THE UNIVERSITY OF CALGARY

# **Water Migration Through**

# **Hydrophobic Soils**

by

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## ABSTRACT

Hydrophobic soils are known to resist moisture penetration resulting in an uneven distribution of water. It is a time dependent property of certain soils with significant effects on plant growth, ground water contamination and surface erosion. A detailed laboratory investigation of moisture migration through hydrophobic soils was conducted. Infiltration tests, wetting and drying tests, mercury porosimeter and x-ray CAT scan were conducted.

The laboratory studies indicated that there is a threshold value of initial moisture content (critical moisture content) beyond which hydrophobic soils become hydrophilic. The critical moisture content varied with soil type. A decrease in soil hydrophobicity and increase in soil wettability was observed when hydrophobic soils were subjected to a number of wetting and drying cycles. Hydrophilic-hydrophobic soil mixtures, hydrophobic soil amended with soil conditioners and kaolinitic clay mixed with hydrophobic soils all displayed improved water holding capacities, improved moisture infiltration, amelioration of water repellency, and better seed germination.

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### Chapter 1

### **1.1 Introduction**

Dry soil usually absorbs moisture due to the strong interaction between soil particles and water. These soils are called hydrophilic or non-repellent soils. Certain dry soils develop water repellent characteristics with time and do not permit moisture infiltration. These soils are called hydrophobic or water repellent soils. Water droplets in contact with these soils tend to form beads. Hydrophobic soils are generally erodible and infertile due to their negligible water holding capacity. The affinity of water is reduced in hydrophobic soil to such an extent that soil resists water infiltration for hours, days or weeks. Some of these soils exhibit extreme water repellent characteristic when dry. The cause of hydrophobicity or water repellency, in soils is not yet understood completely. Its causative agents may be natural or anthropogenic in origin.

Hydrophobic soil has been found in many parts of the world and reported in United States (De Bano, 1981; Reeder and Jungerius, 1979), Australia (Roberts and Carbon, 1971; McGhie and Posner, 1980; Ma'Shum and Farmer, 1985; Ma'Shum et al., 1988), Egypt (Bishay and Bakhati, 1976), Japan (Nakaya, 1982), Poland (Prusinkiewics and Kosakowski, 1986), Great Britain (Shiels, 1982), Canada (Barrett and Slaymaker, 1989; Roy et al., 1999), the Netherlands (Bisdom et al., 1993; Dekker and Ritsema, 1994), Italy (Giovannini and Lucchesi, 1983), Portugal (Doerr et al., 1998) and New Zealand (Wallis et al., 1989).

Though a number of causes for soil hydrophobicity have been hypothesized, it is believed that the presence of hydrophobic organic compounds on soil particles leads to hydrophobicity. The sources of organic matter may include plant derived organic matter

such as decomposing plant tissue, plant derived waxes, industrial pollution (oil and toxic spills) and forest fire. A number of different organic compounds such as long chain fatty acids, humic acid, fulvic acid, and waxes cause hydrophobicity in soils (Ma'Shum et al., 1988; Roy and McGill, 2000). Hydrophobic compounds may leach from plant litter or may be present as humic substances or microbial degradation products on soil particles (Ma'Shum et al., 1988). Humic acids (Tschapek, 1984) and decomposing plant residue (McGhie and Posner, 1980) have been proposed as causative agents in naturally occurring hydrophobic soils. Petroleum residue and its associated compounds can induce hydrophobicity in soil as a result of industrial pollution. Recalcitrant petroleum residue and metabolic products of hydrocarbon degrading bacteria was suggested as another source of hydrophobic matter in soils (Roy et al., 1998). All constituents of crude oils are hydrophobic in nature and could impart hydrophobicity in soils. However, polar fractions of resins and asphaltenes were suggested as major contributors to soil hydrophobicity (Roy et al., 1998) because of their high microbial recalcitrance and high sorption affinity of resin and asphaltene to soils (McGill et al., 1981). Oil degrading microorganism have been known to produce high molecular weight alkanes, waxes, esters and fatty acids. Such compounds are considered as an additional source of hydrophobic substance in oil contaminated soils (Roy et al., 1998).

Moisture migration through hydrophobic or water repellent soil is often irregular and non-uniform. The soil does not wet and moisture mainly moves through fissures and cracks. Ritsema and Dekker (1993,1994 and 1998) conducted field studies on the moisture movement through hydrophobic soils. The moisture migrated only through certain sections of the soil. The soil between these sections was relatively dry (Ritsema and Dekker, 1994). With prolonged wetting, the soil turned less hydrophobic and the wetting front became more uniform.

There is significant interest in eliminating soil water repellency at some old crude oil spills in Alberta, Canada. It is possible that if the moisture distribution in the hydrophobic soil be made uniform and then maintained these soils can sustain normal vegetal growth.

The hydrophobicity in soil results in

• dry patches and poor soil wetting causing increased erosion by wind and water

- accelerated solute migration through preferential pathways
- poor seed germination
- pesticide leaching and nutrient loss

### 1.2 Objectives and Scope of the study

This research focuses on studying and improving moisture migration behavior of hydrophobic soils. The study evaluates the water infiltration response of hydrophobic soil under laboratory conditions. The study can be divided mainly into three parts: problem definition, treatment technique, soil amendments (amelioration of hydrophobicity) and plant growth (confirmation of the change in soil behavior from hydrophobic to hydrophilic).

The specific objectives of this study are as follows:

- To investigate moisture migration through untreated hydrophobic soil
- To study the effect of cyclic wetting and drying on moisture migration through hydrophobic soil
- To study the effects of dilution (mixing of hydrophobic soil with hydrophilic soil, soil conditioners and kaolinitic clay) on the moisture intake ability of hydrophobic soils
- The growth of plants in diluted hydrophobic soil

### 1.3 Thesis layout

This thesis consisted of five chapters. A broad layout of each chapter is given below.

- Chapter 1 gives the general introduction of hydrophobic soil, its worldwide occurrence, and objective of the present study.
- Chapter 2 describes the causative agents of hydrophobicity and their characterization, response of hydrophobic soils to moisture migration, and phenomenon of preferential flow.
- Chapter 3 describes the field soil sampling, hydrophobic soil classification and test methods conducted in this study.
- Chapter 4 describes the results from laboratory investigations and detailed analysis.
- Chapter 5 provides conclusions and the recommendations for future studies.

### Chapter 2

## LITERATURE REVIEW

A soil that is difficult to wet and absorbs water very slowly is called hydrophobic or water repellent soil. Hydrophobic soil resists or reduces water infiltration into soil, for a period that is dependent on the degree of hydrophobicity of soil. However, no empirical relationship has been established between the degree of soil hydrophobicity and the time a water drop takes to penetrate into hydrophobic soil. The water droplet does not penetrate the soil because the soil particle surface may be coated with hydrophobic organic substances that repel water (Doerr et al., 2000) (Fig. 2.1). The time the water drop stays on the soil surface, depends on the degree of hydrophobicity of soil. It is suggested that soil water repellency is not an abnormal phenomena (Dekker and Jungerius, 1990; Wallis and Horne, 1992).

It is generally believed that hydrophobicity of soil is a seasonal property. Hydrophobicity is most extreme when the soil is dry and eventually it disappears when soil is wet during rainy season (Witter et al., 1991). There are two different opinions on the reappearance of soil hydrophobicity when wet hydrophobic soil becomes dry. One group of researchers believe that soil hydrophobicity reappear once the soil becomes dry after wetting (Valat et al., 1991; Walsh et al., 1994; Wessel, 1988) whereas Doerr and Thomas (2000) and Jex et al., (1985) reported that soil hydrophobicity does not re-establish when wet hydrophobic soils become dry. They believe that hydrophobicity would reappear only in soil in which hydrophobicity is caused by fungal or microbial or biological activities (Jex et al., 1985), or in soils located near root zones. Re-establishment of hydrophobicity in dry soils after wetting requires fresh input of hydrophobic material.



Fig. 2.1: Water droplets on hydrophobic soil (modified after Doerr et al., 2000).

### 2.1 Occurrence of hydrophobic soils

Hydrophobic soils have been reported in many parts of the world and under various conditions. Hydrophobicity in burned forest soils were reported by De Bano and Rice (1973). The burned soils were varying in texture from sand to clay loam. The soils were covered with vegetation or trees ranging from grass, hardwoods, and conifers. Hydrophobicity caused by forest fire was also reported in South Africa by Scott and Van Wyke (1990). The soils were derived from sandstone colluvium, shale lenses and deeply weathered granite. The soils were sandy to silty loam with clay content of about 10%.

Hydrophobicity associated with citrus groves was reported by Jamison (1947). The soil underneath citrus trees consisted of fine-grained sand devoid of any clay and contained 1 to 2% organic matter. The soil was very porous and retained very little water. The sandy subsoil under citrus trees remained unwetted.

Barrett and Slaymaker (1989) reported hydrophobicity in sub-alpine soils of British Columbia, Canada. The surficial hydrophobic soil deposits were overlying Cretaceous sedimentary bedrock. Soil mainly consisted of loess, colluvium and talus deposits. In some cases the hydrophobic soil layer was coincident with the root mat.

Hydrophobic soils associated with golf sand greens were reported by Hudson et al., (1994). Hydrophobic soils associated with golf green occur as dry spots and are commonly called as localized dry spot (LDS). The hydrophobic soils usually found in LDS are predominately sand with dry grass cover.

Hydrophobicity found is Australian soils were classified as natural hydrophobicity and were caused by fungal growth (Ma'Shum et al., 1988; Ma'Shum and Farmer, 1985; McGhie, 1980; King, 1981). The hydrophobic soil consisted of siliceous sand, less than 5% clay and 1% organic carbon content.

Hydrophobic soils in New Zealand were reported by Wallis et al., (1991) and Horne and McIntosh (2000). These hydrophobic soils consisted predominately sand. The sand was covered with grass and lupins. The soils were derived from a parent material largely of quartz and feldspar mineral. The proportion of silt and clay varied between 1% in youngest sands to 10% in the older soils.

In the Netherlands, hydrophobicity associated with urban sandy soils was reported by Bisdom et al., (1993). Dekker and Jungerius (1990) and Dekker and Ritsema (1993) found hydrophobic soils associated with dune sand in the Western coast of the Netherlands. The sandy soils were of eolian origin. Dekker and Ritsema (1996a) also reported the presence hydrophobicity in peaty clay soil in the Netherlands. All the clayey hydrophobic soil was covered with pastures. The organic content of the peaty soil or clayey peaty soil was in the range of 20-70%. The development of hydrophobicity under cultivated soil was reported by Chan (1992) in Australia. The soil samples were obtained by coring from 10 years old tillage site. The soil was classified as medium textured clay loamy soil.

Hydrophobicity caused by industrial pollution as a result of crude oil spill in Alberta, Canada, was reported by Roy et al., (1998). Three sites that were studied were Ellerslie (ELL), Devon (DEV) and Bruderheim (BRU). The hydrophobic soil from ELL was classified as silty clay loam developed in saline glaciolacustrine sediments. The DEV hydrophobic soil consisted of clay loam developed in alluvial lacustrine environment of deposition. The hydrophobic soil from BRU site consisted of dune sand and developed on eolian parent material.

### 2.2 Soil texture and hydrophobicity

Soil hydrophobicity in the past has been related to the coarse-grained soil (McGhie and Posner, 1980; De Bano, 1991). It was assumed that coarse-grained particles are more susceptible to develop hydrophobicity because of their small surface area per unit volume as compared to the fine-grained soil (Blackwell, 1993). A relatively small amount of hydrophobic organic matter is needed to coat coarse soil particles as compared to the fine soil particles. An increase in soil hydrophobicity with increasing grain size was reported by Crockford et al., (1991). De Bano (1991) concluded that soil hydrophobicity is most

likely to develop in soil with less than 10% clay content. With increasing clay content in soil, an increased amount of hydrophobic organic matter is required for developing hydrophobicity. It is now established that addition of dispersible clay is very effective in reducing hydrophobicity in sandy soils in Australia (Carter and Hetherington, 1994). Soil hydrophobicity is not only restricted to sandy soils but also common in soil with considerable clay contents. Giovannini et al., (1983) reported that water repellency was observed in soil containing 40% clay in Italy. Dekker and Ritsema (1996c) also reported hydrophobicity in heavy basin clay soils in Netherlands.

Some studies have found that finer fractions of hydrophobic soil exhibit higher degree of hydrophobicity than the coarser fractions due to higher sorption of hydrophobic organic matter by fine-grained soil (Doerr et al., 1996; de Jong et al., 1999). The higher degree of hydrophobicity in finer soil fraction may be due to the presence of fine particulate hydrophobic organic matter (de Jong et al., 1999). A more general conclusion is that soil hydrophobicity when encountered in fine-grained soil can be more severe whereas coarse-grained sandy soils are more prone to develop hydrophobicity due to their small surface area per unit volume (Crockford et al., 1991; Chan, 1992; Doerr et al., 1996).

### 2.3 Characterization of soil hydrophobicity

The characterization and identification of naturally occurring hydrophobic substances is very difficult and complex. The substances that cause soil hydrophobicity are definitely organic but the amount of organic carbon and the degree of hydrophobicity are not directly related (De Bano et al., 1976). Despite the advancement in analytical techniques, the identification of compounds causing soil hydrophobicity is yet not known.

Attempts have been made to isolate and characterize the nature of organic substances by using different extractants. Methanol, ethanol, chloroform, iso-propanol, ether, and benzene have been used for this purpose. The relative effectiveness of each solvent depends on the nature of hydrophobic coatings (Ma'Shum and Farmer, 1985; Ma'Shum et al., 1988; McGhie and Posner, 1980; Roy and McGill, 1998). The effectiveness of

solvent used for the extraction of hydrophobic substances depends on the polarity of the solvent. Non-hydroxylics solvents like ether, chloroform and tetracholoretylene had almost no effect in reducing hydrophobicity (Ma'Shum et al., 1988). The effective solvents may render hydrophobic soil to hydrophilic, but the extracted hydrophobic molecules may or may not resemble the hydrophobic material present in hydrophobic soil layer due to cleavage between the solvents and the extractants.

The techniques that have been used for the characterization of hydrophobic coatings are chromato-graphic, infrared, nuclear magnetic resonance (NMR), gas chromatography / mass spectrometry (GC/MS) (Ma'Shum et al., 1988; McGhie, 1980; Giovannini and Lucchhesi, 1984; Horne and McIntosh, 2000; Roy et al., 1998).

Ma'Shum et al., (1988) used TLC (thin layer chromatography) techniques. Hydrophobic material extracted with a Soxhlet apparatus by various organic solvents was applied to thin layer chromatographic plates (TLC) and the plates were developed with chloroform. Infrared spectra for hydrophobic material were obtained by using KBr (potassium bromide). Gas chromatography-mass spectrometer analyses were carried out by using Helium (He) as a carrier gas.

