range of -10 to -20 millivolts, which does not appear to depend on the type of

sludge (Campbell, 1978). In general, conventional mechanical dewatering will concentrate the solids content to only about 10% solids. Studies on sludge dewatering have led to the following conclusions :

1. With chemical conditioning the solids concentration can be increased to about 15%.
2. The performance achieved by a dewatering unit on any sludge is greatly affected by particle size distribution and the strength of the sludge floc.
3. One method of promoting sludge flocculation is to reduce the electrical charge on a particle by chemical addition to move the zeta potential towards the isoelectric point.

There are obvious analogies between peat and sludge dewatering and it is reasonable to apply the above conclusions to both operations.

5.1. Chemical pretreatment

In table 5.1 the data obtained with the 7 dewatering compounds is summarized. The agents are listed in order of effectiveness. Dodecyltrimethylammonium bromide improved the percent water removed over the control by the greatest amount.

Two of the polyelectrolytes were only slightly less effective than the surfactant and produced very similar results. Chitosan increased the dewatering by 32.2% and Praestol 444K by 30.3%. Optimum dewatering ratios and optimum settling ratios corresponded quite well for both compounds. This again indicates that when the particle size of the resuspended pressed peat is at a maximum water removal is greatest.

Polyelectrolytes are commonly used in separating suspended solids from sewage water. The term "flocculation" is used to describe the mechanism by which polyelectrolytes function.

Table 5.1. Summary of water removal data from chemically treated peat.

Agent	Optimum ratio (wt/wt)	Increase in water removed (%)
DTAB	0.055	35.7
Chitosan salt	0.019	32.2
Ferric Chloride	0.062*	31.8
Praestol 444K	0.015	30.3
Aluminum Chloride	0.018*	27.6
Ammonium Chloride	0.030*	14.6
Praestol 411K	0.040	14.0

* This is actually the point at which the water removal curve levels off. There is no true optimum.

Flocculation involves two processes: surface charge neutralization and bridging. The charge density on the polymer is an important factor in determining its capacity to cause flocculation (Svarovsky, 1981). Bridging occurs when several particles become associated with adjacent charged sites on the same molecule.

The particle settling data for pressed peat which had been treated with chitosan and Praestol 444K indicate that bridging is an important mechanism in enhanced peat dewatering. The maximum, which occurs in the settling curves for both these compounds, is commonly observed in suspensions destabilized with polyelectrolytes. Too little of the polymer results in some charge neutralization and less than optimum bridging. When more than the optimum quantity of polyelectrolyte is added one particle will consume more than one charged site. The polymer wraps itself (in varying degrees) around the particle, inhibiting bridging.

Peat treated with Praestol 411K, the low charge density polymer displayed poorer dewatering than that treated with the other polyelectrolytes. The lower charge density sites are probably less efficient at adhering to peat particles and causing bridging.

The data obtained for ammonium chloride additions is further evidence that bridging is occurring with the chitosan salt. Ammonium chloride improved water removal by only 14.0%. Although this is a significant improvement it is much less than for chitosan salt. Ammonium chloride is capable of executing charge neutralization only. Its charged group is similar to the primary

amine which when protonated provides the positive charge on chitosan. (The hydrocarbon group does not have significant electron withdrawing capabilities.) Therefore, the superior dewatering caused by chitosan salt additions must be due to its bridging capabilities.

The two other salts tested, aluminum chloride and ferric chloride, are, like ammonium chloride, also capable of neutralizing the charged peat particles. They are, of course, incapable of causing conventional bridging. However, both proved to be very effective dewatering agents. Ferric chloride and aluminum chloride improved water removal by 31.8% and 27.6%, respectively. The dewatering and settling curves for both of these compounds show essentially a leveling off at high ratios instead of maxima. This is typical of data obtained when inorganic salts are used to enhance particle settling.

