ACCELARATION OF FREEZE - THAW TESTS BY MICROWAVE HEATING

by

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I thank everybody who encouraged me throughout this study.

ABSTRACT

The strength characteristics and hydraulic conductivity of clay liners used in waste landfills are affected by freezing and thawing cycles. To evaluate the performance of clay liners, freezing and thawing tests are needed. These tests take a long time anywhere from twenty days to twenty weeks. Microwave thawing is a new technique that can be useful to cut the testing time by half which will make these tests more practical.

There are several factors that affect the strength of compacted clay among which the most important ones are method and effort of compaction, duration of one freeze and thaw cycle, number of the freeze and thaw cycles, the freezing and thawing temperatures, and when applicable the type and amount of soil additive used.

In the tests, hourly and daily freezing time intervals were applied. After each freezing cycle, samples were thawed in the microwave oven at different power levels with approximately seven minutes duration. The number of cycles were five and ten. Also there was a control group with no freeze and thaw cycle.

The tests were conducted on pure kaolinite, 10 per cent by weight polymer added kaolinite, pure bentonite and 40 per cent bentonite added kaolinite samples. The polymer used was waste tire fibers obtained from tire retread industry as a byproduct.

The unconfined compression and direct shear tests were conducted on the compacted kaolinite and rubber added kaolinite samples before and after freeze and thaw cycles to determine the shear strength. The test results obtained by microwave oven thawing were compared with the ones obtained by conventional oven thawing. Water content determination test results have shown that microwave oven is a practical way for rapidly obtaining moisture content of soils for the specified conditions. Strength test results revealed that shear strength of the samples subjected to microwave thawing in freeze and thaw cycles was slightly higher than the shear strength of the samples subjected to conventional oven thawing, whereas the cohesion was smaller in the first case with respect to the other.

ÖZET

Geçirimsiz toprak perdelerin kesme mukavemeti ve geçirgenliği donma ve çözülme devirlerinden etkilenir. Erime ve donma devirlerinin geçirimsiz toprak perdeleri üzerindeki etkisini araştırmak için birçok deney gereklidir. Bu deneyler yirmi günden yirmi haftaya kadar zaman alabilir. Mikrodalga çözülme yöntemi ile bu süreci yarıya indirmek mümkün olabilir.

Kil zeminin kesme mukavemetini etkileyen birçok faktör bulunmaktadır. Bunlar arasında en önemlileri; kil zemine karıştırılan katkı maddesi, sıkıştırma metodu ve enerjisi, donma ve erime devirlerinin süresi, donma ve erime devirlerinin tekrar sayısı, ve donma ve erime sıcaklıkları olarak sıralanabilir.

Yapılan deneylerde donma ve çözülme devir tekrar sayısı beş ve on olarak uygulanmıştır. Donma ve çözülme devir süreleri saat ve gün olarak seçilmiştir. Her donma devrinden sonra yaklaşık yedi dakikalık mikrodalga fırının farklı enerji seviyelerinde çözülme devri uygulanmıştır. Her grup test için bir de normal çözülme yöntemi uygulanmış kontrol grubu bulunmaktadır.

Deneyler saf kil, yüzde on polimer katılmış kil, saf bentonit, ve yüzde kırk kil katılmış bentonit numuneleri üzerinde yapılmıştır. Kullanılan polimer, izopren ve karbon karası karışımından üretilen araba lastiğinin sırt kısmından oluşmuş ve rendelenmiş halde sağlanmıştır.

Donma ve çözülme devirleri öncesinde ve sonrasında kil ve polimer katılmış kil numunelerinin mukavemetini belirlemek amacıyla kesme ve basınç deneyleri yapılmıştır. Su muhteviyatı belirleme deneyleri kaolin, bentonit, ve yüzde kırk bentonit katılmış kaolin üzerinde uygulanmıştır. Su muhteviyatı belirleme deneyleri, mikrodalganın su muhteviyatını çabuk olarak belirlemek amacıyla belirtilen şartlarda pratik bir yöntem olduğunu göstermiştir. Mukavemet deneyleri mikrodalga çözülme yöntemi uygulanmış numunelerin, normal çözülme yöntemi uygulanan numunelere göre daha yüksek kesme mukavemetine sahip olduklarını göstermiştir. Kohezyon değerleri ise mikrodalga çözülme yöntemi uygulanmış numunelerde daha düşük çıkmıştır.

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c	Cohesion
D	Penetration depth
E	Magnitude of internal electric field
f	Frequency
I_P	Plasticity index
P	Power absorbed per unit volume
T_c	Temperature where thermal runaway would start to occur
T_{crit}	Critical temperature
ε	Dielectric constant
ε'	Real permittivity
ε''	Dielectric loss factor
ε^*	Complex permittivity
ε_0	Permittivity of free space
ε_{eff}''	Effective relative dielectric loss factor
ε_r'	Relative dielectric constant
ϕ	Internal friction angle
λ_0	Free space wavelength
σ	Effective conductivity
σ_n	Normal stress
$\tan\delta$	Loss tangent
ω	Water content
ω_c	Conventional oven water content
ω_L	Liquid limit
ω_m	Microwave oven water content
ω_p	Plastic limit
w_s	Dry mass of soil
w_w	Mass of water in the soil

I. INTRODUCTION

Compacted clay is widely used as a barrier in waste containment structures. A major application is landfill cover and liner construction. The basic principle behind hydraulic barriers are to minimize the flow of water. Landfills are used to keep the waste confined. To do this the entrance of water should be prevented. Therefore the permeability of the clay cover is very important. Studies have shown that freeze/thaw effects can increase permeability of clay liners one to two orders of magnitude. This could be very detrimental to the landfill and the surrounding environment. The increase in permeability is caused by the formation and expansion of ice in soil. When the soil thaws, cracks are left creating pathways for water movement.

One of the problems caused by freeze and thaw effect is that the liner should be kept from freezing before it is covered with enough waste material. By burying compacted clay liner, freezing depth should be decreased which in turn can increase the total cost of the design.

Freeze and thaw effects should be considered in the design of the pavements, foundations of the structures, retaining walls, and utility poles.

The objective of this study is to investigate the effects of freeze and thaw on compacted clay samples by means of microwave thawing. The use of microwave energy for processing materials has been the subject of a large amount of exploratory research. In this study, the aim of using microwave energy is to decrease the duration of freeze and thaw cycle by half as compared to conventional heating method and to see whether if microwave energy affects the strength and structure of compacted clay or not with respect to conventional heating in freeze and thaw cycles. Different types of strength tests were applied after freeze and thaw cycles in the laboratory. Kaolinite was used for compacted clay samples since it has the largest hydraulic conductivity among different types of clays.

II. LITERATURE REVIEW

2.1. Frost Effects

Two phenomena must be distinguished in considering frost action in fill areas, subgrade or subbases of roads, highways and structures, frost heaving during freezing and loss in bearing capacity during thawing.

Subgrade soils under roadway service conditions frequently experience increases in the degree of saturation because of the capillarity rise of groundwater or the infiltration of surface waters. Experience indicates that an increase in moisture content is accompanied by a reduction in dry-unit weight and a coincidental loss of soil strength. This problem is further complicated by seasonal variations in climate. When the ambient temperature falls below freezing, the moisture within the upper reaches of the soil mass freezes and results in a volume increase and the development of ice lenses within the soil matrix, which are reflected as a loss of strength. Of even greater significance to the function and maintenance of highways and airfields is the loss of subgrade strength, which occurs during thaw. As ambient temperatures rise, thawing of the subgrade progresses from the surface downward. Free water from the thawed ice cannot easily drain downward because of the underlying ice barriers, and, in the absence of adequate drainage facilities, it produces highly saturated conditions in the upper parts of the soil and significant loss of strength. Consequently, pavement failures frequently occur during spring thaw as a direct result of the loss of strength of the underlying base course or subgrade soils [1].

Freezing causes volume to increase in soils because of volume expansion of interstitial water and formation of ice lenses in the soils. Frost heaving in soils may result in engineering problems and failures in hydraulic structures, highway pavements, and buildings. Because of the detrimental effects of the frost action, considerable progress has been made in clarifying the mechanism of frost action, and in developing technics for handling frost-susceptible soils [2].

Anytime the air temperature falls below freezing, especially for more than a few days, it is possible for the pore water in soils to freeze. Frost action in soils can have several important engineering consequences. First, the volume of the soil can immediately increase about 10 per cent just due to the volumetric expansion of water upon freezing. A second but significantly more important factor is the formation of ice crystals and lenses in the soil (Figure 2.1). These lenses can even grow to several centimetres in thickness and cause heaving and damage to light surface structures such as small buildings and highway pavements. If soils simply froze and expanded uniformly, structures would be evenly displaced since ice is significantly stronger than these light structures. However, just as with swelling and shrinking soils, the volume change is usually uneven, differential movement occurs, and that is what causes structural damage [2].

During spring, the ice lenses melt and greatly increase the water content and decrease the strength of the soil. Highway pavements especially can suffer serious structural damage during the spring thaw.

Freezing temperatures depend on the climatic conditions at the site. Ground cover, topography, presence of snow, and other factors locally affect the rate and depth of frost penetration. A groundwater table within the height of capillary rise provides the water to feed growing ice lenses. The soil must be fine enough for relatively high capillary pressures to develop and yet not so fine that the flow of water is restricted. Even though the capillary pressures are very high, unless the clay is relatively sandy or silty, the amount of water that can flow during a freezing spell is so small that ice lenses have little chance to form.

Just as with swelling and shrinking soils, frost action seriously affects structures such as small buildings and highway pavements which are found directly on the ground surface. Damage to highways in the northern countries because of frost action is estimated to amount to millions of dollars annually. But because of good fundamental understanding of the factors involved in frost action and heave, engineers have developed relatively successful methods for dealing with these problems. Positive measures for dealing with the potential damage to structures and highways include lowering of the ground water table and, depending on the depth of frost penetration, removal of frost susceptible soils in the subgrade or foundation.

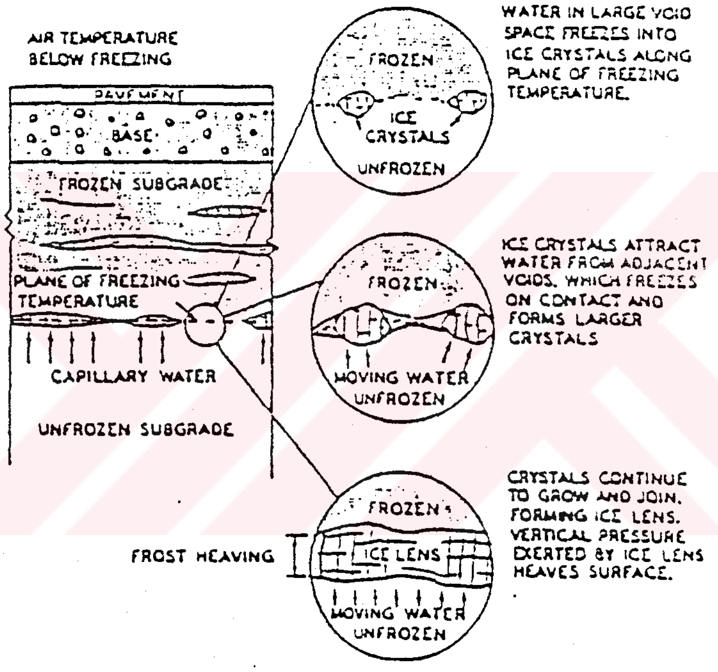


FIGURE 2.1. Formation of ice lenses at frost line (after Sutton, 1989), [2].

Frost action is dynamic, unsteady-state process, the nature of which is complex and involves a number of variables that are strongly interrelated. Because the intensity of the frost action depends on so many factors that are indeterminate, it is almost impossible to predict the amount of frost heave in a soil under field conditions by means of laboratory tests and available theory.