Horne and McIntosh (2000) identified the extracted fraction by using TLC, gas-liquid chromatography (GLC), ultraviolet (UV) and infrared (IR) spectroscopy. They conducted TLC studies by using silica plates through sequential development, first in hexane, then toluene, and finally in a hexane:diethyl ether:acetic acid (80:20:1) mixture. The lipid material was detected under UV light. The fatty acids were converted into their methyl esters and analyzed using GLC.

Roy et al., (1998) used solid-state <sup>13</sup>C nuclear magnetic resonance (NMR) spectroscopy with cross-polarization and magic angle spinning (CPMAS) to characterize the organic carbon extracted from Devon hydrophobic soil.

It is reported that hydrophobicity can be eliminated when hydrophobic compounds were extracted with mixture of IPA (iso-propanol)/NH<sub>4</sub>OH (ammonium hydroxide) in Soxhlet extraction (Ma'Shum et al., 1988). Aromatics, aliphatic compounds like n-fatty acids.

Normal-alkanes and cycloalkanes can also be extracted with IPA/NH4OH. Roy et al., (1998) applied eight solvents with different polarity for the extraction of hydrophobic material from Devon soil. These eight solvents were cyclohexane (CCH) and dichloromethane (DCM) (non-polar), methanol (polar), n-propanol and iso-propanol (amphiphilic) and three mixtures of isopropanol in different aqueous concentrations. MED value was measured on soil samples after extraction. It was found from MED value that non-polar solvents (CCH and DCM) had the lowest extraction efficiency. Extraction with CCH and DCM slightly increased soil hydrophobicity (Roy et al., 1998). The amphiphilic solvents had the highest extraction efficiency. Methanol reduced the soil hydrophobicity but not as effective as amphiphilic solvents. Extraction of hydrophobic compounds with NaOH (Miller and Wilkinson, 1977; Karanok et al., 1993) and H<sub>2</sub>O<sub>2</sub> (Bisdom et al., 1993) was also successful in eliminating soil hydrophobicity. However, NaOH and  $H_2O_2$  extracted organic matter non-selectively. McGhie and Posner (1980) showed that hydrophobicity of soil increased when treated with chloroform. This increase was attributed to molecular re-orientation of organic matter or redistribution of hydrophobic matter on the hydrophilic surfaces (Ma'Shum et al., 1988). McGhie and Posner (1980) concluded that the aqueous solvents reduced hydrophobicity in soils whereas extraction with organic solvents increased soil hydrophobicity.

The characterization of hydrophobic compounds that imparts soil hydrophobicity is very difficult. There are a number of reasons for this.

- Firstly, the non-destructive extraction methods do not eliminate soil hydrophobicity completely.
- Secondly, the extracts from hydrophobic soils contain naturally occurring hydrophobic compounds that are also present in hydrophilic soils.
- Thirdly, it is shown by Ma'Shum and Farmer (1985) that soil hydrophobicity can be eliminated with out the removal of any compound from hydrophobic soil. Conformational changes in hydrophobic organic compounds have been described as a cause in changing soil behavior from hydrophobic to hydrophilic.

Compounds that have been extracted from hydrophobic soils and identified can be divided into two broad groups.

- First are the aliphatic hydrocarbons in which hydrogen and carbon atoms are arranged in an elongated chain. These aliphatic hydrocarbon compounds are non-polar and insoluble in water.
- The second consists of polar substances of amphiphilic structure. These compounds compose of hydrocarbons chain with one end being hydrophobic and other end being hydrophilic. These amphiphilic compounds can produce hydrophobicity depending on the orientation.

### 2.4 Causes of soil hydrophobicity

Hydrophobicity is generally attributed to the presence of organic matter on soil particles. Organic matters includes roots, decomposing plant tissues, plant derived waxes, industrial pollution (oil, toxic spills and old waste dumps) and forest fire. The current theory proposes that naturally occurring amphiphilic compounds (such as fatty, fulvic and humic acids) cause the soil to behave as hydrophobic (De Bano, 1981; Doerr et al., 1998; Wallis et al., 1990; Ma'Shum et al., 1988; Franco et al., 1994; Roy and McGill, 2000). However, it is suggested that two conditions are necessary to give rise to soil hydrophobicity (Roy, 1999).

- Accumulation of hydrophobic organic material in soil
- Re-distribution and re-arrangement of accumulated hydrophobic organic material

Vegetation and soil fungi and microorganism contribute to organic matter in soil.

Fresh and decomposed plant material provides a source of organic matter in soil. Green plants are commonly associated with soil hydrophobicity. Trees with amount of resins, waxes or aromatic oils (eucalyptus and pines) are the major contributor of organic matter in soil. Soil hydrophobicity has been found under shrubs (Malik and Rehman, 1985) and grassland (Crockford et al., 1991).

Soil hydrophobicity is also associated with fungal growth and soil microorganism. Soil fungi and microorganism that have been associated with soil hydrophobicity are *Penicillium nigrican* and *Aspergillus sydowi* (Savage et al., 1972) and *Actinomycetes* (Jex et al., 1985). Effects have been found to be species dependent. It is observed that some species induce hydrophobicity, and others reduced hydrophobicity in already hydrophobic material (McGhie and Posner, 1981). It is very difficult to isolate microbes and fungi because they are also involved in the decomposition of hydrophobic organic matter into soil is given in Fig. 2.2.

Different mechanisms have been proposed as causative agents of soil hydrophobicity. These are:

- Organic matter coating of soil particle
- Forest fire
- Conformational changes in hydrophobic organic matter

#### Organic matter coating

Hydrophobicity has been attributed to the coating of hydrophobic organic compounds on soil particles (De Bano, 1969). The composition of organic matter coating is not yet known but presumably it consists of waxes and oils (Ma'Shum et al., 1988). Ma'Shum et al., (1988) reported that long chain fatty acids are the primary cause of water repellency. Franco et al., (1995) mentioned that the transfer of lipids causes hydrophobicity in soils from particulate organic matter onto soil particle. Their studies concluded that hydrophobic organic coatings were an important factor in the development of soil hydrophobicity.



Fig. 2.2: A summary of causative agents that contribute in the development of hydrophobicity in soils (redrawn after Doerr et al., 2000).

Soil particle can be coated with organic matter in a number of ways.

- Organic matter leached out of plant litter coat the soil particles. This phenomenon was observed in sandy coarse-grained soils (Robert and Carbon, 1971; Van't Woundt, 1959).
- Hydrophobic microbial by-products coating of a mineral soil particle may impart soil hydrophobicity (Bond, 1969).
- Mixing of non-coated mineral soil particle with organic coated particles may
  partially coat the non-coated soil particle, thereby inducing soil hydrophobicity
  (De Bano, 1969; Bisdom et al., 1993; Franco et al., 1995). Heating is one source
  of mixing organic matter coated soil particle with non-coated soil particle.
  Heating distills hydrophobic organic coating and causes the organic matter to coat
  the adjacent mineral soil surfaces.

#### **Forest Fire**

The second cause of soil hydrophobicity, which is common in North America, is forest fire. Organic matter accumulates in the litter layer during intervals between fires. The soil layer becomes hydrophobic due to the mixing of partially decomposed organic matter and mineral soil as well as due to the leachate from decomposing plant material deposited on the upper surface layer (De Bano, 1981). Apart form redistributing and concentrating hydrophobic substances in the soil, heating also improves the bonding of hydrophobic substance to soil particles (Savage et al., 1972). However, fire-induced soil hydrophobicity is temporary in nature (Doerr et al., 1998). De Bano (1991) suggested that the heating of hydrophilic soil containing more than 2-3 % organic matter could induce hydrophobicity. A fire can cause hydrophobicity when heating temperature is between 175 to 200°C.

#### Conformation changes in hydrophobic organic matter

The third and the most widely accepted mechanism involve the orientation of naturally occurring substances. Tschapek (1984) and Ma'Shum and Farmer (1985) suggested that

it is the orientation of naturally occurring organic substances that determines whether the soil will absorb or repel water.

According to this mechanism, an amphiphilic or surface-active organic molecule, such as humic, fulvic and fatty acid can impart hydrophobic characteristics to the soil when their hydrophobic ends are oriented towards soil open space and the hydrophilic ends are towards soil particles (Fig. 2.3) (Wallis et al., 1990). Various solvent treatment methods usually change the interfacial conformation of amphiphilic organic molecules, causing a change in soil behavior (Ma'Shum and Farmer, 1985). Some naturally occurring organic substances, which are associated with natural hydrophilic soils, can induce hydrophobicity depending on the conformation of the organic molecules. It is suggested that when soil is wet, polar groups interact with water molecules. Upon drying when water is lost, polar groups interact with each other and the organic matter present on soil surface in non-polar grouping which resist wetting of soil (Tschpek. 1984; Ma'Shum and Farmer, 1985; Ma'Shum et al., 1988).

Hydrophobicity is not only dependent on the presence of hydrophobic substances in soils, but also on their ability to display conformational flexibility at the solid-liquid interface in the soil (Roy et al., 1999).

Persistence of hydrophobicity in soil depends on the characteristics of the hydrophobic compounds present in the soils. Higher molecular weight hydrophobic organic compounds like asphaltenes, steranes or triterpanes are recalcitrant and insoluble. The whole compound or molecule may be entirely non-polar or consists of molecules that are segregated into polar and non-polar regions. Their recalcitrant nature is due to their large molecular size and strong interaction with soil surfaces.

Oil contamination is another mechanism that causes soils to become hydrophobic. The soil hydrophobicity persists several years after the oil spill took place. Polar constituents of crude oil like resin and asphaltenes fractions resist degradation and have a high affinity for mineral surface sorption. In addition to these high molecular weight crude oil

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Fig. 2.3: Schematic representation of amphiphilic molecule and change in molecules orientation on mineral surface (redrawn after Doerr et al., 2000).

constituents, low molecular weight fraction is also important because some soils have been known to develop water repellant characteristics following contamination with natural gas (McGill et al., 1981).

### 2.5 Hydrophobicity and moisture infiltration

Hydrophobicity or resistance to wettability, is a physical property of soil that fluctuates with climatic changes (De Bano, 1981). Hydrophobic soil exhibits water repellent characteristics and is very difficult to wet. However, upon prolonged exposure to moisture, hydrophobic soil can absorb water (Dekker and Ritsema, 1996a; Crockford et al., 1991). Hydrophobicity was found to be persistent in clayey peat with moisture content up to 38% (Dekker and Ritsema, 1996a) and in sandy loam containing moisture up to 22% (Doerr and Thomas, 2000).

The time period required for wetting of a hydrophilic, slightly hydrophobic and extremely hydrophobic soils are very different. Wetting is almost instantaneous in dry hydrophilic soil due to high capillary pressure. The time required to wet an extremely hydrophobic soil is significantly higher than that of a slightly hydrophobic soil (Ritsema and Dekker, 1996).

In hydrophilic soil, the initial rate of infiltration is higher though with time it tends to decrease. In hydrophobic soil, the reverse is observed. The initial rate of infiltration is low, though with time the rate of infiltration increases (Fig. 2.4). The initial infiltration rates through hydrophobic soils are extremely slow or even non-existent due to very high initial liquid solid contact angle (De Bano, 1981; Wallis at al., 1991). The hydrophobic soil once it gets wetted conducts water as rapidly as a hydrophilic soil. Soil hydrophobicity can be prevented by maintaining a high soil moisture level and by irrigating the soil frequently so it does not become dry. As long the soil remains moist, soil water repellency phenomenon does not occur (Cisar et al., 2000).



Fig. 2.4: Water infiltration rates in hydrophobic and hydrophilic soil (modified after De Bano, 1981).

The following mechanisms have been suggested to explain water infiltration through hydrophobic soils.

- Water vapors moves freely through hydrophobic soil. The vapor migration of water causes soil water to be re-distributed (Barrett and Slaymaker, 1989). The process of vapor migration and redistribution continues until the hydrophobic soil has reached its maximum adsorption capacity for individual water molecules (Miyamoto et al., 1972).
- Some hydrophobic soil particle may have changed to a hydrophilic state during vapor adsorption, allowing further water intake (Doerr et al., 2000)
- Wettability of hydrophobic soil increases with time once the soil is placed in contact with water as the hydrophobic substances in soil may somewhat dissolve in water, thereby, increasing wettability.
- Wettability of finer fraction of hydrophobic soil present in the pore-spaces of hydrophobic soil matrix may be responsible for partial wetting of the hydrophobic soil (Imeson et al., 1992).
- Changes in soil hydrophobic characteristics upon drying may also aid in improving soil wettability. The organic matter coating the soil particle may not be continuous and water may move into soil by vapor diffusion. Repeated drying of hydrophobic soil may enhance the discontinuities in organic matter.

#### 2.5.1 Infiltration and overland flow

Moisture moving through hydrophobic soil is generally uneven (Fig. 2.5). Moisture does not penetrate uniformly into hydrophobic soil but it infiltrates downward unevenly through narrow channels, leaving soil in sections between these channels dry. This leads to varying moisture distribution in hydrophobic soils. Moisture penetrating through a hydrophilic soil layer overlying a hydrophobic layer may:



Fig. 2.5: Water movement through hydrophobic soil and resulting surface runoff (redrawn after De Bano, 1981).

- be stored in hydrophilic layer and later taken up by plant transpiration
- be spread as a distributed flow and move vertically downwards through preferential channels or structural gaps or through vertical cross-sections of less hydrophobic soil layer
- move laterally down slope above the hydrophobic layer
- enter the matrix of hydrophobic soil layer if water entry pressure is sufficient, and change the soil characteristics to hydrophilic

### 2.6 Variation of hydrophobicity with soil moisture

Hydrophobicity is usually most severe in dry soils and decline as soil moisture content increases. Below critical moisture content, the soil is hydrophobic and above it the soil is hydrophilic (Dekker and Ritsema, 1994; Witter et al., 1991; Carter et al., 1994). The critical moisture content is the minimum amount of moisture at which soil changes its behavior from hydrophobic to hydrophilic (Dekker and Ritsema, 1994). The value of the critical moisture content varies with soil type. The degree of water repellency can be influenced by seasonal changes, i.e. rise and lowering of water table. That is why it is suggested that hydrophobicity measurements always be made on the oven-dried samples.

Dekker and Ritsema (1994) distinguished actual water repellency and potential water repellency. Potential water repellency represents the maximum water repellency a soil can have. Hydrophobicity measured on the field moist samples has been termed as Actual Water Repellency, whereas that measured on oven-dried samples has been called as Potential Water Repellency (Dekker and Ritsema, 1994). Potential water repellency is not a time dependent parameter but may vary from place to place due to local variations in vegetation type and organic matter.