To quantify exactly and explain the difference in ability of these two salts to enhance peat dewatering, a knowledge of all of the hydrated cations present in the aqueous phase would be essential (Faust, 1983). Aluminum and iron salts frequently form aluminum hydroxide and ferrous hydroxide when added to suspensions. The precipitation of either of these two compounds, in addition to charge neutralization by the hydrated cations, may enhance coagulation. As the precipitate forms it tends to entrap the particles and floc is formed. It should be noted that using large quantities of inorganic salts to dewater fuel-grade peat may prohibitively increase ash content, an undesirable combustion product. Organic polymers typically have acceptable combustion properties and would not create this problem (Campbell, 1978).

Peat treated with DTAB released 35.7% more water than untreated peat. DTAB is a cationic surfactant and its use as a dewatering agent has been studied thoroughly by Ayub and Sheppard (1985). They compared water removal data with zeta potential measurements and found that optimum dewatering occurred when the electrophoretic mobility was near zero. When less than the optimum amount of DTAB was added the particles were not completely neutralized. When more than the optimum was added the particles obtained a positive charge. This is believed to be the result of "extra-equivalent adsorption". Excess DTAB is adsorbed onto the particles due to the attraction between the hydrophobic tails of the surfactant. The picture explains the shape of the DTAB dewatering curve.

The optimum dewatering ratio observed for DTAB corresponds almost exactly with the optimum settling ratio. Therefore, at this ratio, where electrostatic repulsions between particles are minimized, the greatest amount of agglomeration has occurred and it is this mechanism which, at least partly, enhances the solid-liquid separation. However, this does not explain why DTAB is more effective than either cationic polymers or inorganic salts at enhancing dewatering, both of which are also capable of executing charge neutralization.

The superior dewatering of peat treated with DTAB can be explained by the compound's surfactant properties. When the positively charged head is adsorbed onto a peat particle the hydrocarbon tail is oriented outward. The decrease in electrostatic repulsions between particles caused by the cationic head facilitates coagulation. When the particles become aggregated a

lower energy state is achieved; the hydrocarbon tails are brought together. This increases the driving force for particle-water separation. The surfactant coated particles are more hydrophobic and hence more easily separated from the aqueous phase.

5.2. Chemical and thermal pretreatment

Results from the heat pretreatment experiments presented here aid in characterizing the reactions which are believed to be the cause of the enhanced dewatering. The changes which improve dewatering are further elucidated by considering the affects of combined chemical and thermal pretreatment.

The changes which occur in peat during heating are apparently initiated at low temperatures. Small amounts of heat added at low holding temperatures (eg. 75C) enhanced dewatering.

At low temperatures the 0 minute and 15 minute residence time experiments yielded similar results. This indicates that the additional heat added by holding at low temperatures for 15 minutes is insignificant. However, with holding temperatures above 100C a change in the amount of heat added will change the amount of water removed. Less water was removed when the heat up time was shortened (less heat added) and more water was removed when the residence time was lengthened (more heat added).

Heating under either an inert atmosphere or air produced the same results. This implies that oxidation of the peat materials is not a part of the water releasing changes which occur. That there was no change in the heat content of heat treated peat confirms that the oxygen content does not change substantially during the pretreatment process (Sheppard et al., 1985).

Both direct and indirect methods have proven that peat particles possess a surface charge and that this plays a significant role in determining the water retention properties. Heating alters the surface charge of peat particles by inducing reactions which liberate or rearrange charged groups. Both Fuschman (1980) and Schnitzer and Kahn (1972) have written that during heating of peat at low temperatures decarboxylation of carboxylic groups occurs and low-molecular-weight alcohols are generated by simple rearrangement of esters.

These reactions do not only destabilize the colloidal nature of peat; they also effect the hydrogen bonded structure which is believed to be partly responsible for peat's hydrophilicity (Chornet et al., 1981). By liberating or rearranging the surface groups the interactions which exist with the oxygen and hydrogen atoms in the water layers surrounding the particles are weakened.