2.1.1. Heaving Effects

Frost heaving of soil is caused by crystallisation of ice within the larger soil voids and is usually a subsequent extension to form continuous ice lenses, layers, veins, or other ice masses. The growth of such distinct bodies of ice is termed ice segregation. A lens grows in thickness in the direction of the heat transfer until water supply is depleted, as by formation of a new lens at a lower level, or until freezing conditions at the freezing interface will no longer support further crystallisation. Numerous investigations have shown that ice segregation occurs only in soils containing fine particles. Such soils are said to be frost susceptible; clean sands and gravels are non-frost susceptible soils. The degree of frost susceptibility is principally a function of the percentage of fine particles and, to a lesser degree, of particle shape, distribution of the grain sizes, and mineral composition [3].

2.1.1.1. Conditions Necessary for Ice Segregation

The following three conditions of soil, temperature, and water must be present simultaneously in order for ice segregation to occur in the subsurface materials:

1. The soil must be frost susceptible.
2. Freezing temperatures must penetrate the soil. In general the thickness of the particular layer or lens of ice is inversely proportional to the rate of penetration of freezing temperature into the soil.

3. A source of water must be available from an underlying groundwater table, infiltration or gravitational flow, an aquifer, or the water held within the voids of fine grained soil.

2.1.1.2. Uniform Heaving

Uniform heaving results when soil and moisture conditions conducive to ice segregation exist under a pavement but are uniform in longitudinal and transverse directions, and freezing temperatures penetrate into the pavement structure at the same rate throughout the paved area. Under those idealised conditions, the surface of the pavement is raised uniformly.

2.1.1.3. Differential Heaving

Differential heaving is indicated by the presence of surface irregularities, bumps, and general surface roughness in winter as a result of nonuniform conditions of soil and moisture availability. Distinctive cracking of the wearing surface may cause damage to vehicles and loss of vehicle control. Surface drainage may be impeded. Pavement roughness is reflected in a marked decrease in serviceability during the frozen condition; subsequent weakening during thaw causes accelerated deterioration and impaired pavement performance.

2.1.1.4. Development of Permanent Roughness

Permanent pavement roughness develops from the cumulative effects of traffic loads and from various nontraffic-load-associated causes. It is directly affected by frost action because the weakening of the subgrade during thawing of segregated ice leads to acceleration of rutting in flexible pavements and to cracking and faulting in rigid pavements.

2.1.2. Thawing Effects

2.1.2.1. Temperature Distribution During Thawing

One of the most critical phases in the annual cycle of environmental changes affecting pavements in seasonal frost areas are periods of thawing. Depending on the rapidity of the thaw and the drainage capabilities of the pavement system, these thawing cycles can be very disruptive in many cases. Considerable melting of snow may occur during thaw periods, and after that, melt water infiltrates into the pavement from the shoulders and through the surface cracks. Bearing capacity is worthy reduced during thawing periods, and after midwinter thaw periods, frost heaving frequently is more severe. In areas where deep frost penetration takes place, the most damaging of the thaw periods is the period of complete thawing in spring that affects the thicker pavement structures because it gives damage both to the subgrade, subbase and also to the base layers [4].

Thawing can proceed from the top downward, or from the bottom upward, or both. The direction of thawing process is dependent on the pavement surface temperature. Melting will proceed almost entirely from the surface downward during a spring thaw which leads to extremely adverse drainage conditions [5]. The still-frozen soil beneath the thawed layer traps the water that is released by the melting of the ice lenses so that lateral and surface drainage are the only means of egress. Lateral drainage may be restricted by still-frozen shoulders resulting from the insulating effect of snow and/or different thermal conductivity and surface reflectivity characteristics in the granular soils. If in the spring, air temperatures remain cool and frosty at night, heat stored in the ground from the previous summer is conducted upward and thawing will principally proceed from the bottom upward. Such thawing permits soil moisture from melted ice lenses to drain downward while the material above it remains frozen.

2.1.2.2. Loss of Bearing Capacity

During spring thawing periods, loss of bearing capacity is a recognised effect of frost action that severely affects pavements' performance. Thawing causes a sharp decrease in the subgrade bearing capacity, to levels below the summer/fall values, which is followed by a gradual recovery over a period of several weeks or months.

There are several different thaw weakening mechanisms. As Chamberlain stated [6], the first mechanism occurs by the migration of moisture toward the freezing front during the development of ice segregation and also by the following volume increase of the soil in order to feed the ice formation. Field and laboratory freezing test results show that in frost-susceptible soils, a migration of moisture takes place toward the freezing zone so as to form ice lenses of various thickness. The thickness of ice lenses are governed by the temperature gradient and the available water. These lenses may form by redistribution of moisture within a narrow range of soil depth and that causes saturation of the soil and leads to a depletion of moisture from an adjoining area or level. If ice segregation caused by thawing occurs at a rate faster than the released moisture can escape into underdrain or into more pervious layers of the pavement system, or if it is reabsorbed into adjacent drier areas, excess pore water pressure occurs. This pore water pressure causes a decrease in the load carrying capacity. Therefore the inability of the water, that is melt by thawing, to escape is an important criteria for the load carrying capacity of the soil (Figure 2.2). Going deeper, thawing proceeds from below at a slower rate, these causes a reduction in pore water pressure development so loss of supporting capacity becomes less important (Figure 2.3) [2].

2.1.2.3. Increasing Hydraulic Conductivity

An increase in the permeability of a fine-grained soil results from freeze and thaw cycles. This increase was found to be more in the first few cycles and then observed to level off. This effect of freeze and thaw cycles is more important for clay liners as the

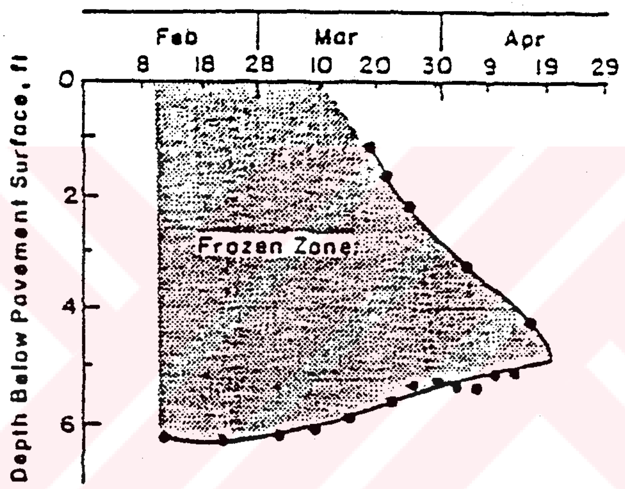


FIGURE 2.2. Limits of frost (after Sutton, 1989), [2].

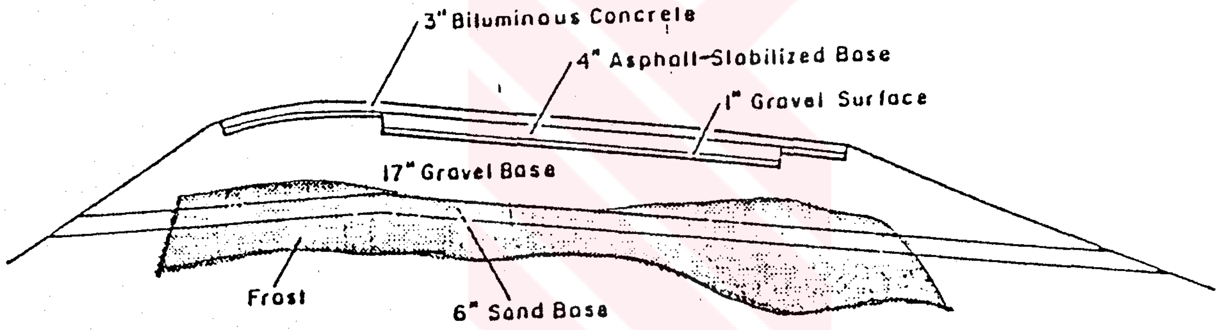


FIGURE 2.3. Frost remaining in partially thawed subbase and subgrade (after Sutton, 1989), [2].

effectiveness of compacted clay in a liner system is controlled by the hydraulic conductivity. These cycles cause problems in two cases:

1. If a liner is exposed to freezing temperatures before it is covered with enough waste material, it should be protected from freezing.
2. If freezing damages compacted clay, the clay within the cover system must be buried at a sufficient depth to prevent freezing.

2.1.2.4. Effect of Snow Melt

The thawing of seasonal frost is not greatly dependent on the effect of snow melt. The melting of snow cover occurs entirely at its surface, and, until it becomes fully ripened, very little melt water is allowed to percolate to the soil and snow interface. If at the same time ripening has taken place and melt-water, and the base of the snow cover are all nearly in thermal equilibrium with each other.

The drainage of water from the thawed soil layer is restricted by the impermeability of the still-frozen soil below if the snow cover has disappeared and seasonal frost has begun to thaw from the ground surface downward. In this case, the melt-water that is remained at the pavement edge, may flow or percolate to the site and contribute to the saturation of the thawed soil layer. Thus the snow-melt, that is directly added to the moisture content of the surface soil layers at the shoulder and under the pavement, leads still to greater reductions in bearing capacity. Another disadvantage is that, surface water that is exposed to solar radiation absorbs heat, and this heat helps the thaw process [7].

2.1.2.5. Differential Thaw

If over all parts of the pavement, thawing does not occur uniformly, nonuniform subsidence of the previously heaved surface results. These nonuniform surface causes pavement roughness and nonuniform subgrade support. Laboratory and field test results showed that, especially with the rigid pavements, cracks may develop more rapidly during spring frost-melting period. Factors of differential thawing can be given as:

1. Different thermal properties of adjacent sections of pavement, such as type of soil. water content, density, and the nonuniformity of the subsoil.
2. Nonuniform exposure to sun's rays and differing angle of incidence.
3. Shaded portions on pavement due to deep cuts, trees, overpasses, or buildings.
4. Conditions for surface and subsurface drainage.
5. Different colors of pavement. Darker colored pavement reflects less of the sun's rays than do the lighter ones.

2.1.2.6. Subsidence of Coarse Layers into Fine-grained Soils

Subsidence of coarse open-graded base or subbase materials into fine-grained soils is one of the undesirable consequences of thawing for frost susceptible areas. By impregnation of a silt subgrade upward into the base course may cause a nonfrost susceptible base to become frost-susceptible. Also irregular subsidence of the pavement surface can occur as a result of the base course contaminated with fine subgrade soils which occupy less amount of volume. These effects can be prevented if a soil containing more medium to fine sands is selected as the base course [8].

2.1.2.7. Pavement Cracking

Frost or low temperatures are the causes of two important types of pavement cracking. The first type of pavement cracking, is an advanced stage of distortion with random type of orientation and spacing which is caused by differential frost heaving. This type of cracking can both occur in asphaltic concrete and portland cement concrete pavements. With relatively thin asphalt-bound layers, flexible pavements can bear some differential heave without severe cracks. On the other hand, rigid pavements have less tolerance of differential heave. On older and secondary roads underlain by frost-susceptible soils with inadequate frost protection and adverse moisture conditions, cracking caused by frost heave is a major problem as it accelerates the deterioration of the pavement. Major roads of recent design and construction, with effective control of differential heaving, have no such type of cracks [9].

The second type of cracking occurs as a result of the thermal contraction. Thermal contraction induces stresses in the surfacing materials as they are partially restrained by friction along the interface with the supporting layer. In both flexible and rigid pavements, differential horizontal movements occur, but in rigid ones they are prevented by joints or resisted and distributed by steel reinforcing. Therefore, these movements only cause problems with the flexible pavements.

2.1.2.8. Identification of Factors Contributing to Frost Problem

Climate is the main factor to frost problem. Wind, air temperature, solar radiation received at the surface, and precipitation are the major factors of climate that affect the severity of frost action. Air temperature, solar radiation received at the surface, and wind mainly affect the temperature regime in the pavement system, those can change the depth of frost penetration, number of freeze and thaw cycles, and duration of the freezing and thawing periods. Precipitation mainly affects the moisture regime but by interacting with other climatic factors, it has also an effect on the ground temperatures as well.