Heating also has an effect on the severity of the water repellency of soil (Dekker and Ritsema, 1996b; Franco et al., 1995). However, there is contradicting evidence about the

effect of moisture content and heating on degree of hydrophobicity. Kings (1981) suggested that water repellency does not change by changing moisture content from oven to air-dry whereas Berglund and Perrson (1996) observed that water repellency decreased with an increase in moisture content. Franco et al., (1995) found that hydrophobicity increased when determined on soil samples dried at higher temperatures (Franco et al., 1995), whereas Ritsema et al., (1997) reported no effect of temperature on water repellency of a soil when dried at 25°C, 45°C and 65°C. At this stage no conclusion can be drawn about the effect of temperature and moisture content on the degree of soil hydrophobicity.

### 2.7 Re-establishment of hydrophobicity after wetting

As already discussed soil hydrophobicity is dependent on the soil moisture content. It is usually absent or low under wet conditions and is most severe during dry periods (Ritsema and Dekker, 1994; Crockford et al., 1991). It is observed that hydrophobicity becomes reestablished upon drying of wet hydrophobic soil (Valat et al., 1991; Walsh et al., 1994). The usual explanation for reestablishment of hydrophobicity is that after drying, polar ends of amphiphilic molecules associate and interact with hydrogen bonds. This forces the molecules to re-orientate in a position in which polar ends (water repellent) towards pore-spaces as shown in Figure 2.3 (Ma'Shum and Farmer, 1985; Tschapek, 1984; Valat et al., 1991).

Bruch et al., (1989) observed in the field that some hydrophobic soils became hydrophilic upon exposure to moisture, but retained hydrophilic characteristics even upon drying. On the other hand Crockford et al., (1991) found that it took 6 to 9 days of hot and dry weather for hydrophobicity to re-establish in the field.

However, under laboratory conditions, Doerr and Thomas (2000) has found that hydrophobicity of a fine and medium textured soil did not re-appear upon drying once it had disappeared after wetting. Furthermore, it has been shown that heating (oven-drying)
of force wetted hydrophobic soil can restore hydrophobicity but to a reduced level. This might be due to the reattachment of the released organic molecules on soil surface as a result of heating (Doerr et al., 2000).

Research to date indicates conflicting and varying explanations about the principles controlling the disappearance and reappearance of hydrophobicity, and the effects of soil moisture on soil hydrophobicity.

## 2.8 Variation in hydrophobicity with the depth of soil profile

The degree and extent of soil hydrophobicity depends on the process that rendered the soil hydrophobic. In unburnt soil, the degree of hydrophobicity decreases with depth (Barrett and Slaymaker, 1989; Jungerius and De Jong, 1989). This reduction in soil hydrophobicity with depth is attributed to an increase in soil moisture content, due to recharge from water table. For burnt soil, the pattern is quite different. The high temperature at ground surface vaporizing pyrolysize hydrophobic organic compounds, which then move deeper until they condense at the colder underlying soil layers (Fig. 2.6). The forest fire produces temperatures of about 280°C (De Bano, 1981). This movement of hydrophobic compounds renders topsoil hydrophilic, with increasing hydrophobicity with depth. The variation of hydrophobicity with depth depends on the soil characteristics and the moisture content of soil.

# 2.9 Wetting Phenomenon in Soil

Wetting is the process of interaction between liquid and solid phase at the liquid-solid interface. This process includes the formation of contact angle between liquid and solid, spreading of liquid (in case of hydrophilic soil) and displacement of fluid (air in solid) from solid phase. Soil wettability is a complex function of many factors such as surface



Fig. 2.6: Fire induced soil hydrophobicity. A. hydrophobic substances accumulate in top soil layer; B. fire burns vegetation and the hydrophobic substances move to deeper layers and C. after fire, hydrophobic substances present below the burned soil (redrawn after De Bano, 1981). chemistry, roughness, porosity and fluid characteristics. These factors vary with temperature, atmospheric humidity and soil moisture (King, 1981; Scot, 2000). Because of these complexities, it is difficult to measure soil wettability.

It is suggested that hydrophobicity occurs only when cohesive forces of water molecules are greater than the adhesive forces between soil surface and water molecules (Wessel, 1988). This phenomenon is demonstrated by placing a drop of water on smooth soil surface (Fig. 2.7). If the attraction between water molecule and the solid surface is greater than the attraction between the individual water molecules, then water will spread out on solid surface, else the solid surface will repel water. The mechanical equilibrium of a water droplet on solid surface as in term of free energy can be written using Young's equation:

$$\gamma_{\rm sv} - \gamma_{\rm si} = \gamma_{\rm lv} \cos\theta \tag{2.1}$$

where

 $\gamma_{sv}$  = Specific surface free energy at the solid-vapor interface

 $\gamma_{sl}$  = Specific surface free energy at the solid-liquid interface

 $\gamma_{lv}$  = Specific surface free energy at the liquid-vapor interface

 $\theta$  = Wetting angle (angle between the solid-liquid and liquid-vapor interface)

Spreading of water over solid surface only occur when work of adhesion (Wa) is greater than work of cohesion (Wc). The difference between two quantities is called spreading coefficient (S).

$$\mathbf{S} = \mathbf{W}\mathbf{a} - \mathbf{W}\mathbf{c} \tag{2.2}$$

Spreading coefficient in term of surface free energies



Fig. 2.7: Water droplet placed on the surface of hydrophobic soil and interfacial tension forces (redrawn after Watson and Letey, 1970).

$$S = \gamma_{sv} - (\gamma_{lv} + \gamma_{sl})$$
(2.3)

Spontaneous wetting only takes place when there is reduction in surface free energies and S > 0. This only occurs under non-equilibrium condition. Spreading coefficient is usually positive during adsorption of water vapors at liquid-solid interface.

At equilibrium, contact angle is static and spreading coefficient can only be zero or negative as given in equation (2.4)

$$S = \gamma_{lv} (\cos\theta - 1) \tag{2.4}$$

The fundamental principle controlling the wetting process is the surface tension of liquid. A decrease in surface tension of liquid increases its wettability. On the other hand, a decrease in the surface tension of solid substances to be wetted reduces its wettability. Thus it is the surface tension of a liquid, which controls the liquid-solid contact angle. It was suggested by Watson and Letey (1970) to use the contact angle as an index of soil wettability or hydrophobicity. If the contact angle is less than 90°, the soil was classified as wettable otherwise it was hydrophobic.

Rietveld (1978) suggested that the soil can be classified into three classes

- if contact angle is zero, then soil is hydrophilic
- if contact angle is between 0-90°, the soil is termed as low to moderately hydrophobic
- if contact angle is more than 90°, then soil is termed as extremely hydrophobic

## 2.10 Soil temperature and effect of fire on hydrophobicity

When hydrophobic soil is heated to a sufficient temperature, the heating will result in denaturing or vaporizing the organic matter or coating of organic matter present on soil particles. De Bano (1991) suggested that heating of hydrophilic soil containing more than

2-3% organic matter could induce hydrophobicity. The process of heating soil to a certain temperature changes the soil from hydrophobic to hydrophilic. On the other hand, if heating temperature is not sufficient, then heating of soil will result in an increase in soil hydrophobicity.

The heating of a soil causes the hydrophobic substances to move downward in the soil profile (Fig. 2.6) due to existing temperature gradient. The downward moving substances may have been produced by the pyrolysis. These substances are usually produced in bulk quantities when fire intensity exceeds 350°C. Fractionation of these pyrolytically produced substances results in the production of more hydrophobic substances. However, the exact nature of hydrophobic compounds produced during fire has not been known (De Bano, 2000). These moving hydrophobic substances will condense on the soil particles with increasing depth. It has also been reported that soil hydrophobicity increases when soil is heated from 200-250°C and decreases when heated above 270°C (Nakaya, 1982). Most of the researchers observed that organic substances responsible for soil hydrophobicity were eliminated or destroyed when hydrophobic soils were heated to a temperature of 270 to 300°C (Scholl, 1975; Savage, 1974). Nakaya et al., (1977) observed that the wetting front in hydrophobic soil ascends from temperature 105°C to 250°C. The similar phenomenon was observed in humic-coated quartz sands. At temperature of 250°C the humic acid coated guartz sand behaves like uncoated guartz wettable sand. On the other hand, it has been shown that hydrophobicity can be totally eliminated if hydrophobic soil heated at 200°C for 24 hours (Roy, 1999). There are several studies that conclude that hydrophobic substances could be destroyed by burning (Doemaar and Lutwick, 1975). However, there is a lack of agreement about the role of temperature and the degree at which soil should be heated to eliminate or reduce soil hydrophobicity. However, there is a general agreement among various researchers that hydrophobic behavior of soils change when exposed to heating. The difference in threshold value of temperature in various studies might be due to the difference in measuring method, length of heating time and type of chemical compounds present in the soil. It can be concluded that temperatures necessary for elimination of soil

hydrophobicity depends on soil type, degree of soil hydrophobicity (whether a soil is moderately or severely water repellent) and causative agents of soil hydrophobicity.

# **2.11 Preferential Flow**

When a small area of subsurface carry large portion of flow, it is called preferential flow. Preferential flow is a characteristic of hydrophobic soils. Hydrophobic soils often show irregular, erratic and non-uniform wetting patterns. The dry hydrophobic soil prevents downward flow of water whereas wet zones acts as channels for concentrated transport to the deeper region. These preferential flow paths may be due to the spatial distribution of hydrophobicity with in soil profile. As the degree of soil hydrophobicity decreases with depth due to increase in soil moisture, the preferential flow path begins to disappear. This suggests that the effect of hydrophobicity on water flow depends on soil moisture content (Dekker and Ritsema, 1994). Wallis et al., (1991) concluded that water infiltration in initially dry hydrophobic soil is retarded and water is retained in a top layer at first. With prolonged infiltration, minor perturbations in an originally uniform wetting front may go to form channels or fingers.

The theory of preferential flow given by Raats (1973) states that if the rate of moisture migration changes with depth, the wetting front will become unstable. This means that initially uniform wetting front will split into non-uniform flow with time and depth.

If the shape of the preferential flow path is finger or tongue like, then it is called finger or funnel flow. Finger flow is caused by instability of wetting front whereas funnel flow occurs when percolating moisture encounters lens of fine grain material interspersed within coarse material. The fingers form only during dry weather when soil moisture level in sandy hydrophobic layers is below a critical value (Ritsema and Dekker, 1994). Fingered flow can recur at the same places after successive storm events following dry spells (Ritsema et al., 1998). Bauters et al., (1998) mentioned that sandy soils with different degrees of hydrophobicity exhibit fingered flow.

There are a number of situations that can cause the water front to become unstable in unsaturated soils. Preferential flow may originate from

- cracks and macropores (Bevan and Germann, 1982)
- soil heterogeneities (stones and cavities) (Philips et al., 1989)
- textural discontinuities (along bedding) (Kung, 1990 a and b)
- from unstable wetting front (Raats, 1973; Glass et al., 1989 a and b; Selker et al., 1989; Hendrickx and Dekker, 1991)

The factors that cause the wetting front to become unstable are

- soil layering (Baker and Hillel, 1990)
- air entrapment (Glass et al., 1990)
- water repellency (Ritsema et al., 1998; Van Dam et al., 1990; Dekker and Jungerius, 1990; Hendrickx et al., 1993)
- non-ponding infiltration, with a rainfall intensity lower than saturated hydraulic conductivity (Dekker and Ritsema, 1994)

Numerous attempts have been made to study the phenomena of preferential flow both at laboratory scale and in the field using lysimeters, column infiltration tests, tracers and color dyes. Laboratory studies were mostly conducted from modeling point of view whereas field studies were conducted to understand the mechanism of preferential flow through hydrophobic soil (De Bano, 1975, 1981; Hendrickx and Dekker, 1991; Van Dam et al., 1990; Hendrickx et al., 1993; Dekker and Ritsema, 1994; Selker et al., 1989).

## 2.11.1 Laboratory studies

Most of the laboratory studies were conducted on hydrophilic soil in order to develop models to predict the flow and contaminants transport through fingers. Laboratory studies have been conducted to determine finger width and velocity of water moving through fingers. In laboratory studies, two types of setups have been employed. One setup consisted of a layered configuration, where a fluid is ponded on the upper layer of fine sand over a coarse sand layer (Fig. 2.8) (Diment and Watson, 1985, Glass et al., 1989c; Baker and Hillel, 1990). In another setup, homogenous soil was infiltrated under continuous non-ponding conditions (Diment and Watson, 1985). From field studies Hendrickx and Dekker (1991) reported the evidence of unstable wetting front in homogenous soils after rainfall. From all these laboratory studies, it was concluded that the tip of the advancing finger is saturated with water and there is decreasing moisture content behind it. The wettest zone is found at the top and soil water content decreases with depth. With increasing depth, finger merger occurs, reducing the number of fingers with depth (Glass et al., 1989b; Selker et al., 1989). Finger merger has not been observed in the field (Ritsema and Dekker, 1994).

### 2.11.2 Field studies

Most of the field experiments have been conducted on hydrophobic soils. Color dyes were used to trace the imprints of finger or preferential flow paths. (Ritsema and Dekker, 1994; Van Dam et al., 1990; Hendrickx and Dekker, 1991; Hendrickx et al., 1993. It was suggested that spatial variation of degree of water repellency in soil profile and uneven topography of the soil surface cause the water flow through certain section of soils (Ritsema et al., 1998). In all field experiments, changes in volumetric water content with depth, degree of water repellency and traces of preferential flow paths were traced (Fig. 2.9). It was observed that preferential flow decreases with successive infiltration after



Fig. 2.8: Formation and merging of finger in heterogeneous soil, i.e. finer over coarselayer of hydrophilic sandy soils (redrawn after Glass et al., 1989c).



Fig. 2.9: Uneven wetting patterns caused by soil hydrophobicity in sandy soils (modified after Doerr et al., 2000).

long dry period (Dekker and Jungerius, 1990). The dry soil in between these preferential flow paths remained dry in contrast to the preferential flow path, which became wet. This preferential flow path can persist for long periods and may disappear due to rise of water table saturating the entire soil, or during an extensive drought (Ritsema and Dekker, 1994). Fingers in hydrophobic soil may recur at same location during new rainfall events (Ritsema and Dekker, 1996). A two-dimensional flow pattern through hydrophobic soil was developed and is shown in Fig. 2.10. Figure 2.10 illustrates the flow and solute transport through hydrophobic soil. Ritsema and Dekker (1993) conducted a field study of moisture movement through hydrophobic soil. They observed:

- a distributed flow in top layer (it is lateral flow over and through soil profile towards places where vertical infiltration actually occurs)
- preferential flow through hydrophobic soil below top soil and
- diverging flow in wettable soil layer below hydrophobic soil

A new aspect emerged from this study was the occurrence of diverging flow in a wettable zone above hydrophobic soil layers. This diverging layer may counteract the rapid transport through hydrophobic soil layer as water and solute from hydrophobic soil layer may be redistributed in the wettable zone. So the solute cannot bypass any soil matrix in wettable layer. From these field studies, it is concluded that (Hendrickx et al., 1991; Dekker and Ritsema, 1994).