A combined chemical and thermal pretreatment process works on destabilizing the peat's colloidal properties and weakening hydrogen bonding. The effect of heating peat to 160°C, for example, can also be achieved by heating to 110°C and adding a small amount of chitosan salt. Chitosan removes a larger percent of water from peat which has been heated to lower temperatures. Apparently heating to higher temperatures significantly alters the particle's surface chemistry and the charge neutralization which chitosan usually causes has already partially occurred. However, additional heat will further disrupt the hydrogen bonding.

This conclusion is further supported by the particle settling data from peat treated with heat or heat and chitosan salt (section 4.2.8). The data shows that heating alone improved the particle settling rate by a small but significant amount. Increases in holding temperature, in fact, yielded larger resuspended particles. This indicates that heating causes coagulation. The percent of particles which settled after 1h from the pressed peat treated with heat and chitosan, regardless of holding temperature, was approximately constant. This indicates that there is a limit to the amount of coagulation which can occur for heat and chitosan treated peat. Therefore, a disruption of the hydrogen bonding must be partly responsible for the improved dewatering of peat heated to the higher holding temperatures.

5.3. Expressed water quality

The Kirby-Bauer type growth inhibition studies demonstrated that a variety of microorganisms may be grown on expressed peat water. The microbes would produce compounds to be either sold or used as dewatering agents (after chemical modification if necessary). Fermentations are also capable of removing toxic chemicals from process waters which are unfit to be returned directly to the environment.

The Microtox test, however, showed that unless holding temperatures above 190C are used the expressed water has very low toxicity levels. Chitosan addition had no detectible effect on the results of the Microtox tests. Water expressed from pre-treated peat has not been used as a substrate for microorganisms

in this study. It has been shown that expressed water from heat treated peat contains significant amounts of organic molecules (Smith et al., 1984). Cooper et al. (1985) have suggested that these substances could be utilized by microbes and actually reduce the cost of nutrients which would have to be added to expressed water from unheat treated peat.

The peat particles present in the expressed water may be recovered and recycled, used in the substrate in a downstream fermentation or released into the environment. Therefore, their concentration is a parameter which must be considered in a dewatering process. Zimpro's and J.P. Energy's dewatering processes, both of which employ wet carbonization, have estimated the average concentration of suspended solids (TSS) in their effluents to be 1400 mg/L and 500 mg/L, respectively (Monenco, 1983).

The initial concentration of peat particles in the expressed water increased at higher holding temperatures. The addition of chitosan salt to heat treated peat did not consistently increase or decrease this parameter. However, particles in the expressed water from chitosan treated peat were consistently larger, they settled quicker. This indicates that (1) as already explained, chitosan coagulates the peat particles and (2) the initial concentration of suspended peat particles in the expressed water is not dependent on the size of the particles. The heating breaks down the hydrogen bonded structure of peat and this apparently promotes the release of particles into the aqueous phase upon pressing. In a commercial process both the initial suspended solids concentration and the size of the particles would depend on the pressing apparatus.

5.4. Economic assessment

An economic assessment of the process is presented in table 5.2. In processes 1 through 4 the pretreatment is thermal only, in processes 5 through 7 pretreatment entails heating and chitosan salt addition. It is based on a plant capacity of 270,000 dry tonnes/year of dry peat. Cost data published by Cooper et al. (1984) were used exclusively. The moisture content of the product would be 10% from an 88% water content feed.

After pretreatment water is removed by pressing with a Larox filter press which works automatically in a semi-continuous batch mode. The water removed in processes 5 through 7 is used to grow the organism *R. arrhizus*. *R. arrhizus* produces chitosan in excess at a price comparable to that of other flocculating agents. Flash drying, which has been used reliably for feed peat, (Monenco, 1983) follows the pressing stage to yield the product.

The thermal pretreatment step is fueled by the product. Higher temperature processes consume greater amounts of raw peat per unit of product, as can be seen in the fourth column in table 5.2. Process 3 would consume almost 10% more raw peat than process 7, therefore, depleting resources quicker. This may make process 7 more attractive when considering long run parameters.