Frost penetration depth in the soils depend mainly on the intensity and duration of subfreezing air temperatures, surface conditions, type of soil and its density, and moisture content. Frost penetration is greater in paved areas that are underlain by granular soils with low moisture content than in fine-grained areas with high moisture contents.

Studies have shown that a soil is frost-susceptible if it contains fine particles. Also it has been observed that grain shape, grain size distribution, mineral composition, and plasticity characteristics greatly contribute to the ice segregation.

Frost action consists of both heaving and thaw-weakening effects. But thaw-weakening is not directly related to the heaving action because well-drained sandy gravelly materials are observed to recover their bearing strength quite rapidly. On the other hand clayey soils may show little heave but recover stability very slowly over a long period of time.

Solution of the problem of characterising the susceptibility of soils to frost action, probably will require the acceptance by highway engineers of more complex testing and analytical procedures. Heave pressure tests, permeability tests, suction measurements, void size distribution determinations, and rapid freeze tests for identifying frost-susceptible soils can be suggested.

The thermal properties; volumetric heat capacity, volumetric latent heat of fusion, and coefficient of thermal conductivity, have significant influence on the rate and magnitude of frost heaving. Each of these thermal properties is influenced by the type of the soil, density, and moisture content. The volumetric latent heat of fusion and the thermal conductivity are most important and generally have opposing influences on the depth and rate of frost penetration.

Restriction of drainage by frozen soil is a common problem in seasonal frost areas. The moisture that may have accumulated in the base, subbase, and in the subgrade is released during thawing. If it could drain downward and/or laterally, little loss of strength would result. Vertical and lateral drainage may be blocked during thawing periods. That is why consolidation of the frost-loosened subgrade is hindered, and the base and the subbase materials also may become weakened by excess moisture and pore water pressure, leading to accelerated pavement distress.

2.2. Microwave Effects

2.2.1. What is microwave?

Microwaves are electromagnetic waves oscillating at a frequency within the range of roughly 0.3 to 300 Ghz and corresponding wavelengths ranging from 1 m to 1 mm. Industrial microwave processing is usually accomplished at the frequencies set aside for industrial use, 915 Mhz, 2.45 Ghz, 5.8 Ghz, and 24.124 Ghz. In contrast with visible waves, except for lasers, microwaves are coherent and polarised. Microwaves also obey the law of optics and can be transmitted, absorbed or reflected, depending on the material type as shown in Figure 2.4, [10].

2.2.2. Microwave Processing Systems

In general, microwave processing systems consist of a microwave source, an applicator to deliver the power to the sample, and systems to control the heating. Microwave generators are generally vacuum tubes, but solid state devices are sometimes used. The magnetron is the most common microwave source in materials processing applications. Microwave energy is applied to samples via microwave applicators. The most common applicators are multimode (eg., home ovens), where numerous modes are excited simultaneously, and single mode, where one resonant mode is excited. Control of temperature in microwave heating processes is generally accomplished through variation of input power or through pulsed sources [10].

Many different types of devices have been developed to produce microwaves, but, for industrial applications, the magnetron operating at 2.45 Ghz is the most popular. Regardless of the production device used, the microwaves, once generated, are conducted

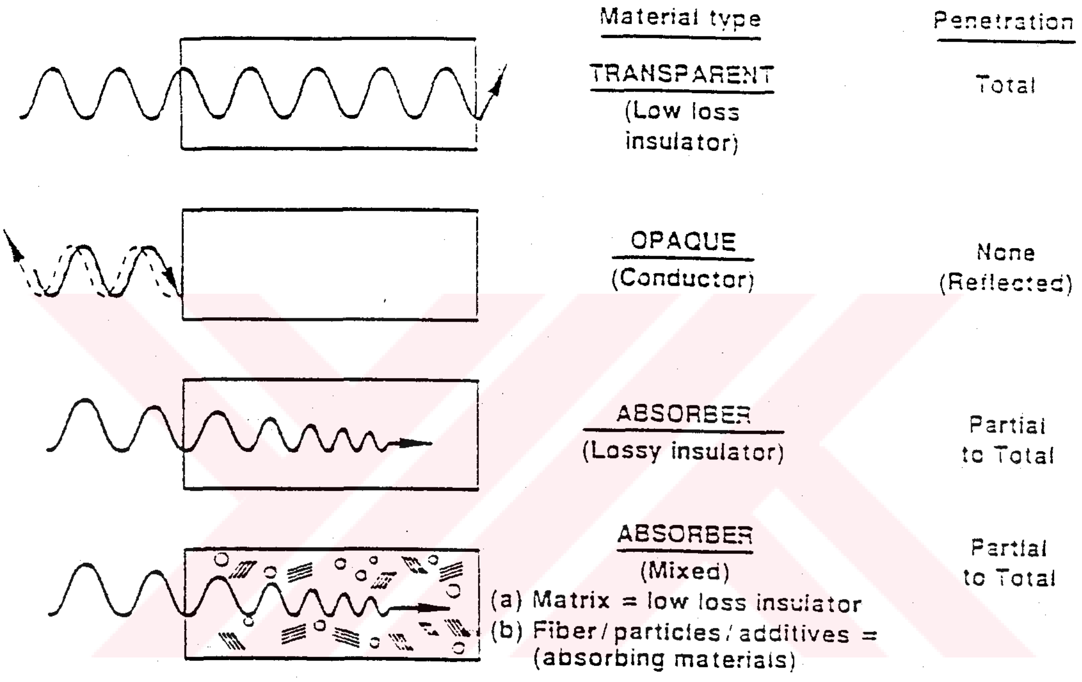


FIGURE 2.4. Interaction of microwaves with materials (after Sutton, 1989), [10].

by an electromagnetic transmission line to an 'applicator' in which the ceramics to be joined are placed.

2.2.2.1. Features of Microwave Processing Equipment

Some of the key features of microwave processing equipment are shown in Table 2.1.

TABLE 2.1. Features Required for Microwave Processing Equipment [11].

Capable of Processing

- Gases, solutions, powders, bulk solids, composites, multilayers, etc.
- Temperature range <<0° to > 2000°C
- Environment
  - vacuum, air, controlled atmosphere, pressures to many atmospheres
  - controlled reactive gas compositions

Controls

- Simple to complex (automated, computer) controls for power, temperature, pressure, gas composition, etc.

Power

- Few watts to 100's kilowatts (present time)

Frequency

- 0.915 GHz and 2.45 GHz (predominant); industrial, R&D
- 2.5 - 100 GHz- R&D
- 37 and 84 GHz - Industrial (Ukraine)

Applicators

- Types - multimode, single mode, travelling wave, mm-beam
- Simple cavity to complex and customised
- Clean cavity (or reaction vessel) remote from power source
- Hot press, hot isostatic press, and pressure chambers
- Continuous vs. batch processor

2.2.3. Characteristics of Microwave Processing

Microwave processing is complex and multidisciplinary in nature and involves a wide range of electromagnetic equipment design and materials variables, many of which change significantly with temperature. A high degree of technical and other knowledge is required to determine how, when, and where to use microwaves most effectively, and when not to use them.

Commercially successful applications of microwave processing take advantage of characteristics unique to microwaves.

2.2.3.1. Advantages of Microwaves over Conventional Heating Methods

Unlike other heat sources, such as conventional ovens and furnaces, lasers, plasmas, and electron beams, where heat is applied externally to the surface of the material, microwave irradiation penetrates and simultaneously heats the bulk of the material. This feature, along with other microwave characteristics, presents new and unique opportunities to process materials with greater flexibility, greater speed and energy savings, improved quality and product properties, and to create or synthesise entirely new types of materials that cannot be produced by other heating methods.

There are several reasons for the growing interest in microwave processing over conventional processing methods, including the potential for

1. Significant reductions in manufacturing costs due to energy savings and shorter processing times,
2. Improved product uniformity and yields,
3. Improved or unique microstructures and properties,
4. Synthesis of new materials.

Microwave heating is fundamentally different from conventional processes. In the microwave process, heat is generated internally within the material instead of originating from external heating sources. As a result of this internal and volumetric heating, the thermal gradients and the flow of heat in microwave-processed materials are the reverse of those in materials processed by conventional heating as indicated in Figure 2.5.

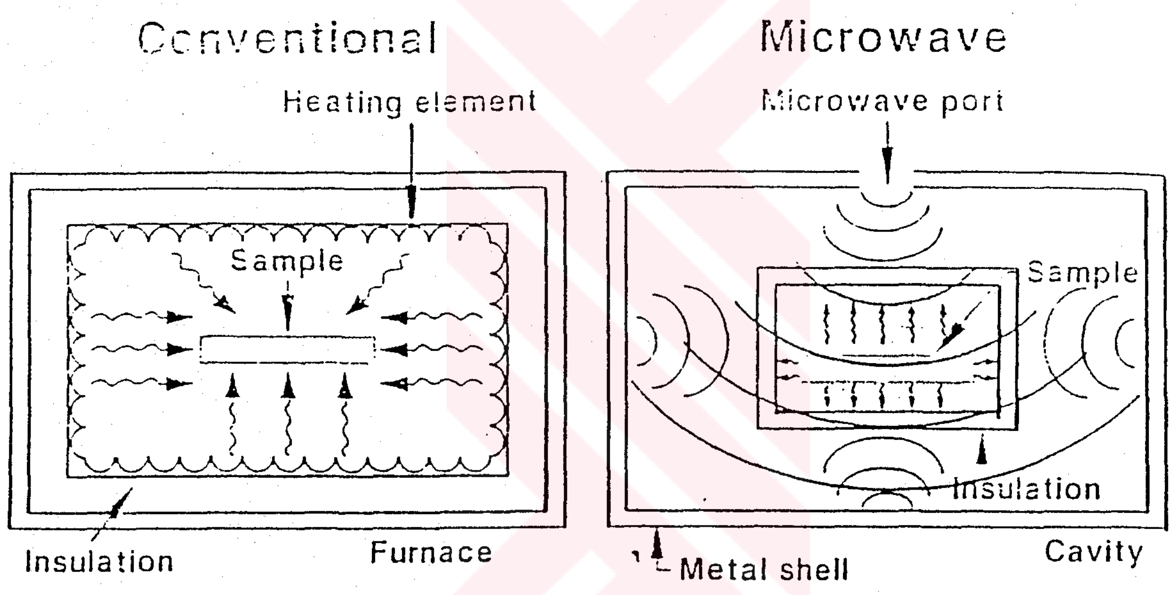


FIGURE 2.5. Heating patterns in conventional and microwave furnaces (after Sutton, 1989), [10].

Consequently, microwave processing makes it possible to heat both small and large shapes very rapidly and uniformly; to efficiently remove volatile constituents (binders, moisture, etc.) from thick sections; and to reduce thermal stresses that cause cracking during processing. Since the absorption of microwave energy varies with the composition and structure of different phases, selective heating is also possible and presents new opportunities. These and other characteristics and advantages of microwave processing are summarised in Table 2.2, [10].

Microwaves possess several characteristics that are not available in conventional processing of materials, including:

1. penetrating radiation,
2. controllable electric-field distributions,
3. rapid heating,
4. selective heating of materials through differential absorption, and
5. self-limiting.

These characteristics, either singly or in combination, present opportunities and benefits that are not available from conventional heating or processing methods and provide alternatives for the processing of a wide variety of materials, including rubber, polymers, ceramics, composites, minerals, soils, wastes, chemicals, and powders. The characteristics of microwaves also introduce new problems and challenges, making some materials very difficult to process. First, bulk materials with significant ionic or metallic conductivity cannot be effectively processed due to inadequate penetration of the microwave energy. Second, insulators with low dielectric loss factors are difficult to heat from room temperature due to their minimal absorption of the incident energy. Finally, materials with permittivity or loss factors that change rapidly with temperature during processing can be susceptible to uneven heating and thermal runaway. While the use of insulation or hybrid heating can improve the situation, stable microwave heating of these types of materials is problematic.