- Infiltration rates are slower in hydrophobic soils than wettable soil
- Wetting patterns are irregular, erratic and incomplete in hydrophobic soils
- Water repellency has greatest effect on dry soil



Fig. 2.10: Two-dimensional flow pattern visualization in hydrophobic soil (redrawn after Ritsema et al, 1993).

# 2.12 Remediation of hydrophobic soil

Reduced crop growth is associated with hydrophobic soil. It is estimated that hydrophobicity and its associated phenomena caused 40% reduction in crop production in Australia (Blackwell et al., 1994). Different techniques have been developed and applied to overcome the problem of water repellency and to enhance water infiltration, thereby increasing crop growth. The amelioration or removal of soil hydrophobicity requires a knowledge of causative agents that cause hydrophobicity in soils (Franco et al., 1994). These techniques include application of clay as top layer (claying), spraying of wetting masking. furrow sowing. and wax degradation by microorganism agents, (biodegradation) and deep cultivation (sub-soiling) (Blackwell, 1993; Franco et al., 2000). The success of any individual or multiple treatments technique depends on soil type, cost-benefit ratio and the long-term effect of soil behavior. The different techniques used in amelioration of soil hydrophobicity are shown in Fig. 2.11.

Holzhey (1969) suggested that dilution of hydrophobic soil with hydrophilic soil would allow water infiltration into the soil profile. The dilution is usually done through cultivation. The cultivation also involves the abrasion of soil particle. It is generally believed that soil particle abrasion remove or decrease the soil hydrophobicity (Bisdom et al., 1993; Ma'Shum and Farmer, 1985).

Soil claying, spreading of large amount of clay on top of the hydrophobic soil layer, is very common in Australia. Robert (1966) found that amending of hydrophobic soil with fine textured soils (clay, fly ash and silica) could overcome the effect of hydrophobicity. McGhie and Posner (1981) reported that an addition of 3% clay in hydrophobic soil decreased water drop penetration time (WDPT) from minutes to seconds. Harper and Gikes (1994) reported that an addition of 1-2% clay in hydrophobic soil changed the soil from hydrophobic to hydrophilic. The success of claying depends on clay mineralogy, dispersitivity, exchangeable cations (Blackwell et al., 1994; Deller et al., 1994; Wardes

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Fig. 2.11: Schematic of techniques used in the management of hydrophobic soils (redrawn after Blackwell, 2000).

and Oades, 1993). The dispersive clay does not aid in removing hydrophobicity but it is used to mask soil hydrophobicity by concealing non-wettable soil particles. Ward and Oades (1993) showed that clay addition to hydrophobic soil is not effective unless clay mixed hydrophobic soil is exposed to a wetting and drying cycle. The wetting phase caused clay to disperse in hydrophobic soil and during drying the surface tension forced sand and clay into intimate contact. These resulted in lowering of hydrophobicity. Cann (2000) reported that application of clay on hydrophobic soil also improves pH of soil from acidic to neutral. The addition of clay may also provide a source of nutrients like potassium and phosphorous. It has been shown in Western Australia that addition of 100 t/ha of kaolinitic clay can reduced water repellency to near zero (Blackwell, 1993). The application of clay on hydrophobic soil resulted in increased infiltration of moisture in soil, uniform wetting of soil and germination, reduced erosion of soil by crust formation and increased microbial activity due to soil wetness. The main concern with clay mixing and spreading is the expense of adding and mixing with the soil.

Masking is a technique used in amelioration of hydrophobic soils. In masking, clay is applied on hydrophobic soils. The clay particles cover the hydrophobic soil surface, and improve water infiltration slowly. The masking process also helps in reducing surface water contamination by acting as an adsorption sites (Deller et al., 1994).

Lime has been employed to improve the wettability of hydrophobic soil by providing additional fine material, enhancing mineralization of organic matter and dissolution of hydrophobic organic compounds. It has been found that lime does not remove hydrophobicity but alter soil physical properties, i.e. density and particle size distribution. On the other hand, a high rate of application of lime can cause nutrition problems in soils (Blackwell, 1993).

The mechanism reducing soil hydrophobicity by liming is described as follows. Fulvic acid (FA) is soluble in water at any pH and humic acid (HA) is soluble in water at pH > 6.5. It is known that both the fulvic and humic acid decreases the surface tension of water, which in turn decrease the solid-liquid contact angle. The reduction of solid-liquid contact angle means increase in water infiltration. The surface tension of fulvic and

humic acid in solution decreases with increasing pH. Addition of lime to soil increases pH and hence increases the ability of fulvic and humic acid to increase water infiltration (Chan and Schnitzer, 1978).

Wetting agents are chemicals frequently employed for combating hydrophobicity in turf grass. Mustafa and Letey (1970), and Miller et al., (1975) employed wetting agents to treat hydrophobic soils in order to increase water infiltration. Heavy irrigation is required to limit the toxicity when wetting agents are used in removing hydrophobicity. This factor may have limited the use of wetting agent in amelioration of hydrophobic soil. These studies concentrated in evaluating the role of wetting agents in treating hydrophobic soils, evolution of their movements and leaching through hydrophobic soils.

Addition of wetting agents in water allows the water to flow through soil. The addition of wetting agents in water decreases the surface tension of solution. A decrease in surface tension of solution will increase infiltration into hydrophobic soils (Miyamoto, 1985). Hydrophobic soil treated with wetting agents remains water wettable after drying because the wetting agents molecules are absorbed by the soil particles (McGhie, 1987,). The benefit-cost ratio have limited the use of wetting agents on large scale application and the success of using wetting agents in the field to remediate water repellent soil vary (De Bano, 2000).

Sowing plant in wide furrows was suggested to increase water infiltration into hydrophobic soil (Blackwell, 1993). Widely spaced furrows increased ponding and the ponded water slowly infiltrate into soil. The main problem with furrow sowing is the erosion of soil caused by wind and rapid loss of moisture due to evaporation.

Fire produced high heating temperatures. Though the relationship between heating temperature and hydrophobicity is complex but high heating temperatures have been used to render hydrophobic soil to hydrophilic (Scholl, 1975).

Extraction techniques have been used to remove soil hydrophobicity. It is found that extractions do not extract all lipid materials but they may facilitate the change in surface

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chemistry (Horne and McIntosh, 2000). The limitation of extraction techniques is that these cannot be applied in field at large scale.

A new approach for removing hydrophobicity involves the addition of wax degrading bacteria into hydrophobic soils. The bacteria remove the hydrophobic substances from the surface of soil and improve its water repellent character (Blackwell, 1993). The biological activity and rate of break down was very slow in hydrophobic soils. Slow releasing of fertilizer into hydrophobic soil increased the microbial population and activity and as a result, break down of hydrophobic substances was stimulated (Franco et al., 2000).

# **Chapter 3**

# **MATERIALS AND METHODS**

# 3.1 Soil Sampling and classification

Hydrophobic soils from three different sites were obtained for this study. The three sites are located in central Alberta near the towns of Devon, Stettler and Redwater. Hydrophilic soils were also collected from adjoining locations in Devon and Stettler.

The Devon soil is classified as a Gleyed Eluviated Black Chernozem (The Canadian System of Soil Classification, 1987) that developed from alluvial kind of parent geological material. The Devon hydrophobic soil was dark brown whereas the hydrophilic soil from adjoining locations was brownish black. At hydrophobic location hard soil layer was present about 10 cm below surface. A crude oil spill occurred at the Devon site in 1947 from Atlantic No. 3 oil well and later the site was remediated and returned to the land owner (Kerr, 1986). Today, the site contains patches of barren hydrophobic soil (Fig. 3.1). Hydrophobic soil was collected after scrapping off the top 1 cm of soil from surface. Hydrophilic soil was sampled from adjoining areas where crops were growing. Field identification of hydrophobic and hydrophilic soils were made by placing a drop of water on soil surface. The residual oil contents of hydrophobic and hydrophilic soils were  $6.5 \pm 0.3$  and  $1.5 \pm 0.1$  gm per kg of soil respectively (Roy, 1999).

Stettler hydrophobic soil is classified as Gleyed Black Chernozem (The Canadian System of Soil Classification, 1987). The soil is associated with alluvial deposits. The hydrophobic soil was brown in color whereas the hydrophilic soil was light gray. Lumps of asphalt or tar were found mixed with the surface soil. A hard pan layer underlay the surface soil. Hydrophobic and hydrophilic soil was collected after removing the top 1



Fig. 3.1: Patch of hydrophobic soil surrounded by hydrophilic soil at Devon site.

cm of the soil. Barren hydrophobic patch surrounded by hydrophilic soil at Stettler is shown in Fig. 3.2.

Hydrophobic soil from Redwater was provided by Imperial Oil Ltd., Calgary. The Redwater hydrophobic soil was classified as an Orthic Humic Regosol (The Canadian System of Soil Classification, 1987) developed from eolian (wind-blown) parent geological material. It was medium to coarse-grained soil, mainly sand, and brown in color.

All laboratory tests were conducted on soil passing through 2 mm-sieve. Sieved soil was thoroughly mixed for homogeneity before use in laboratory analysis. Particle size analysis was performed using ASTM 422-63 (ASTM, 1994) and results are shown in Fig. 3.3. According to USDA (1975) textural classification, the Devon soil was classified as loamy clay whereas both the Stettler soil and the Redwater soil were classified as sand.

All three hydrophobic soils were categorized as severely water repellent with molarity of ethanol droplet (MED) (King, 1981) values of 5.2, 5.2 and 4.2 for Devon, Stettler and Redwater soil respectively. They were also classified as extremely water repellent according to water drop penetration time (WDPT) test (Dekker and Jungerius, 1990). A summary of the soil classification is given in Table 3.1.



Fig. 3.2: Patch of hydrophobic soil surrounded by hydrophilic soil at Stettler site.



Fig. 3.3: Particle size distribution of three hydrophobic soils.

Sampling Site	Soil Classification				
	Canadian System of Soil Classification <sup>1</sup>	USDA <sup>2</sup>	MED <sup>3</sup>	WDPT*	
Devon	Gleyed Elluviated Black Chernozem	Loamy clay	Severe repellency	Extremely non-wettable	
Stettler	Gleyed Black Chernozem	Sand	Severe repellency	Extremely non-wettable	
Red Water	Orthic Humic Regosol	Sand	Severe repellency	Extremely non-wettable	

 Table 3.1: Soil Classification

<sup>1</sup>The Canadian system of soil classification, Agriculture Canada, (1987)

<sup>2</sup>United States Department of Agriculture textural classification (1975)

<sup>3</sup>Soil classification as per King (1981)

<sup>4</sup>Soil classification as per Dekker and Jungerius (1990)

# 3.2 Soil Conditioners

Five types of soil conditioners were used. These were Green Compost (made from tree barks), Peat Moss (Sphagnum moss), Organic Compost (contained minimum 25% organic matter), Sheep Manure, and Dolomitic Lime. All soil conditioners were obtained commercially. Manufacturer given names were used in this study.

In addition to soil conditioners, hydrophobic soil was also mixed with Pioneer kaolinitic clay. The clay consisted of 55-65% particles finer than 2  $\mu$ m. SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> were the major constitutients. Kaolinitic clay was preferred over other types of clays due to its high dispersitivity. The clay was oven-dried before use.

# 3.3. Methods

The following tests were conducted in this study. A detailed description of the tests conducted on different soils is given in Table 3.2.

- WDPT and MED tests for soil classification according to degree of hydrophobicity
- Laboratory infiltration tests and moisture content measurements with depth (gravimetrically), i.e. mapping of wetting front movement with depth
- Cyclic wetting and drying (oven and air) followed by infiltration tests
- Infiltration tests on soil samples having ratios of hydrophobic and hydrophilic soil
- X-ray CAT scanning (Computer Assisted Tomography) for 3-D imaging of moisture profiles
- Pore size distribution in hydrophobic soil by mercury porosimeter
- Soil conditioners and kaolinitic clay application
- Abrasion test to study the effect of particle abrasion on soil hydrophobicity

Figure 3.4 gives a schematic of the sequence in which tests were conducted to study moisture migration through hydrophobic soils and remediation mechanism of these soils to improve the moisture distribution in them.

#### 3.3.1 Measurement of water repellency

Two tests were used to classify soil according to degree of hydrophobicity

#### 3.3.1.1 MED Test (Molarity of ethanol droplet test)

Soil Name	Tests Conducted			
Devon	1. WDPT and MED test			
	<ul> <li>2. Critical moisture content determination</li> <li>3. Infiltration tests <ul> <li>Infiltration test for 1 day on hydrophobic soil when mixed with initial moisture contents of 0.5, 1, 2, 3, 5, 7.5, and 10% (w/w)</li> </ul> </li> </ul>			
	• Infiltration tests for 15 and 30 days on hydrophobic soil prepared at initial moisture content of 3, 5, and 7.5% (w/w)			
	• Infiltration test after 9 cycles of wetting and oven-drying for 1 and 3 days			
	• Infiltration test after 12 cycles of wetting and air-drying for 1 and 3 days			
	• Infiltration test for 1, 3 and 7 days on soil samples having 80-20, 70-30, 60-40, 50- 50, 40-60, 30-70, 20-80, and 10-90% (by weight) of hydrophobic-hydrophilic soil mixtures			
	• Infiltration test on hydrophilic soil for 1 day			
	• Infiltration tests for 1 and 3 days on soil conditioners amended hydrophobic soil and for 1 day on kaolinitic clay mixed with hydrophobic soil			
	4. X-ray CAT scanning for Devon hydrophobic and hydrophilic soil			
	<ol> <li>Mercury porosimeter test on Devon hydrophobic soil mixed with 5, 7.5 and 10% mois content</li> </ol>			
	6. Infiltration test on Devon soil after 5 cycles of freeze-thaw (Devon hydrophobic and wet and dry treated soils)			
	7. Wetting and drying test			
	<ul> <li>9 cycles: Each cycle consisting of wetting up to critical moisture content followed by oven-drying</li> </ul>			
	<ul> <li>12 cycles: Each cycle consisting of wetting up to critical moisture content followed by air-drying</li> </ul>			
	8. Plant growth			

## Table 3.2: Testing protocol

Stettler	1. WDPT and MED test			
	2. Critical moisture content determination			
	3. Infiltration tests			
	• Infiltration test for 1 day on hydrophobic soil prepared mixed with initial moisture content of 0.5, 1, and 2.5% (w/w)			
	• Infiltration test for 1 day after 12 cycles of wetting and air-drying (2 samples)			
	• Infiltration test for 1 day on hydrophobic-hydrophilic soil mixtures having 60-40%, 50-50%, 40-60%, 30-70%, 20-80% and 10-90% (by weight) of hydrophobic-hydrophilic soil.			
	• Infiltration test for 1 day on hydrophilic soil			
	• Infiltration tests for 1 and 3 days on soil conditioners amended hydrophobic soil and for 1 day on kaolinitic clay mixed with hydrophobic soil			
	4. Wetting and drying			
	• 12 cycles: Each cycle consisting of wetting up to critical moisture content followed by air-drying			
	5. Plant growth			
Redwater	1. WDPT and MED test			
	2. Critical moisture content determination			
	3. Wetting and drying			
	<ul> <li>5 cycles: Each cycle consisting of wetting up to critical moisture content followed by oven-drying</li> </ul>			
	• 5 cycles: Each cycle consisting of wetting up to critical moisture content followed by air-drying			
	4. Infiltration test			
	• Infiltration test for 1 day after 5 cycles of wetting and oven-drying			
	• Infiltration test for 1 day after 5 cycles of wetting and air-drying			



Fig. 3.4. Schematic of tests conducted and parameters measured

#### Introduction

The MED test was proposed by Watson and Letey (1970) and later developed by King (1981). The MED tests takes less time than WDPT test and hence is widely used. It is considered fairly reliable when used on air-dried or oven dried soil sample (King, 1981). A hydrophobic soil classification based on MED test given by King (1981) is given in Table 3.3.