Production of chitosan lowers the chemical oxygen demand of the pressate (Mulligan and Cooper, 1985), and eliminates the need for pollution control systems. Some form of treatment would be required for water released from the high temperature processes in which chitosan is not produced. However, this cost has not been assessed here. Furthermore, it should be noted that no

Table 5.2. Economic assessment of processes

#	Holding Temperature (C) b	Water Removed w/ press (%)	Input Raw Feat (tonnes/ tonne product)	Total Cost (\$)a
1c	120	51.6	10.06	151.54
2c	140	59.1	10.79	146.58
3c	165	68.2	11.93	132.59
4c	185	74.7	13.09	127.76
5d	101	59.1	9.39	141.37
6d	142	68.2	10.91	137.22
7d	165	74.7	11.93	134.02

a. Costs in 1983 Canadian dollars/dry tonne of product.

b. Residence time is zero, heat up time is 30 min.

c. Thermal pretreatment only.

d. Thermal pretreatment and chitosan salt addition.

process optimization has been performed for the biological component which would reduce costs and increase yeilds.

Rohrer (1981) has shown that adding a thermal pretreatment stage to a mechanical dewatering process is economically attractive. Capitalizing on the soluble organics that heating releases into the expressed water yeilds a product of comparable cost. The fermentation produces a dewatering agent which reduces the quantity of peat needed to fuel the thermal pretreatment and the water may be returned to the environment without further treatment.

6. Conclusion

Dewatering processes must be efficient if a fuel-grade peat is to be produced economically in northern climates. Mechanical dewatering processes preceded by either thermal or chemical pretreatment have shown the most promising results to date. This research examined these two types of pretreatment to determine how and why they enhance mechanical dewatering. The improved dewatering of pretreated peat can be attributed to changes in the peat particle surface chemistry. Attention was also paid to the expressed water quality and the possibility of applying microbiology to a peat dewatering process.

The surfactant, inorganic salts and cationic polymers all enhanced dewatering. Each of these compounds caused the peat particles to coagulate. Of the inorganic salts, which are capable of effecting a charge neutralization, ferrous chloride improved dewatering the most. Inorganic salts would not be used when producing a fuel product because of their undesirable combustion properties. The polymers Praestol 444K and chitosan salt, which neutralize particle surface charge and form bridges between the particles, were both effective at improving dewatering. A dewatering process enhanced by a chitosan derivative (e.g. chitosan hydrochloride) would incur lower costs and yield better quality expressed water than a process using Praestol 444K or any other synthetic polyelectrolyte because the chitosan can be produced biologically by fermentation with peat pressate water as the substrate. Dodecyltrimethylammonium bromide improved the mechanical dewatering of peat more than any other compound tested. This is due to the

way in which the surfactant coats the particle rendering the surface more hydrophobic. This is in addition to the water removal enhancing charge neutralization cationic polymers and salts affect.

The addition of chitosan can be applied in conjunction with thermal pretreatment. Heating the peat, even to relatively low temperatures, before pressing causes a significant improvement in water loss. Heating allows reactions such as decarboxylation to occur. The loss of polar functional groups from the peat solids decreases the amount of water retained by hydrogen bonding. The addition of chitosan to heat treated peat results in an even larger water loss. The effects of the two pretreatments are additive. It would be possible to choose an optimum combination of thermal pretreatment, chitosan addition and thermal drying after pressing to obtain dried peat at the lowest possible cost.

The chitosan has a secondary effect on the economics of the process. If the agent is added before pressing the particles in the expressed water are larger. This means that the particles settle faster. These systems yield a clear aqueous phase and a sludge. The clarified water reduces cost of pollution abatement and the solids can be recycled to the press to improve final product yield.

The thermal pretreatment work done here is perhaps further from commercial applications than the chemical pretreatment work. The batch apparatus used was very small scale. A continuous operation would be more likely. The effectiveness of low temperatures and short residence times in such an operation has not been studied thoroughly, however, the results presented here prove that research in this area merits attention.

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