Due to the high cost of microwave generators and the relatively poor efficiency of electric power for heating applications, factors other than energy generally account for savings realised from microwave processing. Such factors include process time savings, increased process yield, and environmental compatibility.

Microwaves can penetrate up to many meters in electrically insulating materials, such as ceramics, polymers, and certain composite materials. The depth of penetration depends on several factors, including the wavelength of the radiation and the dielectric (and magnetic) properties of the material. During the time that a material is exposed to penetrating microwave radiation, some of the energy is irreversibly lost (absorbed), which in turn generates heat within the volume (or bulk) of the material. This bulk heating raises the temperature of the materials such that the interior portions become hotter than the surface, because the surface loses heat to the cooler surroundings. This is the reverse of conventional heating, where heat from an external source is supplied to the exterior surface and diffuses toward the cooler interior regions. Thus, the reverse thermal gradients in microwave heating provide several unique benefits, which include rapid volumetric heating without overheating the surface, especially in materials with low thermal conductivity; reduced surface degradation during the drying of wet materials; and removal of binders or gases from the interior of porous materials without cracking, or conversely, penetration of reactive gases (during chemical vapour infiltration) or fluids into the hotter interior portions of porous materials and preforms, then condensation into solid matter prior to the filling of voids or pores at the outer regions.

Since heating is instantaneous with power input, the temperature of a material can be precisely controlled by controlling the power input. However, this is not a simple or straightforward situation, since the internal generation and surface dissipation of heat depends on many factors, which also have to be taken into account during the full heating and cooling schedule. Consequently, a detailed understanding of the microwave/material interactions, and the ability to numerically simulate, model, and predict the heating patterns for a specific material in a given microwave applicator, will play an increasingly important role in developing practical and effective controls over the various parameters for precise heating.

TABLE 2.2. Advantages of Microwave Heating over Conventional Heating Processes [10].

Characteristic	Advantages over conventional heating processes
Direct coupling (absorbing) of microwave creates volumetric (bulk) heating	*Potential to heat large sections uniformly *Reversed thermal gradients; surface cooler than interior *Process materials at lower surface temperatures *Rapid removal of water, binders, and gases without rupture or cracking *Internal stresses reduction by lower thermal gradients *Heat in clean (pure) environment; air, controlled atmospheres, vacuum or pressure *Control partial pressure of reactive gases for selective oxidation/reduction of certain phases *Improvement of product quality, uniformity, and yields *Instantaneous response to microwave power changes *Low thermal mass; precise and automated temperature control
Dielectric losses (and heating) accelerate very rapidly with increasing temperatures above T_{crit}	*Ability to heat "transparent" materials above T_{crit} *Very rapid processing (2 to 50 times faster than conventional) *Densify materials rapidly with minimum grain growth (accelerated sintering) *Reduce process costs (time, energy, and labor) *Ability to heat ceramics well above 2000°C (in air, vacuum, or controlled atmospheres)
Microwaves are polarised and coherent; location of maximum electric and magnetic fields can be controlled	*Capability of high-energy concentration in short times and in selected regions *Frequency and power level optimization for given material, size and shape *Potential for process automation, flexibility, efficiency, and energy savings *Precise heating of selected regions, ie., brazing or sealing of joints, fiber drawing, and plasma generation *Acceleration of sintering and diffusion due to high electric fields; thus densification at lower temperatures
Differential microwave coupling of phases, additives, and constituents leads to selective heating	*Synthesis of new materials and microstructures *Heating of selected zones (brazing and scaling) *Enhanced coupling of microwave transparent materials *Use of fugitive coupling materials for preheating of otherwise transparent materials *Use of microwave-coupling materials as shapes or containers to heat the more transparent materials *Superior control over state of oxidation through selective heating of phases and control over oxygen partial pressure

Since microwaves can be transmitted through air, various gases, or vacuum for long distances without significant loss in electric-field strength, the power source can be remote from the applicator and sample. This makes it possible to heat in a very clean or controlled environment.

With these benefits, new problems have been encountered, such as the difficulty of trying to heat microwave-transparent materials from room temperature without generating hot spots, cracking, and arcing. At higher temperatures, the dielectric losses (and microwave absorption) of many of the materials accelerate rapidly with increasing temperature, which speeds nonuniform heating, warpage, and rupture problems.

In a single-mode applicator, the electric-field distributions can be focused and controlled to provide very high field strengths. This provides a means (with proper tuning) to heat low-loss materials at low temperatures; to heat materials of irregular shape; to heat selected regions between two materials to promote welding, brazing, or bonding; or to generate plasmas for microwave-assisted sintering or chemical vapour deposition. While the single-mode processing systems allow for precise and localised heating, they are very much costly than the multimode systems and at present are not set up for large volume processing (other than plasma applications).

At very high microwave frequencies (ie., above 30 Ghz), the wavelengths are sufficiently short that they can be beamed, focused, and rastered with metallic mirrors over desired locations on a given sample. The microwave beams are far more penetrating to some classes of materials than those of lasers or electrons, so the depth of processing can be greatly increased.

For many materials, dielectric losses above a critical temperature accelerate with increasing temperature, allowing very rapid (bulk) heating that can result in very significant reductions in processing time. This rapid increase in dielectric loss can lead to uneven heating and thermal runaway. If thermal runaway is controlled using hybrid heating or insulation, this phenomenon can allow the processing of low-loss ceramic materials, where both rapid heating and very high processing temperatures are desired.

The range of dielectric responses of different materials and their ability to couple with (absorb) microwaves is one of the most widely used features of microwave processing. For example, water is a strong, broad frequency-band absorber of microwaves.

This characteristic is widely used in selective heating for processing and heating food and in drying or dehydrating a wide range of wood products, chemicals, and many other materials. The processing of rubber, asphalt, and many composite materials is dependent on the selective or widely differential heating of at least one of the constituents. Hybrid microwave heating is another example of where selective heating has been used to significant advantage.

In several cases, microwave heating will cease once the source of differential absorption, such as water, has been removed or has been altered during a phase change in the material during processing. Self limiting absorption can also occur when two materials with different coupling characteristics, such as SiC and ZrO_2, are simultaneously irradiated with microwaves. This principle is used in hybrid heating, where suspecters are used initially to hybrid-heat low-loss materials from room temperature.

The characteristics of processes that may potentially make them attractive for microwave processing are listed below.

1. The size or thickness of the material should be large.
2. The cost of the material should be high.
3. Improvements in properties obtainable from microwave processing are significant.
4. Plant space is limited.
5. Electricity is cheap.
6. Minimising handling is advantageous.

Other characteristics may include:

- heat from the combustion of coal, oil, or natural gas are not practical (ie., electricity is the only power source).
- maintaining a very clean, controlled processing environment is important.

2.2.4. Materials Interaction

When an electric field interacts with a material, various responses may take place. In a conductor, electrons move freely in the material in response to the electric field, and an electric current results. Unless the material is a superconductor, the flow of electrons will heat the material through resistive heating. However, microwaves will be largely reflected from metallic conductors, and therefore such conductors are not effectively heated by microwaves. In insulators, electrons do not flow freely, but electronic reorientation or distortions of induced or permanent dipoles can give rise to heating. The common experience of using microwaves to heat food is based primarily on the dipole behaviour of the water molecule in the food and the dipole's interaction with microwaves. Because microwaves generate rapidly changing electric fields, these dipoles rapidly change their orientations in response to the changing fields. If the field change is occurring near the natural frequency at which reorientation can occur, a maximum in energy consumed is realised, and optimum heating occurs. In the terminology of microwave processing, when this happens it is said the material is well "coupled" [10].

The material properties of greatest importance in microwave processing of a dielectric are the permittivity (often called the dielectric constant), ε, and the loss tangent, $\tan\delta$. The complex permittivity is a measure of the ability of a dielectric to absorb and to store electrical potential energy, with the real permittivity, ε', characterising the penetration of microwaves into the material and the loss factor, ε'', indicating the material's ability to store the energy. The most important property in microwave processing is $\tan\delta$, indicative of the ability of the material to convert absorbed energy into heat. For optimum coupling, a balanced combination of moderate ε', to permit adequate penetration, and high loss (maximum ε'' and $\tan\delta$) is required [10].

The degree of interaction (or absorption) of microwaves by a dielectric material is related to the material's complex permittivity, ε^* (F/m), which is composed of a real part (ε', dielectric constant) and an imaginary part (ε'', dielectric loss factor) by

$$\varepsilon^* = \varepsilon' - j\varepsilon'' = \varepsilon_0 (\varepsilon'_r - j\varepsilon''_{eff}) \tag{2.1}$$

where $j=(-1)^{1/2}$, ε_0 is the permittivity of free space ($\varepsilon_0 = 8.86\text{x}10^{-12}$ F/m), ε'_r is the relative dielectric constant, and ε''_{eff} is the effective relative dielectric loss factor [10].

When microwaves penetrate and propagate through a dielectric material, the internal electric fields generated within the effected volume induce translational motions of free or bound charges (e.g.,electrons or ions) and rotate charge complexes such as dipoles. The resistance of these induced motions due to inertial, elastic, and frictional forces, which are frequently dependent, causes loses, volumetric heating results. For convenience, the loss mechanisms are all combined together in one loss parameter, ε''_{eff}. However, the loss tangent (tanδ) is commonly used to describe these losses as

$$\tan\delta = \varepsilon''_{eff} / \varepsilon'_r = \sigma / 2\pi f \varepsilon_0 \varepsilon'_r \tag{2.2}$$

where σ is the total effective conductivity (S/m) caused by conduction and displacement currents and f is the frequency (Ghz).

The power absorbed per unit volume P (W/m^3), provides the following basis for heating:

$$P = \sigma |E|^2 = 2\pi f \varepsilon_0 \varepsilon'_r \tan\delta |E|^2 \tag{2.3}$$

where E(V/m) is the magnitude of the internal field. Equation shows that the power absorbed varies linearly with the frequency, the relative dielectric constant, and tanδ, and varies with the square of the electric field.

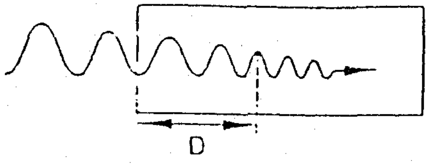
Since the electric fields are attenuated as the microwaves penetrate and propagate through an absorbing material, a useful parameter describing this is D, which is the penetration depth at which the incident power is reduced by one half.

$$D=3\lambda_0 / 8.686\pi \tan\delta \, (\varepsilon'_r / \varepsilon_0)^{1/2} \tag{2.4}$$

where λ_0 is the incident or free-space wavelength. For greater wavelengths (and lower frequencies), the penetration depth is also greater, as shown in Figure 2.6. for water. Although low frequencies result in greater penetration depths, the heating does not necessarily increase, since the internal field E could be low, depending on the properties of the material.

The relative dielectric constant (ε'_r) and the loss tangent ($\tan\delta$) are the most widely used and measured parameters that describe the behaviour of a dielectric under the influence of a microwave field. They both affect the power absorbed and the half-power depth, and thus, they influence the volumetric heating behaviour of a given material. The value of ε'_r is a measure of the polarizability of a material in an electric field, whereas the value of $\tan\delta$ is a measure of the loss (or absorption) of the microwave energy within the material. During heating ε'_r and $\tan\delta$ change with temperature, and a knowledge of these changes is important for process control. In contrast to ε'_r , $\tan\delta$ is far more affected by temperature. In general, $\tan\delta$ initially rises slowly with increasing temperature, until some critical point (T_{crit}) is reached, beyond which $\tan\delta$ rises rapidly. At temperature below T_{crit}, $\tan\delta$ rises very rapidly, which causes a condition of thermal runaway in a microwave-heated material. As $\tan\delta$ begins to increase rapidly, the material begins to absorb microwave energy more efficiently, which also raises the temperature. This causes $\tan\delta$ to rise even faster. The net result is an exponential increase (runaway) in temperature. The rate of temperature rise and the triggering temperature (T_{crit}) vary widely for different materials.