In the MED test the molarity of ethanol droplet necessary for moisture infiltration in the hydrophobic soil within 10 seconds is measured. Ethanol lowers the surface tension of the liquid and enables infiltration regardless of the soil contact angles (Wallis et al., 1991). Ethanol concentrations of 0.2 M intervals in the range of 0 - 6.0 M were used to determine soil water repellency. Ethanol (95%, v/v) from Commercial Alcohols Inc., Ontario was used to prepare the solutions. The remaining 5% consisted of acetic acid, ethyl acetate, acetaldehyde, and isobutyl alcohol.

### Procedure

Aqueous solutions of ethanol with varying molarity were prepared. A drop of 100  $\mu$ L of aqueous ethanol solution of a known molarity was placed on the soil surface, using an Eppendorf Reference dropper. The time needed for the aqueous ethanol droplet to penetrate into the soil was recorded. The test was repeated on different soil samples using ethanol solutions of varying molarity. The molarity of ethanol droplet that permeated the soil in 10 seconds was regarded as its MED value. Replicate samples were tested and MED value that remained unchanged for five soil samples was reported. A maximum of 7 samples had to be tested to obtain 5 consistent MED values. This criterion was used due to soil heterogeneities at micro levels and relatively small size of soil sample used in such measurements.

### Table 3.3: Classification of soil hydrophobicity based on MED test (King, 1981)

Class	MED (M. Molar)	
Non-repellent	0	
Low-repellency	<1	
Moderate repellency	. 1-2.2	
Severe repellency	>2.2	

MED (Molarity of Ethanol Droplet) Test

#### 3.3.1.2 WDPT (Water drop penetration time) test

#### Introduction

It is a simple method for determining the degree of hydrophobicity. This test divides the soil into two broad categories; those with contact angles above 90°, i.e. non-wettable soils and those with contact angle below 90°, i.e. wettable soils. This test measures the time taken by a water drop to completely penetrate the hydrophobic soil sample. A drop will penetrate only if the contact angle is less than 90°. As most hydrophobic soils have contact angles greater than 90°, the drop of water does not penetrate immediately but takes some time which can range from few seconds to hours. The contact angle changes with time. The longer the drop stays on soil surface, the more stable and persistent the water repellency (Dekker and Jungerius, 1990).

#### Procedure

WDPT tests were performed on soil samples that were oven dried at 105°C for 24 hrs and then equilibrated at room temperature for another 24 hrs. A drop of 100  $\mu$ L of distilled water was placed on the soil surface, using an Eppendorf Reference dropper. The time needed for the water drop to penetrate into the soil was recorded. Replicate samples were tested and WDPT value that remained unchanged for five soil samples was reported. A maximum of 7 samples were tested to obtain 5 consistent WDPT values. A hydrophobic soil classification table based on WDPT test is given in Table 3.4.

Class	WDPT (s)
Wettable	< 5
Slightly wettable	5-60
Strongly non-wettable	60-600
Severely non-wettable	600-3600
Extremely non-wettable	> 3600

Table 3.4: Classification of soil hydrophobicity based on WDPT test(Dekker and Jungerius, 1990)

## 3.3.2 Critical moisture content

Moisture draws moisture due to cohesion. Presence of enough initial moisture in the soil will change the behavior of hydrophobic soil to hydrophilic. The moisture content at which the soil behavior changes from hydrophobic to hydrophilic is termed as the critical moisture content (Dekker and Ritsema, 1994).

The hydrophobic soils were oven-dried for 24 hrs and cooled in air at ambient temperature and stored in sealed plastic containers. Even though stored soils were kept in closed containers they absorbed moisture from the air in the head space. At equilibrium, the Devon, Stettler and Redwater hydrophobic soils had 1.2%, 0.2% and 0.2% moisture content respectively. Minor variation to this moisture content would occur with the varying partial pressure of water vapor in air, the room temperature, amount of head space and depth from which the soil is retrieved.

The critical moisture contents of the hydrophobic soils were determined by adding varying amount of moisture to the soil and assessing the WDPT value of the forced wetted soil. This moisture was mixed thoroughly with the soil by hand. The objective was to identify the soil moisture content that would produce WDPT value of less than 5 s. The moisture content at which water drop penetrated the soil in less than 5 s was considered as the critical moisture content of the soil.

### **3.3.3 Infiltration Test**

Infiltration tests were performed in a Plexiglas cylinder 7.5 cm in internal diameter and 14 cm in length that was filled with approximately 600 g of soil. The soil was initially oven-dried and cooled in air for 24 hrs. The soil was compacted in three layers of equal thickness using a standard Proctor hammer. Each layer was compacted with 5 blows per layer. The total height of the compacted soil samples varied between 8 to 12 cm depending on the initial moisture content and soil type. The compacted Devon soil had an average dry density between 1.25 to 1.30 g/cc whereas the compacted Stettler hydrophobic soil had a dry density of about 1.40 to 1.46 gm/cc. Hydrophobic and hydrophilic soils were compacted in separate cylinders. No moisture was added to hydrophilic soil in preparation of soil column whereas hydrophobic soil was mixed with varying amounts of initial moisture. After the soil was compacted in the Plexiglas cylinder, an annular ring of 5.2 cm internal diameter and 5 cm long was inserted to a depth of about 2 cm into the soil and the soil was ponded with water through ring (see figure 3.5). This ensured moisture movement through the bulk of the soil and minimized sidewall leakage.

About 24 ml of water, equivalent to 4% of the dry weight of the soil was added to the annular ring. This gave an approximate water head of about 2 cm. Additional water was added only when the previously added 24 ml of water migrated through the soil column. This ensured that the hydraulic stress at the top of the soil was constant and yet minimal. High hydraulic stresses would lead to hydraulic fracturing and this was avoided by



Fig. 3.5. Schematic of infiltration apparatus.

keeping a very low head. The cylinder was capped with aluminum foil to reduce moisture loss by evaporation.

After the completion of the test, the soil columns were sliced vertically. Soil samples were then taken at 1 cm depth interval and variation of moisture content with depth was determined gravimetrically.

To study the effect of higher head, some experiments were conducted using vacuum pressure. The vacuum was applied by connecting the base of the test setup to the house vacuum line. The controlled vacuum pressures were applied for 24 hrs. Varying vacuum pressures of 3, 4, 5, 7 and 10 inches of mercury or 10.16, 13.54, 16.93, 23.70 and 33.86 kpa were used.

Infiltration tests were also conducted on virgin and wet and dry treated Devon hydrophobic soil samples that were subjected to cyclic freeze and thaw. The oven-dried soil was packed into infiltration column as already described above and subjected to  $-5^{\circ}$ C for 24 hrs. The soil column was then thawed for 48 hrs at room temperature in ambient air. This process was repeated for 5 freeze-thaw cycles. After 5 freeze-thaw cycles, the soil column was subjected to water infiltration for 1 day.

### 3.3.4 Wetting and Drying

The effect of forced wetting and drying hydrophobic soil were studied. MED and WDPT tests were initially conducted on soils passing through 2 mm sieve and dried at 105°C for 24 hrs followed by equilibration for another 24 hrs in open air at room temperature. After estinated the initial MED test, some of the soil sample was subjected to forced wetting and oven drying whereas the rest of the sample was subjected to forced wetting followed by air-drying. The oven-drying was done for 24 hrs at 105°C. The air-drying was conducted for 48 hrs at room temperature. The wetting and drying cycle was repeated and MED value and moisture content noted after each cycle. The soil was forced wetted by

## 3.3.5 Dilution studies

### 3.3.5.1 Hydrophobic-hydrophilic soil mixture

Studies on moisture infiltration through mixtures of hydrophobic and hydrophilic soil, from Devon and Stettler, were conducted. Hydrophobic soil (oven-dried) was mixed with hydrophilic soil (oven-dried) in different proportions. Eight different mixtures were prepared with Devon soil and six with Stettler soil. MED values were assessed for each soil mixture. Different hydrophobic-hydrophilic soil mixtures studied are given in Table 3.5. The detail procedure of infiltration tests conducted on the soil mixtures is given in section 3.3.3.

Devon		Stett	Stettler	
Hydrophobic (%) <sup>1</sup>	Hydrophilic	Hydrophobic (%) <sup>1</sup>	Hydrophilic	
80%	20%	-		
70%	30%		-	
60%	40%	60%	40%	
50%	50%	50%	50%	
40%	60%	40%	60%	
30%	70%	30%	70%	
20%	80%	20%	80%	
10%	90%	10%	90%	

 Table 3.5: Hydrophobic-hydrophilic soil mixtures

<sup>1</sup>mass fraction based on oven-dry weight basis

#### 3.3.5.2 Soil Conditioners

Five soil conditioners namely, Green compost, Peat moss, Dolomitic Lime, Organic Compost and Sheep Manure were used to modify the hydrophobic soil properties. Soil conditioners were mixed with hydrophobic soils in six different proportions 5, 7, 10, 15, 20, 30% w/w (dry soil weight basis). All the soil conditioners contained more than 50% moisture content and were oven dried before being mixed with hydrophobic soil. Infiltration tests were conducted for 1 day on each soil mixed fraction. Three-day infiltration tests were conducted only on these soil mixed fractions that were not partially wettable after 1 day of moisture infiltration.

The hydrophobic soil was also mixed with oven dried kaolinitic clay. The mix proportions that were prepared were 90-10%, 80-20%, and 70-30% of oven-dried hydrophobic soil and oven-dried kaolinitic clay.

#### 3.3.6 Water content by gravimetric method

Moisture contents of hydrophobic soil samples taken from infiltration test and wetting and drying tests were determined gravimetically by standard ASTM test method (ASTM D-2216-92 and D-4643-93). Soil samples were weighed and put in oven for 24 hrs at a temperature of 105°C. After 24 hrs of oven drying, the samples were taken out of oven and placed in a dessicator to reach ambient temperature and dry weight was determined. The moisture content was determined as given below.

$$M.C. = (w_w - w_d) \times 100 / w_d$$
(3.1)

where

M.C = Moisture content (percentage)

 $w_w$  = Wet weight of soil sample (gm)

w<sub>d</sub> = Dry weight of soil sample (gm)
### 3.3.7 Abrasion test

Abrasion test on Stettler hydrophobic soil was conducted under two different conditions.

### **Condition 1**

One hundred grams of soil was added with 2.5% moisture (critical moisture content) in a Hamilton Beach mixer similar to the one used in a hydrometer test. The hydrophobic soil was mixed mechanically for six times intervals of 5, 10, 15, 20, and 30 minutes. The soil was then air-dried for 48 hrs. Standard MED test was conducted on each soil sample and soil moisture content was measured. Different soil samples were used for each time interval.

### **Condition 2**

In the second set of experiments, hydrophobic soil was tested in both wet and dry states. Fifty grams of oven-dried soil was used. A Fisher Thermix Stirring Hot Plate (Model 210M) and magnetic stirrers were used for soil mixing. The soil was rigorously mixed using a magnetic stirrer. The soil was stirred for varying periods of 10, 20, 30, 40, 50 and 60 minutes and the MED values were determined after each time interval.

In wet mixing, 20 ml of water was added to oven-dried soil. After mixing soil for 10, 20, 30, 40, 50 and 60 minutes, the soil slurry was filtered through Whatman glass micro fiber filter with pore opening of 0.425  $\mu$ m. The soil was then oven-dried for 24 hrs at 105°C followed by cooling at room temperature for another 24 hrs. MED tests were then conducted on each fraction.

### 3.3.8 X-ray CAT Scan

### **3.3.8.1 Introduction**

A medical X-ray scanner has been used for non-destructive measurement of porous media properties as well as for multiphase fluid flow studies. Densities of core samples

and their spatial variation can be calculated by passing x-rays through soil or rock specimens (Auzerais et al., 1990). The direct measurement of porosity in dual porosity matrix can also be measured. These porosity measurements involve the saturation of core with a dopent to increase the resolution of the image. The X-ray CAT scanning process and apparatus is shown in Fig. 3.6

### 3.3.8.2 Theory of x-ray Computer Assisted Tomography (CAT) Imaging

In CT scanning, a cross-sectional image of an object is created from x-ray attenuation measurements taken around the object from different directions. Fundamental to the CT scan is the ability of x-rays to pass through almost all the matter and the process of mathematically re-constructing the x-rays projection data into an image.

In order to determine spatial distribution of porosity, CAT data for samples with saturation of 0 and 100 % are required. This condition restricts the use of CAT scanning technique in case of hydrophobic soils because it was nearly impossible to get 100 % saturation. There was no water infiltration even after 30 days in Devon hydrophobic soil.