Thermal runaway is an important aspect of microwave heating. It can cause undesirable hot spots within a material; it can also be used to heat materials at rapid rates. There are several means to control or prevent thermal runaway, such as by controlling or pulsing the microwave power level (where changes in the power cause an instantaneous



$$D = \frac{3\lambda_o}{(8.686\pi)(\tan\delta)\sqrt{\epsilon_r/\epsilon_o}}$$

D = penetration depth at 1/2 power
λ_o = free space wavelength
$\tan\delta$ = dielectric loss tangent
ϵ_r/ϵ_o = dielectric constant

D_{H_2O} (cm)	f (GHz)	λ_o (cm)
7.6 (3")	0.915	32.8 (12.8")
1.3 (0.5")	2.450	12.3 (4.8")

FIGURE 2.6. Absorption of microwaves to half-power depth, D (after Sutton, 1989), [10].

response in the material) or by designing the microwave system and applicator to deposit the microwave energy within the material or product in a described manner.

The trick in microwave processing is to find a material that is polarisable and whose dipoles can reorient rapidly in response to changing electric-field strength. Fortunately, many materials satisfy these requirements and are therefore candidate materials for microwave processing. However, if these materials possess poor thermal conductivity, heat does not rapidly dissipate into the surrounding regions of the material when a region in the solid becomes hot. This difficulty is compounded, because the dielectric loss frequently increases dramatically as the temperature increases. Thus the hot region becomes even hotter, sometimes resulting in local melting. These "hot spots" are a major difficulty and have led to the use of hybrid systems, combining microwave heating with other heat sources to reduce uneven heating.

The successful use of microwaves requires the processor to have a good understanding of the strengths and limitations of microwaves. Among the strengths are penetrating radiation, controllable electric-field distributions, rapid heating, selective heating, and self-limiting reactions.

The required degree of knowledge and understanding of microwave/material interactions increases with the difficulty of the material to couple with microwave radiation and the complexity of the process. Some materials couple readily and can be processed effectively in relatively simple multimode cavities. A good example is water, which exhibits broad band absorption at microwave frequencies, which undoubtedly led to the earlier successes in food processing and in drying materials. However the coupling efficiency can be quantified, if the dielectric properties are known over a range of temperatures and frequencies. The dielectric loss factor is a direct measure of a materials' ability to absorb microwaves and be heated. Fig shows that even water can be heated much more efficiently at 24.125 Ghz than 2.45 Ghz, which is the frequency used in commercial processing, and where the microwave equipment is far less expensive.

However, it is clear that the loss factor of water varies significantly and nonlinearly with temperature and frequency. For many solids, where there are no phase changes, the loss factor increases continuously with increasing temperatures. The dielectric constant is another measure of a material's ability to interact with microwave irradiation, and provides a quantitative assessment of the material's ability to store (reversibly) energy from the electric field, and to reflect incident radiation (due to an impedance mismatch at the surface with the surrounding medium).

2.2.5. Microwave Applications

Microwave energy has found general, commercial application in very few areas. These include food processing, analytical chemistry, and heating and vulcanisation of rubber. Food processing and rubber manufacture involve relatively high volume, continuous processing. Analytical chemistry applications are broad in scope and involve high-volume, repetitive, batch processing, often with long intermediate drying and reaction steps that can be shortened using microwave heating.

Much work has been undertaken to investigate the use of microwaves for the processing of a wide range of materials, including ceramics, polymers, composites (ceramic and polymer matrix), powders, and minerals. Microwaves have also been investigated in a broad range of plasma processes (surface modification, chemical vapour infiltration, powder processing), chemical synthesis and processing, and waste remediation. Despite the considerable effort that has been expanded in microwave process development, there has been little industrial application to date, with most of the effort still in the laboratory stage. Some of the more significant problems that have inhibited industrial application of microwave processing include:

- the cost of equipment;
- limited applicability;
- variation in dielectric properties with temperature; and
- the inherent inefficiency of electric power.

2.2.5.1. Ceramics/Ceramic Matrix Composites

The use of microwave energy for processing ceramics and ceramic matrix composites has been the subject of a large amount of exploratory research. The range of materials and processes that have been investigated is shown in Table 2.3, [12].

Microwave energy provides added flexibility and versatility as a new tool to process a wide variety of materials over a wide temperature range under air, vacuum, controlled atmosphere or pressure. Table 2.4 shows several types of materials that have been processed using microwave energy, and that the processing occurred when the materials were in the solid, liquid, or gaseous state, depending on the specific product [11].

TABLE 2.4. Microwaves as a Versatile Tool to Process Materials [11].

Material Types	Material Conditions
*Food, Textiles, Wood Products	*Gases
	Plasmas
	- Chemical vapour deposition
	- Induced sintering
*Polymers	- Polymer synthesis
	Gases
	- Chemical vapour infiltration
	- Reaction forming and synthesis
*Minerals	*Liquids
	- Solutions: dissolution, synthesis
	- Polymers: curing, casting, composite processing
*Ceramics	- Sol/gel: powder synthesis, dehydration
	- Suspensions: slip casting, drying
	*Solids
*Composites, Multilayers & Structures	- $T<500^{0"}$C: drying, binder burnout, dewaxing molds
	- $T>500^{0}$C: annealing, calcining, firing, sintering, joining, fiber drawing, combustion synthesis, inorganic synthesis
*Chemicals	

The microwave drying of ceramic materials and products, and the dewaxing of ceramic casting molds has been a commercial practise for over two decades.

Up to present time, many types of potential applications have been investigated and these are categorised in Figure 2.7.

The use of microwave energy in the processing of ceramic materials has been of interest to materials scientists for more than two decades. Microwave sintering, a major focus of current work, may offer superior material characteristics and would most certainly reduce the time required for that process step.

Microwave sintering of ceramics was found to be feasible by W. R. Tinga and his coworkers at the university of Edmonton, AB, Canada in mid-1960's. Some studies have

TABLE 2.3. Examples of ceramic microwave processing research and development (after Sutton, 1985), [12].

	Processes											
Materials	Annealing	Binder Burnout	Calcining	Drying	Fiber Drawing	Joining	Melting	Sintering	Combustion Synthesis	Powder Synthesis	Slip Casting	Clinker
Advanced Ceramics		X		X		X		X	X	X		
Cements												X
Composites						X		X				
Ferroelectrics	X	X	X					X				
Ferrites								X				
Glasses					X		X			X		
Minerals			X	X				X				
Refractories			X	X			X	X				
Superconductors	X			X				X		X		
Whitewares				X				X			X	

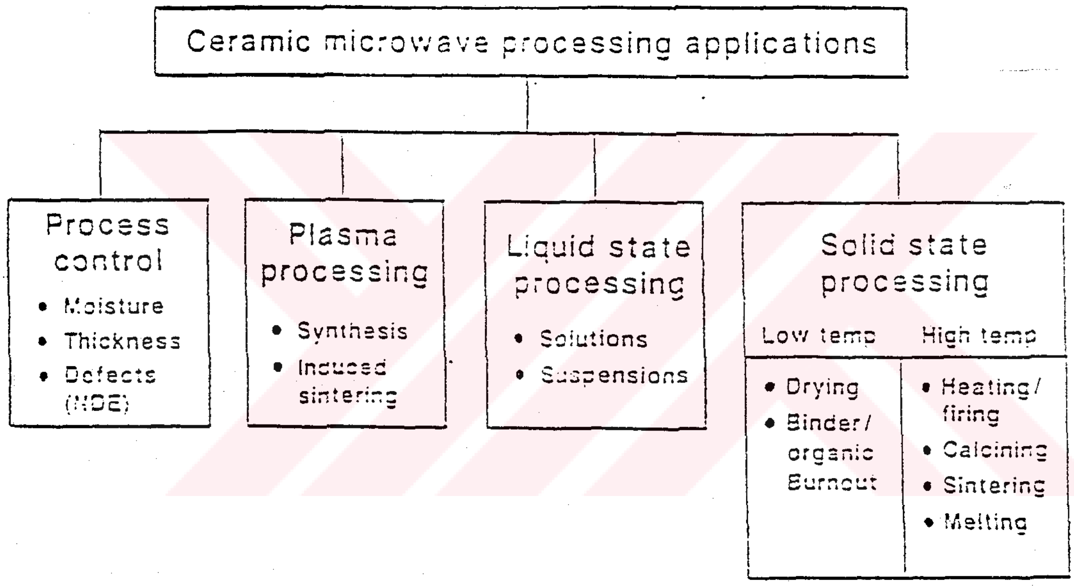


FIGURE 2.7. Application of microwave energy to process ceramics (after Sutton, 1989), [10].

shown that while the rapid heating caused by microwaves offers exceptional densification and grain growth rates, it can also lead to cracking and other undesirable qualities. In most studies, standard 700-W 2.45-GHz magnetrons have been used as the microwave source. The more powerful microwave source may offer benefits in regard to more uniform heating of larger ceramic pieces and greater coupling efficiency [13].

A preliminary investigation of microwave sintering of ceramics indicates that the new technology will not lead to reductions in the use of energy to manufacture ceramic materials. However, if the adoption of the new process leads to higher quality ceramic parts or if the process allows the costs of non energy inputs to be reduced, new technologies based on advanced ceramics may become feasible and in turn lead to significant energy savings.

The question of economic feasibility remains elusive. The adoption of microwave sintering solely on the basis of savings in energy costs does not appears to be promising. Economic feasibility will depend on the effects that microwave sintering will have on other process steps, eg., materials preparation, forming, and finishing. Small changes in the non energy inputs in all processing steps could easily overcome higher energy costs. Furthermore, a small change in yield, especially at the latter processing steps such as sintering, would be particularly valuable. Economic feasibility will also depend on the improved material properties that may result from microwave sintering.

2.2.5.2. Microwave Joining of Ceramics

There are several potential benefits of using microwave energy to join ceramics, all of which derive from the fact that the electromagnetic field penetrates the material, heating it internally. This leads to a rapid temperature rise, since one does not have to wait for the heat to be conducted from the surface to the interior. Extremely rapid heating to joining temperatures can be achieved with modest microwave power (typically a few minutes at 100 W). In addition, entirely new methods of controlling the joining process are possible. For example, the microwave energy may be focused on the joint region through design of the resonant cavity applicator and the sample configuration, or by seeding the materials to be joined with microwave absorbers.

There have been recent accomplishments in the joining of ceramics using microwave energy. These accomplishments include joints made in 3 to 10 min using about 100 W of power and pressures of 0.6 to 9 Mpa in mullite, alumina, and silicon nitride, both with and without an intermediary material (flux), with room temperature strength equal to that of the unjoined material; strength retention for the joined material, in cases where it has been measured, matches that of the unjoined material up to 800^0C. In addition, a method for performing in situ acoustic nondestructive evaluation (NDE) of the joint as it is being formed has been developed [14].

Microwave processing has been applied to an extremely broad range of ceramic materials, from earthenware to superconductors. It has been used for drying, binder burnout, melting, calcining, firing, and sintering. The current emphasis is on sintering. Improved product of structural ceramics, as judged by higher densification, has been achieved in shorter times and at lower temperatures than with conventional methods.

Substantial effort has been devoted to cavity design to optimise heating profiles. With careful attention to the shape and dimensions of the cavity and the placement of the samples and other loads, materials with a range of dielectric properties, from highly lossy insulators to conductors, have been heated. Diagnostic instrumentation has been developed side by side with cavity design. Microwave processing equipment currently in use often incorporates the ability to measure or monitor any or all of the following parameters during processing: electric field strength within the cavity; incident and reflected microwave power; sample surface temperature; sample dimensional change; applied pressure across the sample; and acoustic response of the sample.

It is clear, therefore, that microwave heating technology has developed to the stage where (1) it is broadly applicable to ceramic materials; (2) it is robust with respect to the dielectric properties of the materials; (3) improvements over product quality obtained by conventional heating methods are possible; (4) a variety of applicator designs are possible; and (5) a range of sophisticated diagnostic technics are available, including the potential for on-line process control. In addition, microwave heating is highly compatible with conventional heating approaches, permitting development of hybrid systems.

2.2.5.3. Microwave Processing of Earthenware Ceramics

It is known that the power absorbed by a material exposed to microwaves is proportional to its dielectric loss factor. It can simply be written as

$$P = 2\pi f \varepsilon_0 \varepsilon'' E^2 \qquad (2.5)$$

where P is the power absorbed per unit volume, f the frequency, ε_0 the permittivity of free space, E the internal electric field, and the ε'' dielectric loss factor.

It has been shown that, for ceramic materials, this dielectric loss factor can be highly temperature dependent following a curve as shown in Figure 2.8; thus, the heating effect is subject to the phenomenon of thermal runaway, because the dielectric loss factor starts to increase very rapidly with temperature [15]. T_c in the diagram shows where thermal runaway would start to occur. If the thermal runaway becomes localised within the body, then highly uneven firing can take place within the product; however, if this excessive temperature rise can be detected, then a control system can be introduced to reduce the input power at the relevant time and produce a controllable and repeatable firing schedule.

The microwave source used is a 1.5-kW, 2450-Mhz, air-cooled magnetron coupled to a multimedia cavity via a water-cooled waveguide. The outside of the cavity shell is also water cooled to prevent overheating and possible distortion of the cavity.

The basic material used in the study was a traditional earthenware body. The nominal body composition was ball clay, 25%; china clay, 25%; calcined flint, 35%; and feldspar, 15%.

The time-temperature curves of each of the materials have been considered to attempt to elucidate the mechanism by which the body as a whole absorbs energy (Figure 2.9).

The plot for the earthenware body (Figure 2.9) shows an exponential type of curve, similar to that proposed previously, with the temperature increasing more rapidly above 800^0C, indicating that may be the mechanism responsible for high-temperature heating is not the same as that for low temperatures.

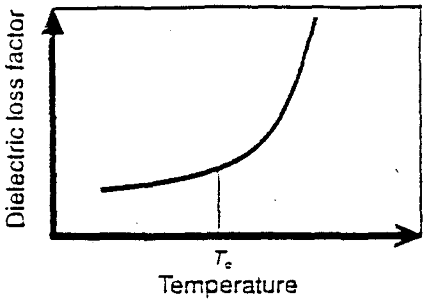


FIGURE 2.8. Typical variation of dielectric loss with temperature (after Hamlyn and Bowden, 1990), [15].

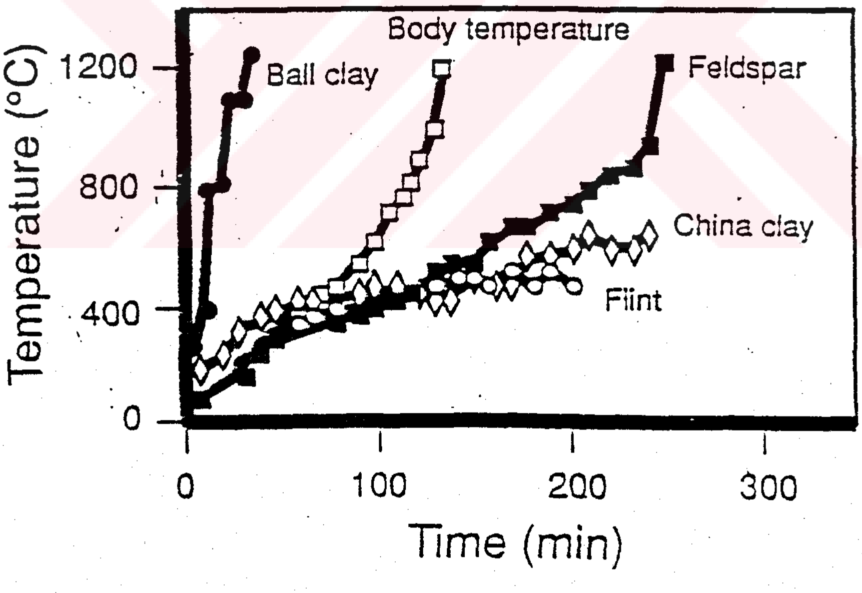


FIGURE 2.9. Variation of temperature with time for all materials (after Hamlyn and Bowden, 1990), [15].

China clay exhibited an initial rise in temperature which then slowed down and reached a plateau between 500^{0}C and 600^{0}C. The ball clay shows a very rapid increase in temperature with time over the entire firing range. Calcined flint showed little response to the microwave energy, and the temperature rise recorded could be due to the heating of the samples by conduction from the refractory materials present in the cavity insulation. The feldspar exhibited behaviour consisting of a slow, but linear, heating rate until about 850^{0}C, when the gradient became much steeper. This suggests that the dominant mechanism of absorption at higher temperatures is different from that at low temperatures.

From these initial trials, it is concluded that earthenware ceramic materials are susceptible to microwave energy and that it may be possible to process them in this manner. The mechanism of absorption of energy is not simple because it varies significantly for the individual raw materials. However, it has been shown that one of the dominant mechanism is the absorption of the energy by the ball clay, which operates over all of the temperature range. At low temperatures absorption is due to carbonaceous material being present, but at higher temperatures absorption is not yet explained. Another mechanism is the high temperature absorption by feldspar of microwaves, which is explained as being due to increase in electrical conductivity of the material as it melts and, therefore, an increase in ionic conduction losses.

2.2.6. Design Principles for Microwave Heating and Sintering

Microwave heating equations are shown to have a complex functional dependence on material and process parameters such as composition, density, temperature, frequency, and geometry. Power flow and its deposition in the presence of simple material shapes are used to illustrate relevant electromagnetic boundary conditions. Various types of applicator design using standing wave and travelling wave structures are discussed with respect to the results on power density, process efficiency and controllability. The problem of measuring and controlling the temperature inside a microwave system is addressed with emphasis on the use of thermocouples and infrared thermometry. Some of the problems peculiar to high temperature processing are considered [16].

Some of the main microwave energy advantages are: 1. instant on-off power control; 2. no thermal inertia; 3. the heat required is generated within the material to be processed; 4. energy focussing is possible via applicator and product geometry; 5. heat generation can even be made to be selective via the material composition.

Disadvantages of microwave energy are: 1. the high cost of capital equipment; 2. the custom design requirements of some of the hardware; 3. the great variation in the electromagnetic properties of process materials.

The mechanisms responsible for the microwave's internal heat generation capability are many, some of which are: ionic conduction, dipole orientation, various magnetic losses, interphase polarisation, molecule twisting and bending, and a number of resonance phenomena. The first two on this list are the most important and dominant mechanisms in many electromagnetic heating processes since they occur at the lower industrial, scientific, and medical frequencies for which adequate power sources are available.

2.2.7. Microwave Material Properties

Basic interaction mechanisms are shown to depend strongly on the dielectric and magnetic properties of a process material. This causes a strong dependence of power absorption on frequency, material particle size, shape, temperature, and density. Sintering dynamics cause the microstructure of the treated material to change resulting in a change in microwave heating uniformity and rate. Microwave power, by its very nature, gives better heating control and efficiency and provides internal heat to aid the material transport during sintering. No inherent temperature limit exists for microwave sintering although refractory materials used to contain the process material create an artificial upper limit. It is shown that very high (1500-2000^{0}C) temperatures in small samples can be readily achieved using commercial microwave ovens if appropriate microwave transparent sample holders are used.

Microwave energy, when interacting with many ceramic materials, creates heating within the material through the interaction of the electromagnetic force fields with the materials' molecular and electronic structure. As a result, microwave energy sources have low thermal inertia and, by appropriate design of the microwave-material interface, energy deposition is both predictable and controllable. Ionic conduction and dipole orientation are the major mechanisms causing microwave heating at the lower industrial, scientific and medical frequencies for which commercial power sources are available.

2.2.7.1. Dielectric Mixture Theory

Practical ceramic materials consist of mixtures of various particles with different shapes. The immediate electromagnetic consequence of this is that the electromagnetic waves come across a variety of microscopic boundary conditions which affect the local field values. Methods of how to calculate the localised microwave field strengths and the resultant bulk dielectric constant are found in dielectric mixture theory.

In multiphase dielectric mixture theory, the underlying assumption is that the mixture can be represented by a macroscopic dielectric constant which is a function of the included material and their geometries. In the generalized multiphase dielectric mixture theory by Tinga [13], equations are given to predict the internal fields in confocal ellipsoidal inclusions which contain the special cases of spherical shells, ellipsoids, spheres, disks, and needles, shapes typically found in sintering mixtures. Implicit in the solution are the first-order effects of neighbouring inclusions and, therefore, the high inclusion-density limit is approached correctly. Figure 2.10 shows an idealized multiphase mixture. By this choice, the problem reduces to one in which the mixture dielectric constant is determined in a self-consistent manner with the internal fields as indicated in Figure 2.11.

In theoretical modelling work on predicting high-temperature dielectric properties and field strengths, the above multiphase mixture theory may prove useful since it produces more realistic upper and lower bounds and yields a closed-form solution to the mixture problem.

Inconclusive evidence from microwave sintering experiments suggests that preferential heating of grain boundaries occurs. The first order shell mixture model can be used to predict such behavior.

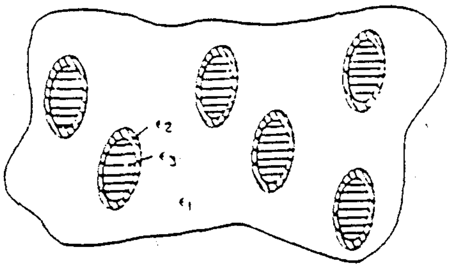


FIGURE 2.10. Multiphase mixture (after Tinga, 1988), [13].

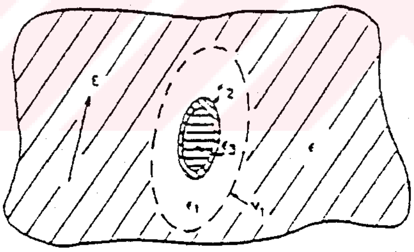


FIGURE 2.11. Homogeneous confocal ellipsoidal shell in homogeneous medium (after Tinga, 1988), [13].

2.2.7.2. Temperature and Frequency Dependence of Microwave Material Properties

Temperature and frequency dependence of the dielectric constant are related, and for relaxation type of absorption mechanisms can be shown to be inverse functions. Therefore, if the frequency dependence of a material's dielectric constant is known, then the temperature variation trend for that material can be estimated. Figure 2.12 illustrates this fact. As temperature increases, the relaxation peak of tanδ shifts to higher frequencies.

Even though large variations in the actual value of dielectric constant and losses occur for different ceramics and oxides, the general trend for many of them seems to be similar. At the commercial microwave heating frequency of 2450 Mhz, the loss factor increases with increasing temperature for many ceramics.

Conductivity, which is proportional to mobility, is strongly dependent on temperature. From comparisons between different ceramic materials, it is apparent that impurities significantly affect the conductivity and the dielectric loss factor. Impurities often enhance the conduction mechanism by reducing energy gap between the valance and conduction bands. As a matter of fact, the introduction of impurities can be used to increase the conductivity and loss factor to the desired level in some cases. Note, however, that impurities may also affect the final ceramic quality. The power penetration depth ($d=1/2\alpha$) must, unfortunately, decrease as the losses go up, opposing the goal of uniform heating. On the other hand, thermal conductivity decreases with temperature and will play a significant role in the heating profile obtained.

Because the heating rate tends to increase with temperature for ceramics such as alumina, it is necessary to limit the power available to the process so as to prevent run-away heating. The dielectric curves also show the need for preheating before microwave treatment, since over the lower temperature range many of the ceramics exhibit relatively low microwave loss which often will rise dramatically at higher temperature levels. Sintering aids which heat well at the low temperatures and burn off before attaining final sintering may be a solution to the preheating problem. It is therefore that a reasonable amount of information about the variation of the dielectric properties with temperature needs to be available before a microwave heating process can be designed effectively.