#### 3.3.8.3 Experimental Set up

Three Devon hydrophobic soil samples were mixed with 3, 5 and 7.5% moisture content and packed in infiltration columns in three layers. These three soil columns were subjected to x-ray CAT scan before the initiation of infiltration test. After infiltration for 30 days the soil columns were again scanned with an EMI-7007, a fourth generation scanner, used in scanning soil samples. The samples were scanned over the entire sample length, with slice thickness of 3 mm. The energy input during CAT scanning was 120 KV, with a scanning time of 3 seconds. A three-dimensional image of the moisture pattern was then constructed by using the AVS/Express computer software.

The scanning was conducted at Tomographic Imaging and Porous Media laboratory in The University of Calgary.



Fig. 3.6: X-ray CAT scan process and apparatus

A FORTRAN program was used for reading CAT scan data from x-ray scanner and Xmgr for producing histograms and PBMPLUS program for 3-D imaging (developed at ACS, University of Calgary).

### 3.3.9 Pore Size Distribution by Mercury Intrusion Porosimeter

Some of the soil samples taken along the vertical profile after the completion of infiltration test were freeze-dried. The freeze-dried specimens were weighed and then placed in the penetrometer which was then placed in a chamber in the porosimeter. The penetrometer was then filled with mercury by using a vacuum pump. The penetrometer containing soil and mercury was placed in a pressure chamber and the mercury in the penetrometer was forced into the soil pores by applying hydraulic pressure. The rise in oil pressure and volume of mercury intruded into soil pores, were recorded. A contact angle of 140 degrees and a surface tension of 480 ergs/cm<sup>2</sup> were used in calculation of pore radius. The equivalent pore radius at each applied pressure value was calculated from equation

$$r_{\rm p} = -2\sigma \cos\theta / P \tag{3.2}$$

where

$$r_p = pore radius (m)$$

- $\sigma$  = surface tension of mercury (J m<sup>-2</sup>)
- $\theta$  = Contact angle of mercury on porous solid
- P = absolute pressure applied (N m<sup>-2</sup>)

## 3.3.10 Nuclear Magnetic Resonance (NMR)

Nuclear magnetic resonance (NMR) spectroscopy was used for the determination of molecular structure. The parameters that are commonly measured in NMR spectroscopy are change in chemical shift and relaxation time (Abraham et al., 1988).

### **Principle**

NMR spectrometer consists of radiofrequency (RF) source and a magnetic field. The sample is placed in a probe and the probe is placed between the poles of the magnet. Radiofrequancy (RF) is transmitted by coil on the probe. Either RF or magnetic field is varied slowly and when the magnetic conditions are favorable for the nucleus under consideration, samples absorbed energy from the transmitted RF radiation. The resulted signals are detected, amplified and recorded.

<sup>13</sup>C-NMR studies were conducted by Ms. Marina Litvina at the Department of Chemistry, University of Calgary.

## **RESULTS AND DISCUSSION**

# 4.1 Critical Moisture Content and Infiltration through Hydrophobic soils

The moisture added to the Devon, Stettler and Redwater hydrophobic soil to achieve the critical state where their behavior changed from hydrophobic to hydrophilic were 10, 2.5 and 2% respectively. The critical moisture contents for the three hydrophobic soils were amount of moisture added to the hydrophobic soils in addition to the moisture contained in the oven-dried soil cooled in air. The critical moisture contents were 11.2%, 2.7% and 2.2% for the three soils. At moisture contents beyond these values, the soils behaved as wettable soils when subjected to infiltration tests.

The moisture content profiles of Devon and Stettler hydrophobic soils after infiltration for 1 day are shown in Figs. 4.1 & 4.2. Figure 4.1 gives the moisture profiles of Devon hydrophobic soil that were pre-mixed with 0.5, 1, 2, 3, 5, 7 and 10% water. Figure 4.2 gives moisture profiles of Stettler hydrophobic soil that was pre-mixed with 0.5, 1 and 2.5% water. Figures 4.1 & 4.2 show that Devon and Stettler soils were completely nonwettable and did not absorb any moisture. There was some moisture migration in Devon soil sample mixed with 7.5% water, but the extent of migration was limited to the top 3 cm after 1 day of water infiltration. Moisture migration through Devon and Stettler soils was possible only when Devon and Stettler soils were mixed with 10 and 2.5% moisture content respectively. This means that at critical moisture content the soil behavior changed from hydrophobic to hydrophilic. The moisture profile for Devon hydrophobic soil premixed with moisture necessary to attain critical moisture content, was similar to



Fig. 4.1: Moisture migration profile in force-wetted hydrophobic soil after 1 day of exposure to a constant, small water head for Devon hydrophobic soil. Moisture contents of 0.5, 1, 2, 3, 5, 7 and 10% (w/w) were premixed with Devon hydrophobic soil that was heated to 105°C for 24 hrs and then cooled at room conditions.



Fig. 4.2: Moisture migration profile in force-wetted hydrophobic soil after 1 day of exposure to a constant, small water head for Stettler hydrophobic soil. Moisture contents of 0.5, 1 and 2.5% (w/w) were premixed with Stettler hydrophobic soil that was heated to 105°C for 24 hrs and then cooled at room conditions.

the moisture profile of Devon hydrophilic soil (Fig. 4.1). The Stettler hydrophobic soil mixed with 2.5% moisture also showed a trend similar to the hydrophilic soil with minor variations (Fig. 4.2). The initial conditions of the hydrophobic and the hydrophilic soil samples were different. Both hydrophilic soils were air dry prior to the initiation of infiltration whereas the Devon and the Stettler hydrophobic soils were premixed with moisture.

The results of the infiltration tests on Devon hydrophobic soil that were mixed with 3, 5 and 7.5% moisture and infiltrated for 15 and 30 days are presented in Figs. 4.3 and 4.4. Figures 4.3 and 4.4 confirmed the water repellent characteristics of Devon hydrophobic soil even after 30 days of continuous water ponding at a constant head. No significant variation in moisture penetration through hydrophobic soil was observed.

Data could not be collected from vacuum pressure assisted infiltration test. Visually no moisture infiltration was observed when the applied vacuum pressure was small, i.e. less than 4 inches of mercury. When vacuum pressure was increased sidewall leakage was observed. Sidewall leakage could not be controlled even when the walls of the plexiglas cylinder were coated with grease. At low pressures no water was drawn, whereas at high pressures excessive sidewall leakage occurred. Hence, the test could not be conducted and had to be aborted.



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Fig. 4.3: Moisture migration profile in Devon hydrophobic soil containing 3, 5 and 7.5% moisture added above air-dried moisture content following 15 days of exposure to a constant, small water head.



Fig. 4.4: Moisture migration profile in Devon hydrophobic soil containing 3, 5 and 7.5% moisture added above air-dried moisture content following 30 days of exposure to a constant, small water head.

### 4.2 Three dimensional moisture profiles from CAT scan data

Three soil samples were subjected to x-ray CAT scan before and immediately after infiltration tests. Fig. 4.5 shows three-dimensional images constructed from CAT scan data.

No change in 3-D images was observed for these three soil samples, before and after infiltration tests. The soil samples that were mixed with 3, 5 and 7.5% moisture were lacking any infiltration as indicated by 3-D images. Moisture migration was indicated by changes in soil density in the 3-D images. No change in soil densities was observed in any of the soil column. Some internal re-adjustment of moisture content took place as indicated by a slight change in 3-D profile before and after test. The 3-D images were constructed at an arbitrary chosen iso-surface level of -120.

Dry hydrophilic and hydrophobic soil samples from Devon were also subjected to x-ray CAT scan. The x-ray CAT scan data on dry soil samples were used to determine porosity variations with depth and plotted as porosity versus depth (Fig. 4.6). From porosity data, it was observed that both hydrophilic and hydrophobic soils had similar porosity distribution throughout their respective soil profiles. The porosity of hydrophobic soil was only marginally lower.

## 4.3 Pore size distribution

Pore size distribution within soil column was determined for Devon soil samples that were mixed with 5, 7.5 and 10% moisture, using mercury porosimetry. In this test, pore throat radius at each cm depth of soil sample was measured. Pore throat radius was determined on soil samples taken at every centimeter from the soil column after conducting an infiltration test. The pore throat radius was plotted against depth. The result is shown in Fig. 4.7. This figure shows that pore throat radius for all soil samples were similar throughout the soil profile.



Fig. 4.5: 3-D images constructed from x-ray CAT scan data for Devon hydrophobic soil.



Fig. 4.6: Variation of porosity with depth in dry Devon hydrophobic and hydrophilic soil obtained from x-rays CAT scan.



Fig. 4.7: Variation of pore sizes with depth of soil column determined from mercury porosimeter after 1 day water infiltration under small, constant water head.

The results indicate that the pore sizes were similar in hydrophobic soil mixed with 5, 7.5 and 10% moisture content with varying moisture migration characteristics. McGhie (1980) suggested that the rate of water entry and movement of water through the pores of hydrophobic soil is always less than that for similar pore size of hydrophilic soil. It is possible that the pore throat radius measured with this test method may not be the actual pore radius because very high pressures had to be applied to force mercury into soil pores. The application of high pressures may have resulted in fracturing of soil.

## 4.4 Wetting and Drying followed by Infiltration tests

Results of cyclic wetting and drying are presented in Figs. 4.8a, 4.9a, and 4.10a. Cyclic wetting and drying progressively decreased the hydrophobicity in all three initially hydrophobic soils. The MED of the soils decreased with number of cycles. A similar trend was observed whether the drying method used was air or oven drying. Figures 4.8b, 4.9b and 4.10b give the moisture content of oven and air-dried soils before MED measurement. The moisture content measured for oven-dried soil before an MED test was found to be lower than that in air-dried soil. However, the difference between these two moisture contents was small. McGhie and Posner (1980) and Crockford et al., (1991) also reported substantial decrease in water repellency when soils were dried after wetting treatment.

It was found that MED test results were not very sensitive to the small variation in moisture content for the air-dried and oven dried soil. Similar results have been reported by King (1981). The WDPT value for hydrophobic Stettler soil after 12 cycles of wetting and air drying was 29.23 min whereas it was > 3.5 h for the non-treated Stettler soil. WDPT measurements were not conducted for Devon and Redwater soils.

Test results of moisture content variation with depth for Devon, Stettler and Redwater soils are plotted in Figs. 4.11, 4.12, 4.13. The results of 1-day infiltration tests confirmed that soil hydrophobicity decreased with increasing number of wetting and drying cycles.



Figs. 4.8 a & b: Variation in MED index and soil moisture content of Devon hydrophobic soil with wetting and drying cycles. a) MED index and b) water content of soil.



Figs. 4.9 a & b: Variation in MED index and soil moisture content of Stettler hydrophobic soil with wetting and drying cycles. a) MED index and b) water content of soil.



Figs. 4.10 a & b: Variation in MED index and soil moisture content of Redwater hydrophobic soil with wetting and drying cycles. a) MED index and b) water content of soil.

Results were similar whether drying was done at 105 °C in an oven or at room temperature in ambient air, except in the finer-textured soil. Devon hydrophobic soil was much more readily wettable after 9 cycles of wetting and oven drying than after the same number of wetting and air-drying cycles. Exposure to three more wetting and air-drying cycles caused the MED value of the soil to decrease to 3.0 and moisture migration was improved. Figure 4.14 shows a photograph of the vertical cross-section of Devon hydrophobic soil infiltrated for 1 day after 9 wetting and oven-drying cycles. An interesting observation is that after 9 cycles of wetting and oven-drying the MED value of the soil was still > 2.2 which means the soil was still classified extremely hydrophobic. However, moisture migration characteristics had improved dramatically. Doerr and Thomas (2000) also observed that soil with very high levels of hydrophobicity (WDPT > 5hrs) became saturated after 30 days.

Both Stettler and Redwater hydrophobic soils seemed to transmit water freely after being tested for 12 and 5 cycles of wetting and drying respectively as indicated in Figs. 4.12 and 4.13.

A 3-day infiltration test on Devon soil revealed that water repellency was not completely overcome after being subjected to 12 cycles of wetting and air-drying (Fig. 4.15) even though the moisture movement characteristics had improved. The results were similar to 1 day infiltration.

Differences in pore size distribution, clay content and soil water repellency may explain the different wetting behavior of Devon, Stettler and Redwater hydrophobic soil. Soil water repellency, as assessed by the MED test, was considerably more severe in Devon soil even after 12 wetting and air-drying cycles than it was in Redwater soil after 5 wetting and drying cycles. Ma'Shum and Farmer (1985) reported that moist soil, when air-dried, was always less water-repellent than moist soil dried in an oven. Similar observations were made only for Redwater hydrophobic soil but the opposite was observed in Devon soil.

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Fig. 4.11: Moisture migration profile of wet and dry treated Devon hydrophobic soil after 1 day of exposure to a constant, small water head.



Fig. 4.12: Moisture migration profile of wet and dry treated Stettler hydrophobic soil after 1 day of exposure to a constant, small water head.



Fig. 4.13: Moisture migration profile of wet and dry treated Redwater hydrophobic soil after 1 day of exposure to a constant, small water head.



Fig. 4.14: Vertical cross section of Devon hydrophobic soil after 9 cycles of wetting and oven-drying and 1 day of water infiltration.



Fig. 4.15: Moisture migration profile of wet and dry treated Devon soil after 3 days of exposure to a constant, small water head.

Figures 4.11, 4.12 4.13, and 4.15 demonstrate that treating soil to cyclic wetting and drying may be an effective way to reduce soil water repellency and to improve soil moisture migration characteristics. In addition, these data indicated that even a soil classified as severely water-repellent according to a standardized scale presented in Table 3.3 can infiltrate water undermining the classification of soil hydrophobicity based on MED or WDPT tests. MED and infiltration test could not be correlated because different moisture penetration time was considered in each test.

### 4.4.1 Mechanism of decrease in soil water repellency

It may be hypothesized that any one or more of four mechanisms may be responsible for the observed decrease in soil water repellency with an increase in number of wetting and drying cycles. These are:

- Chemical change of hydrophobic agents during drying
- Abrasion of hydrophobic agents during wetting
- Production of hydrophilic organic compounds during wetting and drying
- Change in the conformation of hydrophobic agents caused by wetting and drying

Partial degradation of hydrophobic agents during drying may have contributed to the reduction in soil water repellency. However, it probably plays an insignificant role in the resilient hydrophobic soils under study. Studying similar soils, Roy and McGill (1998) reported that the MED index of three hydrophobic soils was slightly reduced by oven drying at 105° C for 14 days. They found that a higher temperature range or longer exposure time was expected to reduce soil hydrophobicity. They also found that hydrophobicity was completely eliminated when soil was heated to 200° C for 24 hrs.

Abrasion could also have contributed by reducing the density of the hydrophobic coating (King, 1981). To check the effect of forced wetting on soil particle abrasion, particle size analysis was conducted on a soil sample after subjecting it to wetting and drying cycles.