There is no doubt that microwave sintering shows promise as a useful processing method for high density ceramics. A number of individual experiments have shown that

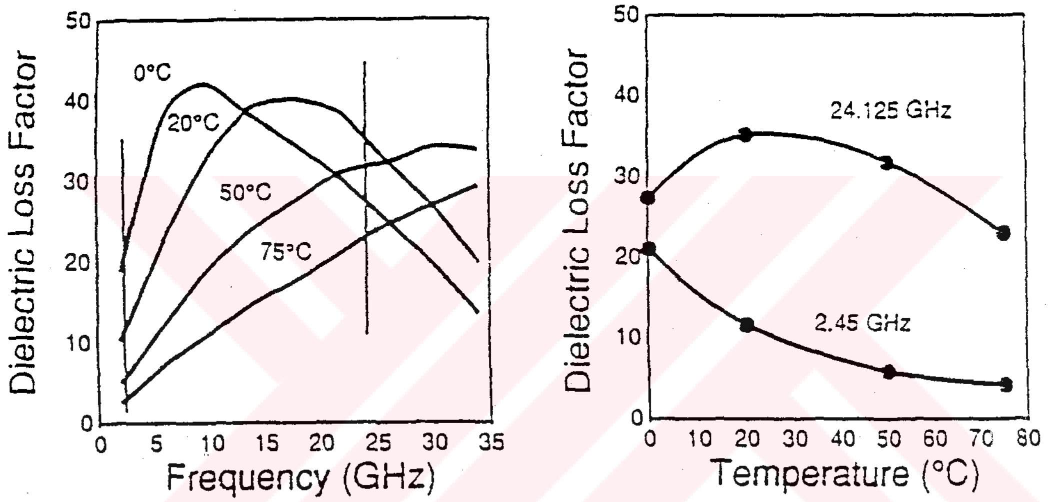


FIGURE 2.12. Effect of frequency and temperature on dielectric loss factor of water (after Sutton, 1991), [11].

microwave sintering can give improved results over conventional methods. Nevertheless, much more work needs to be done in the area of process modelling and combination heating to adequately predict and control the material's temperature and hence its final sintered quality.

2.2.8. Water Content Determination Using Microwaves

The microwave oven offers a viable means for rapidly obtaining the moisture content of soils. Data indicate that results obtained by the standard method and by incremental drying with a microwave oven could reasonably be expected to agree within ±1 per cent for most soil types.

The moisture (water) content, w, of a soil is defined as the ratio, expressed as a percentage, of the mass of water in the soil, w_w, to the dry mass of the soil, w_s:

$$w = (w_w/w_s)100 \tag{2.6}$$

The standard method for determining this value involves drying the soil in an oven at a temperature of 110 ± 5^0C until a constant mass is achieved; in most cases overnight is sufficient to get a constant mass.

The moisture content of a soil is not an absolute quantity, but is dependent on the temperature at which the soil is dried. The difference in moisture content that can be obtained by subjecting a soil to a temperature greater or lesser than that specified in the standard method varies with the type of soil. The main opposition to using the microwave oven has been the belief that it will overheat the soil and yield the results higher than those obtained by the standard method. In addition to the effect on the measured moisture content of a soil, the drying temperature may alter other soil properties such as gradation, plasticity, specific gravity, or compaction characteristics.

2.2.9. Physico-Chemical Properties of Kaolinite Mineral

Dielectric constant is determined by using Impedance/Gain-Phase Analyzer in the laboratory. It changes with the chemical combination and crystalline structure of the clay mineral. It depends on the frequency and decreases as frequency increase. Dielectric constant of a dry clay is between 2-10.

Kaolinite mineral has a two layer structure which is the combination of silica and aluminium layers (1:1). These layers are connected with hydrogen bonds. Hydrogen bonds prevent the water molecules to enter into the layers. Therefore, kaolinite does not loose its stability when mixed with water. It has a low cation exchange capacity due to low isomorphous substitution. Table 2.5 shows the characteristics of kaolinite [17].

TABLE 2.5. Physico-Chemical Characteristics of Kaolinite

Liquid limit (%)	64
Plastic limit (%)	34
Plasticity index (%)	30
Specific surface area (m^2/g)	10
Cation exchange capacity (meq/100 g clay)	3
Dielectric constant (1 MHz)	5

Dielectric constant of clay minerals decreases as frequency increases and the value of it is around 10.

2.2.9.1. Clay-water Electrolyte System

When two particles are brought into close proximity, their respective force fields begin to overlap and may influence the behavior of the system, if the magnitude of these forces are large relative to the weights of the particles themselves. Clay particles, because of their small size and large surface area, are well known to be susceptible to such effects.

An increase in temperature causes an increase in double layer thickness and a decrease in surface potential for a constant surface charge all other factors constant. However, an increase in temperature results in decrease in the dielectric constant. The following table shows the nature of the variation for water.

TABLE 2.6. Variation of dielectric constant with temperature for water.

T (^{0}C)	Dielectric constant (D)
0	88
20	80
25	78.5
60	66

The small variation of the product DT with change in temperature means, theoretically, that the double layer should not be influenced greatly. This means, of course, that the values of dielectric constant are unaffected by particle surface forces and ion concentration. It also accounts, in part, for contradictory findings reported in the literature that the effects of temperature change on such soil properties as strength, compressibility, and swelling.

2.2.9.2. Interaction between Water and Clay Minerals

Engineering properties, such as creep behavior, strength, and thermal properties of frozen and thawed soils are temperature dependent. When the temperature drops below freezing, the phase composition of water in fine-grained soils changes accordingly. Part of the available water turns into ice, whereas the remains stays as supercooled water. The amount of unfrozen water in a frozen soil, its nature, and its equilibrium side by side with ice depends primarily on temperature, mineralogy and particle gradation, water content, and molding conditions of the wet soils. Many theories have been formulated regarding the mechanisms responsible for the presence of unfrozen water in frozen soils. Williams states that capillarity and suction properties of the soil are the cause of the unfrozen water. Others explain it in terms of the oriented water structure. The least structured water freezes first.

Additional cooling freezes lesser amount of water. A film of water, although very thin, exists at the ice-particle interface at very low temperatures. This film, in particular, and the frozen water, in general, seem to have significant effect on stress-strain behavior and strain-time behavior of frozen soils [8].

Clay minerals are very tiny crystalline substances evolved primarily from chemical weathering of certain rock-forming minerals. Chemically, they are hydrous aluminosilicate plus other metallic ions. All clay minerals are very small, colloidal-sized crystals and they can only be seen with an electron microscope. The individual crystals look like tiny plates or flakes, and from X-ray diffraction studies, scientists determined that these flakes consist of many crystal sheets, the tetrahedral or silica, and the octahedral or alumina sheets. The particular way in which these sheets are stacked, together with different bonding and different metallic ions in the crystal lattice, constitute the different clay minerals.

Kaolinite consists basically of repeating layers of one tetrahedral sheet and one octahedral sheet. Because of stacking of one layer of each of the two basic sheets, kaolinite is called a 1:1 clay mineral. The two sheets are held together in such a way that the tips of the silica sheet and one of the layers of the octahedral sheet form a single layer. This layer is about 0.72 μm thick and extends indefinitely in the other two directions. A kaolinite crystal, then, consists of a stack of several layers of the basic 0.72 μm layer. Successive layers of the basic layer are held together by hydrogen bonds between the hydroxyls of the octahedral sheet and the oxygen of the tetrahedral sheet. Since the hydrogen bond is very strong, it prevents hydration and allows the layers stack up to make a rather large crystal. A typical kaolin crystal can be 70 to 100 layers thick.

Water usually does not have much effect on the behavior of granular soils but on the other hand, fine-grained soils, especially clay soils, are strongly influenced by the presence of water. The variation of water content give rise to plasticity, and the Atterberg limits are an indication of this influence. Kaolinite, the largest clay mineral, has a thickness of edge dimension of about 1 mm.

Clay minerals are almost hydrated in nature; that is, these layers of water surrounding each crystal of clay. This water is called absorbed water. Water is a dipolar molecule. Even though water is electrically neutral, it has two separate centers of charge, one positive and one negative. Thus the water molecule is electrostatically attracted to the surface of the crystal. Secondly, water is held to the clay crystal by hydrogen bonding. The third factor is that the negatively charged clay surface also attracts cations present in the water. Since all cations are hydrated to some extend, depending on the ion, cations also contribute to the attraction of water to the clay surface.

The attraction of water to the clay surface is very strong near the surface and diminishes with distance from that surface. The water molecules right at the surface are very tightly held and strongly oriented. Measurements show that some thermodynamic and electrical properties of the water next to the clay surface are different than that of 'free water'.

The source of the negative charge at the surface of the clay crystal results from both isomorphous substitution and imperfections in the crystal lattice, especially at the surface. Broken edge contribute greatly to unsatisfied valance charges at the edges of the crystal. Since the crystal wants to be electrically neutral, cations in the water may be strongly attracted to the clay, depending on the amount of negative charge present. Different clays have different charge deficiencies and thus have different tendencies to attract the exchangeable cations. They are called exchangeable since one cation can easily be exchanged with one of the same valance or by two of one-half the valance of the original cation.

Calcium and magnesium are the predominant exchangeable cations in soils, whereas potassium and sodium are less common. Aluminium and hydrogen are common in acidic soils. The depositional environment as well as subsequent weathering and leaching will govern which ions are present in a particular soil deposit. Cation exchange or replacement is further complicated by the presence of organic matter. The ease of replacement or exchange of cations depends on several factors, primarily the valance of the cation. Higher valance cations are easily replace cations of lower valance. For ions of the same valance, the size of the hydrated ion become important; larger the ion, the greater the replacement power. There are several practical consequences of ion exchange. The use of chemicals to stabilize or strengthen soils is possible because of ion exchange.

2.2.10. Future Growth & Applications in Microwave Processing

Table 2.7 lists several of the issues (barriers and challenges) that will have an impact on the future developments and growth of microwave processing [9].

TABLE 2.7. Key Issues: Future Growth & Applications in Microwave Processing [11].

Experience
*New process, new challenges
*Overexpectations
*Reluctance

Communication
*Equipment supplier/customer
*Total system approach

Fundamentals
*Complex nature of process
*Multidisciplines
*Microwave - material interactions
*Dielectric property measurements

Equipment
*Selection of proper equipment
*Custom designed applicators
*Temperature & process change feedback
*Controls, automation

Economics
*Accurate cost and market analyses
*Unique microwave benefits
*Detailed knowledge of microwave/ materials processing
*Low cost alternatives

Safety & Environmental Regulations

Creative & Innovative Developments
*New equipment & controls
*New process applications
*Intelligent processing
*Synthesis of new materials & structures
*Solutions to new problems

Further investigation is needed to develop maps of the regimes of microwave-power absorption characteristics, batch size, heating rate, and other variables where microwave processing can be reproducible and uniform. This would allow processors to make informed decisions concerning microwave applications and process and equipment selection, while avoiding inefficient heating, uneven heating, and thermal runaway problems that have plagued earlier attempts.

Specific processes that show promise for future development include:

- ceramic processes including drying, chemical vapor infiltration, reaction bonding of silicon nitride, powder synthesis, and joining;
- polymeric composite pultrusion, ultradrawing of polymeric fibers, and adhesive bonding with intrinsically conducting organic polymers;
- chemical processes, including custom organic synthesis, hazardous materials processing, solvent extraction, and drying; and
- industrial waste processing, including treatment or remediation of process wastes, detoxification or consolidation of stored waste, and cleanup of storage or disposal sites.

In general, the elements required for successful application of microwave processing to industrial materials include selection of materials amenable to microwave processing; an understanding of the process requirements; an understanding of the process economics; characterization of material thermochemical properties; selection of equipment and design of applicators suitable for the application; an understanding of how the parts to be processed will interact with the microwave field; and adequate measurement and control of process variables such as incident power, part temperature, and field strength.