The results of sieve analysis indicated that significant aggregate dispersion did not occur. Minor amounts of soil particle abrasion did occur during wetting and drying. However, a conclusion about the hydrophobic skin removal could not be drawn. It was also found that vigorously stirring of the hydrophobic soil on a Fisher plate or in a Hamilton Beach mixer for up to 60 minutes did not reduce water repellency in the wet or dry state because a thorough coating of soil particle with moisture was necessary to observe decrease in soil hydrophobicity. The MED index of hydrophobic Stettler soil was unchanged by vigorous stirring of 30 min at critical moisture content in a Hamilton Beach mixer (Table 4.1).

	Time Inte	rval (minutes)		ME	Moisture Content	
Fisher Plate		Hamilton Beach	Fisher Plate		Hamilton Beach	(%)
Dry	Wet	Wet	Dry	Wet	Wet	Hamilton Beach
10	10	5	5.2	5.2	5.2	0.06
20	20	10	5.2	5.2	5.2	0.06
30	30	15	5.2	5.2	5.2	0.11
40	40	20	5.2	5.2	5.2	0.10
50	50	25	5.2	5.2	5.2	0.13
60	60	30	5.2	5.2	5.2	0.06

Table 4.1: Effect of soil abrasion on MED value for Stettler hydrophobic soil.

Wetting and drying might have caused production of hydrophilic organic compounds in soil and thereby reduced soil water repellency. Partial degradation of organic compounds during wetting and drying, production of microbial metabolites during wetting, and lysis of fast-growing microbial cells during drying could all have added hydrophilic organic compounds on the outer surfaces of soil particles. Roy and McGill (1998) noted that viable non-spore forming bacterial cells were present in a hydrophobic soil similar to the ones used in this study. Partial break down of resistant microbial spores and cysts may have been contributed to the observed decrease in soil hydrophobicity.

The result of <sup>13</sup>C solid state NMR indicated a shift in spectra from Devon hydrophobic towards Devon hydrophilic when wet and dry treated Devon hydrophobic soil was subjected to <sup>13</sup>C NMR spectroscopy. Wet and dry treated soil was subjected to 17 cycles of wetting and air-drying. A relative increase in carbohydrates group and decrease in aromatic compounds was observed in NMR spectrum for force wetted and air-dried hydrophobic soil. It is known that relative abundance of carbohydrates and carboxylic acid group in soil increase soil hydrophilicity whereas aliphatic and aromatic compounds increases soil hydrophilicity. The <sup>13</sup>C NMR spectra for Devon hydrophobic, hydrophilic and wetting and air-dried soils are given in Figures 4.16 a, b and c respectively.

Ma'Shum and Farmer (1985) demonstrated that wetting and drying could induce conformational changes in hydrophobic agents, which in turn may be responsible for changes in soil water repellency. Conformational changes usually occur when the soil is wet. During wetting hydrophilic grouping like hydroxyl, carboxylic acid and amide interact with water molecules, but tend to interact with each other when soil is dry. Organic matter deposit on soil surface when soil is dry and increases soil hydrophobicity. It is possible that wetting may have contributed more to reducing soil water repellency than drying contributed to increase it with increasing number of wetting and drying cycles.

# 4.5 Infiltration test on hydrophobic soil subjected to freezethaw cycles

The results of infiltration test on virgin Devon hydrophobic soil and Devon hydrophobic soil treated with 17 wetting and oven-drying cycles and then subjected to 5 freeze-thaw





Fig. 4.16: <sup>13</sup>C NMR spectra for Devon hydrophobic, hydrophilic and force wetted and air-dried soil.

cycles is shown in Fig. 4.17. No moisture was added during the preparation of the soil column, during freeze-thaw, and before the infiltration test. The infiltration test was conducted for 1 day.

From Figure 4.17, it is seen that cyclic freeze-thaw did introduce change in hydrophobic soil behavior. Devon hydrophobic soil (treated soil that had been subjected to wetting and oven-drying with MED = 1.8) behaved like a wettable soil whereas virgin Devon hydrophobic soil remained non-wettable after 5 cycles of freeze and thaw. It is concluded from Fig. 4.17 that cyclic freeze and thaw did not alter moisture migration characteristics of hydrophobic soil. Cyclic freeze and thaw, a characteristic of winter condition in Alberta, did not have adverse effect on moisture movement through wet and dry treated hydrophobic soil.

The water infiltration in the top 2 cm of the soil column in Devon hydrophobic soil might be due to the micro cracks or structural disturbances resulted from cyclic freeze-thawing. The swelling and shrinkage of hydrophobic soils cause structural breakdown and loss of soil hydrophobicity (Fink and Mitchell, 1973). However, soil swelling and shrinkage cracks were hard to observe in the compacted soil column.

## 4.6 Infiltration test on mixed soil

### 4.6.1 Mixing with hydrophilic soil

MED classification of hydrophobic-hydrophilic soil is given in Table 4.2. The results of the water infiltration tests are shown Figs. 4.18, 4.19, 4.20, and 4.21. These figures indicate that soils that were classified as water repellent by MED test were actually wettable to varying extents after being mixed with hydrophilic soil. Soil fractions that were slightly wettable after 1 day of water infiltration became fully wettable after 3 days of water infiltration (Fig. 4.20). The fraction 10-90% (10% Devon hydrophilic and 90%



Fig. 4.17: Moisture migration profile of Devon hydrophobic and treated soil after 1 day of exposure to a constant, small water head following 5 cycles of freeze-thaw.

Devon hydrophobic) remained non-wettable after 1 day of infiltration (data could not be collected as dry sample crumbled) but started to transmit moisture after 7 days (Fig. 4.21) even though it had an MED value of 4.8. The increase in wettability of the mixed soil may be due to discontinuities in hydrophobic material in mixed soils. When hydrophobic soil was mixed in hydrophilic soil, the hydrophobic coating became discontinuous. The wettable portion of mixed soil absorbed water and slowly improved wettability of the entire soil. Discontinuities in hydrophobic coatings have been known to allow water to penetrate in a severely water-repellent soil (De Bano, 1981). The test results suggest that soil that is non-wettable can be improved by adding wettable soils.

Devon		Stettler		MED value (M)		Soil classification <sup>2</sup>	
Hydro- philic (%) <sup>1</sup>	Hydro- phobic	Hydro- philic (%) <sup>1</sup>	Hydro- phobic	Devon	Stettler	Devon	Stettler
80%	20%			1.4		Moderate repellency	
70%	30%		•	1.8		Moderate repellency	
60%	40%	60%	40%	2.0	1.8	Moderate repellency	Moderate repellency
50%	50%	50%	50%	3.2	2.6	Severe repellency	Severe repellency
40%	60%	40%	60%	4.0	3.2	Severe repellency	Severe repellency
30%	70%	30%	70%	4.4	3.4	Severe repellency	Severe repellency
20%	80%	20%	80%	4.6	4.0	Severe repellency	Severe repellency
10%	90%	10%	90%	4.8	4.6	Severe repellency	Severe repellency

Table 4.2: Hydrophobic-hydrophilic soil mixtures classification

<sup>1</sup>mass fraction based on oven-dry weight basis

<sup>2</sup>classification as per King (1981)



Fig. 4.18: Moisture migration profile of different mixes of Devon hydrophobichydrophilic soil after 1 day of exposure to a constant, small water head.



Fig. 4.19: Moisture migration profile of different mixes of Stettler hydrophobichydrophilic soil after 1 day of exposure to a constant, small water head.



Fig. 4.20: Moisture migration profile of different mixes of Devon hydrophobichydrophilic soil after 3 days of exposure to a constant, small water head.


Fig. 4.21: Moisture migration profile of Devon hydrophobic-hydrophilic soil mix after 7 days of exposure to a constant, small water head.

# 4.6.2 Mixing hydrophobic soil with soil conditioners

The bulk density variation in hydrophobic soil mixed with different soil conditioners are given in Table 4.3.

Soil Conditioner	Soil Type	Bulk Density (gm/cc) Proportion of soil conditioners mixed (w/w)					
		Green Compost	Devon	1.234	1.180	1.137	0.987
Stettler	1.358		1.293	1.180	1.131	1.006	0.754
Peat Moss	Devon	1.022	0.999	0.891	0.822	0.754	0.670
	Stettler	1.113	1.071	0.951	0.775	0.659	0.637
Dolomitic Lime	Devon	1.293	1.358	1.381	1.429	1.476	1.491
	Stettler	1.437	1.579	1.592	1.599	1.603	1.621
Organic Compost	Devon	1.212	1.156	1.132	1.045	0.977	0.823
	Stettler	1.326	1.321	1.271	1.088	1.044	0.767
Sheep Manure	Devon	1.325	1.312	1.275	1.234	1.207	1.200
	Stettler	1.397	1.397	1.380	1.374	1.332	1.257

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Table 4.3: Bulk density variation of hydrophobic soil amended with conditioners.

<sup>1</sup> 5% soil conditioner mixed with hydrophobic soil (w/w, on dry soil weight basis)

#### 4.6.2.1 Green Compost

Moisture migration profiles for Devon hydrophobic soil amended with Green Compost for 1 day and 3 days and for Stettler hydrophobic soil for 1 day are given in Figs. 4.22, 4.23 and 4.24 respectively.

Devon hydrophobic soil conducted moisture when mixed with 10% (w/w, oven dry weight basis) Green Compost. In fact, it imbibed moisture even when it was amended with 5% Green Compost and exposed to moisture for 3 days. It became fully wettable when mixed with 15% or more Green Compost (by weight of dry soil). The increase in water permeation with an increasing quantity of Green Compost was due to the high water retention capacity of compost material and decrease in soil bulk density leading to increase in soil porosity. The compost material, made of tree barks, absorbed moisture rapidly and then slowly transmited water to the surrounding hydrophobic soil particles. This process increased the overall wettability of the Devon hydrophobic soil. The Devon hydrophobic soil became fully wet (moisture profile became vertical) when the soil had 20% or greater Green Compost and exposed to moisture for 1 day or 15% Green Compost and exposed to moisture for 3 days.

Figure 4.23 indicates that the soil fractions (5, 7 and 10% of Green Compost) that were slightly wettable after 1 day of moisture infiltration became almost fully wettable after 3 days of moisture infiltration. It means wetting characteristics of hydrophobic soils can be improved if soil is exposed to moisture for longer period of time.

Stettler hydrophobic soil conducted moisture when it was mixed with 5% green compost. The Stettler hydrophobic soil had 5% moisture content (gravimetric) even at a depth of 10 cm (bottom of soil column) when mixed with 7% green compost. With increasing green compost, infiltration rate for Stettler hydrophobic soil increased significantly (Fig. 4.24). Moisture migration became steady when Stettler hydrophobic soil was mixed with 15% of Green Compost. These results show that lesser quantities of Green Compost can change a coarse (Stettler) hydrophobic soil to hydrophilic than fine-grained Devon



Fig. 4.22: Moisture migration profile of Devon hydrophobic soil mixed with Green Compost and exposed to 1 day of a constant, small water head.



Fig. 4.23. Moisture migration profile of Devon hydrophobic soil mixed with Green Compost and exposed to 3 days of a constant, small water head.



Fig. 4.24: Moisture migration profile of Stettler hydrophobic soil mixed with Green Compost and exposed to 1 day of a constant, small water head.

hydrophobic soil. Even though every attempt was made to ensure that the mixture of Green Compost and hydrophobic soil is uniform, it is believed that high moisture content in some parts of the moisture curves may be due to the non-uniform distribution of compost at that depth.

## 4.6.2.2 Peat Moss

The hydric response of Peat Moss mixed Devon and Stettler hydrophobic soils for 1 day and 3 days are shown in Figs. 4.25, 4.26, 4.27 and 4.28 respectively.

The results in Figs. 4.25 and 4.27 indicate that Peat Moss is not as effective as Green Compost in increasing or improving water intake behavior of both hydrophobic soils. The moisture infiltration was observed only in top 4-5 cm of soil column. This indicates that infiltration rates were very slow. Moisture infiltration was observed in almost all mixes.

Figure 4.26 indicates that moisture intake behavior of Peat Moss mixed Devon hydrophobic soil did not improve significantly when subjected to continuous water infiltration for 3 days. Moisture seemed to be migrating down albeit slowly. Same observations were made for Stettler soil (Fig. 4.28) that subjected to infiltration test for 3 days.

Peat Moss, a highly fibrous material, is known for its high water retention capacity. Its inability to intake or absorb water here may result from oven drying. Valet et al., (1991) founded that the use of Peat Moss after oven drying decrease the water infiltration and Peat Moss itself acts as a hydrophobic material when dry. Masking effect or discontinuation in hydrophobic coatings cannot be achieved with Peat Moss because of its fibrous nature. These two processes, masking and discontinuity in hydrophobic material, are very important in improving moisture characteristics of hydrophobic soils.



Fig. 4.25: Moisture migration profile of Devon hydrophobic soil mixed with Peat Moss and exposed to 1 day of a constant, small water head.



Fig. 4.26: Moisture migration profile of Devon hydrophobic soil mixed with Peat Moss and exposed to 3 days of a constant, small water head.



Fig. 4.27: Moisture migration profile of Stettler hydrophobic soil mixed with Peat Moss and exposed to 1 day of a constant, small water head.



Fig. 4.28: Moisture migration profile of Stettler hydrophobic soil mixed with Peat Moss and exposed to 3 days of a constant, small water head.

### 4.6.2.3 Dolomitic Lime

A small amount of moisture intake was observed after 1 day when Devon soil was mixed with less than 20% of Dolomitic Lime (Fig. 4.29). Significant moisture intake response was not evident in Devon hydrophobic soil until it was mixed with 30% lime (w/w). When the lime content was less than 30%, the moisture intake was only in the top few centimeter of the soil sample.

The results of infiltration test for Devon soil mixed with Dolomitic Lime after 3 days of moisture infiltration are given Figure 4.30. The wetting characteristics of lime amended Devon hydrophobic soil improved significantly. The hydrophobic soil fractions that were almost nonwettable or partially wettable after 1 day of infiltration became fully wettable after 3 days of water infiltration.

Lime addition worked very well for Stettler hydrophobic soil. Steady infiltration was observed after 1 day (Fig. 4.31) when Stettler hydrophobic soil was mixed with 15, 20, and 30% (w/w) of Lime. Significant moisture migration in the top 4 cm of the soil sample was also observed when Stettler hydrophobic soil mixed with 5 and 7% of lime. Since this test was conducted only for 1 day, it is possible that the wetting front did not have time to reach the base of the sample. Moisture migration was observed to greater depth in Stettler hydrophobic soil mixed with 5 and 7 and 10% lime and subjected to infiltration test for 3 days (Fig. 4.32).

It is generally believed that fine-grained soil conditioners work well when mixed with coarse-grained soil (www.whitneyfarm.com). The ability of lime to increase the wettability of hydrophobic soil was attributed to the dispersion of hydrophobic organic coatings due to increased solubility of hydrophobic compounds. The addition of lime to hydrophobic soil also causes the dissolution of hydrophobic compounds as a result of an increase in soil pH (Muneer and Oades, 1989). It is known that both the fulvic and humic acid decreases the surface tension of water and the solid-liquid contact angle with increasing solubility in water. The solubility of humic acid increases with increasing pH.



Fig. 4.29: Moisture migration profile of Devon hydrophobic soil mixed with Dolomitic Lime and exposed to 1 day of a constant, small water head.



Fig. 4.30: Moisture migration profile of Devon hydrophobic soil mixed with Dolomitic Lime and exposed to 3 days of a constant, small water head.



Fig. 4.31: Moisture migration profile of Stettler hydrophobic soil mixed with Dolomitic Lime and exposed to 1 day of a constant, small water head.



Fig. 4.32: Moisture migration profile of Stettler hydrophobic soil mixed with Dolomitic Lime and exposed to 3 days of a constant, small water head.

Addition of lime into the soil increases in pH increases the ability of fulvic and humic acid to increase water infiltration (Chan and Schnitzer, 1978). Dissolution causes the hydrophobic coatings on soil particle to become discontinuous, and increase soil wettability.

### 4.6.2.4 Organic Compost

Moisture migration profiles for Devon and Stettler hydrophobic soil amended with Organic Compost are shown in Figs 4.33 and 4.34 respectively.

Figure 4.33 indicates that Devon hydrophobic soil started conducting moisture when mixed with 7% of Organic Compost. However, the extent of moisture migration was limited to the top 4 cm of the soil column in 1 day. Wettability of Devon hydrophobic soil increased with an increase in the amount of Organic Compost. The Devon hydrophobic soil became fully wettable when Devon soil had 15% or more of Organic Compost content. Moisture content of about 10% was found after 1 day of infiltration at a depth of 9 cm when Devon hydrophobic soil was mixed with 15% of Organic Compost content.

The Organic Compost mixed Stettler hydrophobic soil showed similar behavior (Fig. 4.34). The moisture profiles for Stettler hydrophobic soil was similar when mixed with 5 and 7% of Organic Compost whereas significant increase in water infiltration was observed after 1 day of infiltration when Stettler hydrophobic soil had 10% or more Organic Compost content. Moisture content of about 15% was present at a depth of 10 centimeter when Stettler soil was mixed with 15% of organic compost.

The change in behavior of Devon and Stettler hydrophobic soil to hydrophilic soil when mixed with Organic Compost was due to the high water holding capacity of Organic Compost and was also indicated by the swelling of mixed soil. The Organic Compost material swells as it absorbs water and then transmits water to the surrounding



Fig. 4.33: Moisture migration profile of Devon hydrophobic soil mixed with Organic Compost and exposed to 1 day of a constant, small water head.



Fig. 4.34: Moisture migration profile of Stettler hydrophobic soil mixed with Organic Compost and exposed to 1 day of a constant, small water head.

hydrophobic soil particles. It is also possible that Organic Compost might have acted as a masking material. In masking, particles cover hydrophobic soil surfaces, cause a discontinuation in hydrophobic coatings and improve water infiltration slowly (Deller et al., 1994). The behavior of hydrophobic soil mixed with Green Compost and Organic Compost are similar.

#### 4.6.2.5 Sheep Manure

Moisture profiles for Sheep Manure mixed Devon soil for 1 day and 3 days and for Stettler hydrophobic soil for 1 day are shown in Figs. 4.35, 4.36 and 4.37 respectively.

When exposed to moisture for one day Sheep Manure did not improve hydrophobic behavior of Devon soil until hydrophobic soil was mixed with 20% or more Sheep Manure (w/w). It might be possible that the ability of Sheep Manure to absorb and transmit water was retarded due to its drying. The moisture movement was retarded to the extent that moisture migration could not be observed after 1 day of infiltration. It might be possible that Sheep Manure mixed soil conducted water if the infiltration test was extended for longer period of time (Valet et al., 1991). With increased exposure time, Sheep Manure itself and the development of biological activity with time produces hydrophilic substances that enhance moisture migration.

When less than 10% Sheep Manure was used, moisture intake behavior of Sheep Manure mixed Devon hydrophobic soil improved significantly when the infiltration test was conducted for 3 days (Fig. 4.36). The hydrophobic soil fraction mixed with 5% (w/w) of Sheep Manure showed moisture content of about 5% at depth of 7 cm from top of soil column. These results agree with observations made by Valet et al., (1991). Figure 4.36 indicates that prolonged exposure to moisture increases the moisture intake capacity of Sheep Manure amended hydrophobic soils.



Fig. 4.35: Moisture migration profile of Devon hydrophobic soil mixed with Sheep Manure and exposed to 1 day of a constant, small water head.



Fig. 4.36: Moisture migration profile of Devon hydrophobic soil mixed with Sheep Manure and exposed to 3 days of a constant, small water head.



Fig. 4.37: Moisture migration profile of Stettler hydrophobic soil mixed with Sheep Manure and exposed to 1 day of a constant, small water head.

Moisture migration profiles for mixed Stettler hydrophobic soil amended with Sheep Manure were similar to the standard moisture profiles of partially wetted soils (Hillel, 1980). It means that first 2-4 cm of mixed soil were fully wettable followed by redistribution zone (4-7 cm). The wetting front and dry soil zone extended approximately from 7-9 cm. It was found that moisture content was about 45% in first couple of centimeter and was about 0-2% at a depth of about 10 cm for Stettler hydrophobic soil mixed with 30% Sheep Manure. This indicates that water infiltration and distribution within Sheep Manure (hydrophilic) and from manure to hydrophobic soil was very slow. Moisture migration could have been improved in Sheep Manure mixed soil if the infiltration test had been conducted for longer period of time.

This series of tests with conditioners indicate that some conditioners are better than others whereas, most conditioners improve the moisture distribution and infiltration potential of these extremely hydrophobic soils. The quantity of Green Compost and Organic Compost required to effectively make the wetting front uniform is significantly lower than that of other conditioners. Their difference in affecting moisture migration though hydrophobic soils is due to the difference in physical and chemical characteristics of the conditioners themselves. The fibrous nature of Peat Moss prevents it from providing as good masking effect as others. Lime, on the other hand, increases the pH, which in turns causes, the dissolution of fulvic and humic acids leading to a decrease in the surface tension of the permeating water. These test results further show that the effect of these conditioners on sandy soil is higher than that on clayey soil. Soil hydrophobicity is a property of the chemistry of surface of soil particles. Hence the higher the surface area, the higher the moisture required to interact with the surfaces, the more the amount of masking agent required.

## 4.6.3 Kaolonitic clay

Clay improved the water infiltration through hydrophobic soils. Infiltration was slow in Devon hydrophobic soil and moisture did not reach to the bottom of the soil column (Fig.

4.38). The Devon hydrophobic soil mixed with 20 and 30% clay showed reduced water infiltration rate. The higher the clay content, the lower the hydraulic conductivity as pore size decreases. Rapid and complete water infiltration was observed in hydrophobic soil that was mixed with 10, 20 and 30% clay (w/w) in Stettler hydrophobic soil (Fig. 4.39). A higher rate of water infiltration was observed in coarse-grained soil when mixed with a smaller amount of clay. Increased amount of clay leads to a soil matrix with lower permeability decreasing the rate of moisture movement.

The efficiency of clay in improving water infiltration through hydrophobic soil depends on the dispersitivity of clay and particle shape (Ma'Shum et al., 1989). The wetting front was uniform in clay amended hydrophobic soils. Clay particles being very fine get into the pores between the soil particles. The moisture then transmits between one clay particle to another, improving the moisture movement through the soil mass. This does not mean that the properties of the hydrophobic soil particles have changed. If the clay particles are not mixed thoroughly or if the amount of clay is so small that they do not form a continuous pathway for moisture migration, then the soil may still behave hydrophobically and hinder moisture movement.

## 4.7 Plant growth

Plants were grown on treated hydrophobic soils to see whether plant life can be sustained on these treated soils. It is our belief that if we can sustain plant growth then we should be able to get rid of the barren patches formed due to hydrophobicity. We may not get rid of the hydrophobic soil particles but at least the impact of hydrophobicity will be camouflaged and not visible. Plant growth study was designed to show that treated hydrophobic soils (wet and dry) and kaolinitic clay amended soil can sustain plant growth if these soils start conducting moisture. However, this study was not statistically designed experiment and was not intended to give any information other than an indication that plant growth is feasible in these treated soils.

The following soils from Devon and Stettler were studied for plant growth.



Fig. 4.38. Moisture migration profile of Devon hydrophobic soil mixed with kaolinitic clay and exposed to 1 day of a constant, small water head.



Fig. 4.39. Moisture migration profile of Stettler hydrophobic soil mixed with kaolinitic clay and exposed to 1 day of a constant, small water head.

- 10% kaolinitic clay mixed hydrophobic soil
- hydrophilic soil from areas adjacent to hydrophobic soil (control)
- treated soil (force wetted and air-dried)
- hydrophilic soil from Calgary area (control)

Hydrophobic soils mixed with soil conditioners were not chosen for plant growth because the soil conditioners used in this study for diluting hydrophobic soils may supply the nutrients that contribute to the plant growth itself.

Seeds (peas) were sown in all seven pots in laboratory as shown in Figs. 4.40 and 4.41. The soils were kept moist by watering soil every day. Single seed was sown in each pot at a depth of approximately 2 cm from the soil crust level. The germination of pea plants from seeds was observed after a week in all seven pots. The average heights of the plants were about 8 cm after 3 weeks. Significant differences were not observed in plant germination time and height in control soils and in the different treated hydrophobic soils from Devon and Stettler.

This findings indicates that moisture migration and plant growth in hydrophobic soils can be improved by adding kaolinitic clay and by subjecting hydrophobic soil through a number of wetting and air-drying cycles. The amount of kaolinitic clay added and number of wetting and air-drying cycles depend on the soil type and magnitude of soil hydrophobicity.



Fig. 4.40: Plants (*Lincoln Homesteader*) growth from seeds after 3 weeks in Devon hydrophobic soil. 1. 10% kaolinitic mixed with Devon hydrophobic soil, 2. hydrophilic soil from Devon, 3. treated soil (17 cycles of wetting and air-dried), 7. hydrophilic soil from Calgary area.



Fig. 4.41: Peas plant growth from seeds after 3 weeks in Stettler hydrophobic soil. 4. 10% kaolinitic mixed with Stettler hydrophobic soil, 5. hydrophilic soil from Stettler, 6. treated soil (11 cycles of wetting and air-drying), 7. hydrophilic soil from Calgary area.

# Chapter 5

# **CONCLUSIONS AND RECOMMENDATIONS**

# 5.1 Conclusions

### 5.1.1 Moisture movement through hydrophobic soils

The results of laboratory investigations on moisture movement through hydrophobic soils indicate that hydrophobic soil did not conduct any moisture when its moisture content is below a critical value. The critical moisture content was found to be higher for fine textured soil, i.e. 11.2% and lower for coarse- grained soil, i.e. 2.7 and 2.2% for Stettler and Redwater hydrophobic soils. At or above critical moisture content the soil behavior changed from hydrophobic to hydrophilic.

## 5.1.2 Wetting and drying

The degree of hydrophobicity decreased when the soil was subjected to a number of wetting and drying cycles. The rate of decrease in soil hydrophobicity was higher during initial wetting and drying cycles and the rate became less as the number of wetting and drying cycles increased. The moisture movement pattern of the hydrophobic soil changed after a few cycles of wetting and drying and became more uniform.

The number of wetting and drying cycles necessary to improve the wettability characteristics depended on soil type and on the initial degree of hydrophobicity. The decrease in soil hydrophobicity during wetting and drying cycles may have been caused

by the partial oxidation of organic compounds and production of hydrophilic organic compounds as revealed by solid-state <sup>13</sup>C NMR spectroscopy and conformational changes during wetting and drying.

### 5.1.3 Dilution effect on moisture migration

### 5.1.3.1 Mixing hydrophobic soil with hydrophilic soil

Mixing of hydrophilic soil with hydrophobic soil improved soil behavior and increased water infiltration through hydrophobic soil. Moisture movement became more uniform as the hydrophilic soil content was increased. Even though the classification of soil mixtures remained water repellent the moisture migration capability of the mixed soil improved significantly.

#### 5.1.3.2 Soil conditioners

Amending hydrophobic soil with conditioners increased the moisture infiltration capacity of hydrophobic soil. The amount of soil conditioners required to improve the water intake property of soil depended on soil type and the moisture retaining capacity of the soil conditioners. All the soil conditioners except Peat Moss increased wettability of hydrophobic soil after 1 day of moisture infiltration. 30% of soil conditioners when mixed with hydrophobic soil (by weight of dry soil) changed the hydrophobic soil into hydrophilic soil. Prolonged exposure to moisture, i.e. moisture infiltration for 3 days, further improved the wettability of hydrophobic soils.

#### 5.1.3.3 Kaolinitic clay

Hydrophobic soil behaved like hydrophilic soil when amended with 10% kaolinitic clay (by dry weight of soil). Water infiltration was more significant in hydrophobic soil having 10% clay than those having 20 or 30% clay. This was due to a decrease in permeability as the clay content increased.

## 5.1.4 Plant growth

Treated and diluted hydrophobic soils retained moisture and sustained plant growth. No significant difference in germination time and plant height was observed in treated or diluted hydrophobic soils as compared to the control hydrophilic soil.

# 5.2 Recommendations for future work

From this research work, the following recommendations are made for future work.

- The test results indicated that even though the soil remains repellent after treating it to wetting and drying it still transmits moisture. Hence, further research is needed to clearly define the definition of hydrophobicity in terms of soil wettability or water infiltration with time. It is recommended that research should be conducted on the test methods used to determine degree of soil hydrophobicity.
- A detailed chemical study is required to determine the exact mechanism of decrease in soil hydrophobicity during successive wetting and drying cycles.
- Field studies with hydrophobic soil mixed with different soil conditioners should be conducted.
- Field studies on hydrophobic soil treated with wetting and air-drying should be conducted.

- The theoretical principles governing water movement through hydrophobic soils are not well developed or understood. In fact, unsaturated flow theory is being applied to understand flow through hydrophobic soils. There is a need to check the applicability of existing unsaturated flow principles to water infiltration through hydrophobic soils.
- It is further recommended to develop relationships between the degree of soil hydrophobicity, soil particle size and quantities of soil conditioners needed to increase water infiltration in hydrophobic soils.

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