III. METHODOLOGY

3.1. Materials

Three types of materials are used for the preparation of the test samples: kaolinite, bentonite, and rubber. The properties of these materials are given below.

3.1.1. Kaolinite

The clay used in this study is ceramic grade kaolin clay (K403), which is purchased from Doğa Madencilik Sanayi Ticaret A.Ş., Eczacıbaşı, Pendik, Istanbul. It is chosen because of its uniformity, low-level swelling characteristics, and absence of thixotropic effects. The kaolin is commercially available in paper bags, and is stored in the laboratory in these bags. The geotechnical properties of the clay used are given in Table 3.1. The chemical and rational analysis of the clay are obtained from Doğa Madencilik Sanayi Ticaret A.Ş., and are given in Table 3.2.

TABLE 3.1. Geotechnical Properties of Kaolinite Used in Experiments.

INDEX PROPERTIES	
Liquid Limit, LL (%)	32.00
Plastic Limit, PL (%)	21.60
Plasticity Index, I_p	10.40
Finer than 2μ size, %	26.48
Specific Gravity	2.60
Activity	0.37
PROCTOR COMPACTION PARAMETERS	
Maximum Dry Density, kN/m^3	1.515
Optimum Water Content, %	21.00

TABLE 3.2. Chemical and Rational Analysis of Kaolinite Used.

CHEMICAL ANALYSIS (%)	
SiO_2	78.0
Al_2O_3	15.0
Fe_2O_3	0.5
TiO_2	0.3
CaO	0.2
MgO	0.1
K_2O	0.1
Na_2O	0.1
L.O.I.	5.5
RATIONAL ANALYSIS (%)	
Kaolinite	37.2
K. Feldspar	0.6
Na Feldspar	0.8
Free Quartz	59.7

3.1.2. Rubber

The rubber used in the experiments was purchased in fiber form from tire-retread industry, and stored in black plastic bags in the laboratory in order to prevent the exposure of the material to the light. It is poorly graded material with a specific gravity of 1.1.

The clay-rubber mixtures were prepared at the weight proportions as 100 per cent clay - 0 per cent rubber, and 100 per cent clay - 10 per cent rubber. The compaction curves for these mixtures are given in Figure 3.1.

3.1.3. Bentonite

The type of bentonite used in this study is a type of calcium bentonite which is known as white bentonite. The typical mineralogy, chemical and rational analysis and geotechnical properties of the bentonite which is used in the experiments are given in Table 3.3 and Table 3.4 respectively. The compaction curve of bentonite is given in Figure 3.2.

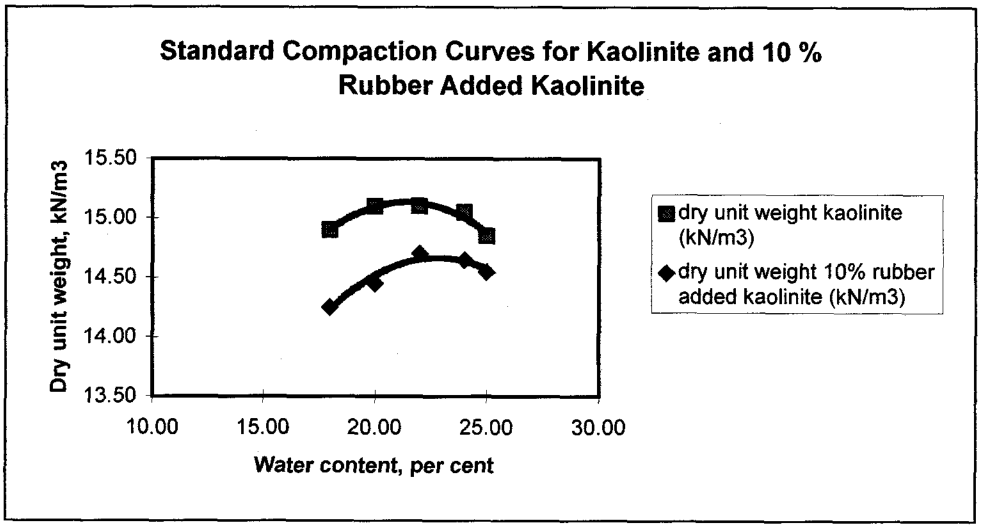


FIGURE 3.1. Compaction curves for pure kaolinite and 10 per cent rubber added kaolinite.

TABLE 3.3. Typical mineralogy, chemical and rational analysis of white bentonite.

Chemical analysis (%)	SiO_2	72.8
	Al_2O_3	14.7
	Fe_2O_3	0.7
	TiO_2	0.1
	CaO	0.6
	MgO	1.5
	Na_2O	1.8
	K_2O	0.3
	L.O.I.	7.3
Mineralogical composition (%)	a - Crystoballite	10 - 15
	Feldspar	3 - 5
	Illite	10 - 15
	Ca montmorollite	65 - 75
Particle size (%)	% > 60 m	2.0
	% < 20 m	90.7
	% < 10 m	87.4
	% < 2 m	75.0
Chemical and Physical properties	pH (6% W/W suspension)	7.5
	Swelling (ml/2 g)	2.0
	Cation exchange capacity (meq / 100 g dry weight)	60.0
	Water absorption (Westinghouse)	80.0
	Clamping test	Positive
	Whiteness (%)	87.0

TABLE 3.4. Geotechnical Properties of Bentonite Used in Experiments.

INDEX PROPERTIES	
Liquid Limit, LL (%)	93.67
Plastic Limit, PL (%)	37.15
Plasticity Index, I_p	56.52
Finer than 2μ size, %	75.00
Specific Gravity	2.14
Activity	0.75
PROCTOR COMPACTION PARAMETERS	
Maximum Dry Density, kN/m^3	1.282
Optimum Water Content, %	32.02

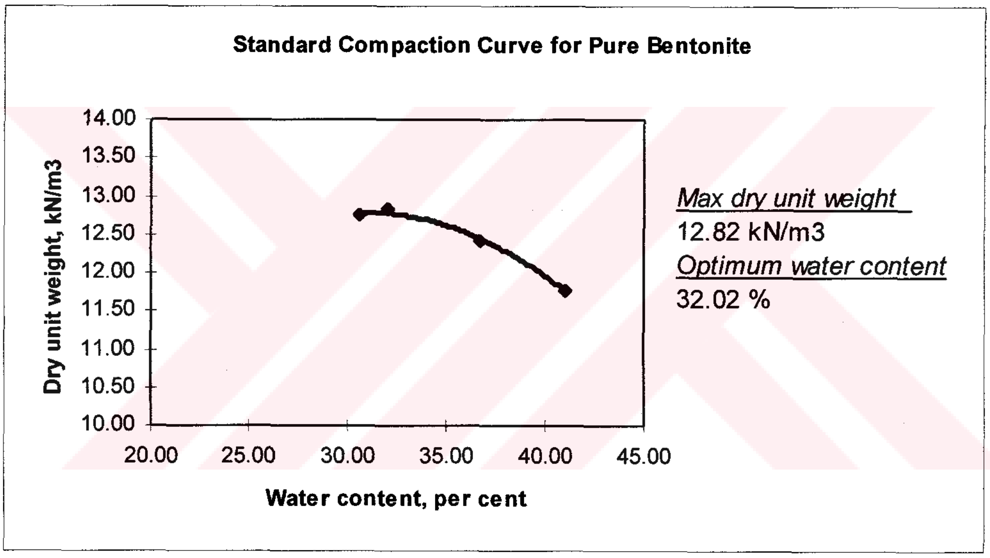


FIGURE 3.2. Compaction curve for pure bentonite.

3.2. Sample Preparation

In this study, the effect of freeze and microwave thaw on four different soils were examined: pure kaolinite, 10 per cent by weight rubber added kaolinite, pure bentonite and 40 per cent bentonite added kaolinite.

Test specimens were prepared at approximately two or three per cent wet of optimum; 24 per cent for pure kaolinite and rubber added kaolinite samples and 34 per cent for pure bentonite and kaolinite-bentonite mixtures. The compaction curves are given in Figure 3.1, and Figure 3.2. The samples were prepared on the wet side of optimum, because it was expected that wet soils would be more vulnerable to changes in soil structure caused by freeze and thaw than dry soils. Also cracking in clay would result from the formation of ice lenses where the possibility of ice lens formation was higher with the higher moisture content.

Strength tests (direct shear and unconfined compression tests), and water content determination tests were conducted. Direct shear tests and unconfined compression tests were conducted on kaolinite and 10 per cent by weight rubber added kaolinite samples whereas the other tests applied to kaolinite, bentonite and 40 per cent by weight bentonite added kaolinite samples.

The compression test samples were prepared using the Harvard miniature compaction apparatus. The volume of the mold is 84.6 cm^3 . Compaction was done in three layers with 25 blows per layer. The weight of the hammer is 0.54 kg dropping from a height of 32 cm. Compression test samples were cylindrical with dimensions of 35 mm diameter and 76 mm height.

The square shear test samples were obtained from the compacted samples by Standard Proctor Compaction procedure following the ASTM D3080 specifications. Sample dimensions were 5x5x2 cm.

Each test specimen was fully wrapped in plastic foil to minimize the loss of moisture during curing and also during the freeze and thaw cycles. For curing, the samples were stored in the humid room for a week at 21°C, at the Geotechnical Engineering

Laboratory of Boğaziçi University, so that they could develop a homogeneous distribution of moisture throughout. After being cured in the humid room for a week, the samples at their original molding water contents, were subjected to different freeze and microwave thaw cycles.

While keeping the temperature of freezing at -40°C and the energy applied during thawing constant, the duration and the number of cycles were changed.

For direct shear and unconfined compression tests, the number of cycles were five and ten with duration of two hours of freezing and seven minute thawing at medium power level and one day freezing and seven minute thawing at medium-high power level of microwave oven. Table 3.5 shows the number of samples prepared for different strength tests for different freeze and thaw cycle duration and different number of cycles.

Duration of cycles were determined after several trials. The purpose of freezing the soil is to freeze all the water present in it. To determine the time required to freeze all the pore water within the soil, samples were waited in the freezer for half an hour, an hour and two hours. After microscopic examinations, it is concluded that two hours of freezing is adequate. The purpose of thawing the soil is to melt all the frozen water in the sample without damaging its molecular structure. Thawing of duration between 5-15 minutes at different power levels of microwave oven have been tried on two hours frozen and one day frozen samples. Again by examinations in the microscope, it is concluded that seven minute thawing at medium power level is sufficient for two hours frozen samples and seven minute of thawing at medium-high power level for one day frozen samples.

TABLE 3.5. Samples prepared for strength tests.

Duration of cycles		2 hour + 7 min				1 day + 7 min			
Number of cycles	no cycle	5	5	10	10	5	5	10	10
Tests run	K & K+10%R	K	K+10%R	K	K+10%R	K	K+10%R	K	K+10%R
Unconfined compression	4	2	2	2	2	2	2	2	2
Direct shear	6	3	3	3	3	3	3	3	3

For a cycle to be completed, the test specimens were kept in the microwave oven for a thawing period and in the freezer for a freezing period. After the completion of the freeze and thaw cycles, unconfined compression and direct shear tests were run.

For water content determination, 100 gr of different soils were mixed at various moisture contents. Soil samples were pure kaolinite, pure bentonite, 10 per cent bentonite plus 90 per cent kaolinite, 20 per cent bentonite plus 80 per cent kaolinite, 30 per cent bentonite plus 70 per cent kaolinite, 40 per cent bentonite plus 60 per cent kaolinite. One set was dried by conventional heating, over night at 110°C and the other set by microwave oven. For the microwave heating, samples were dried until constant weight is achieved. The distribution of water content over time were recorded. The water contents obtained by two methods were compared.

3.3. Experimental Set-up

Unconfined compression and direct shear tests were performed in order to obtain the shear strength parameters of the kaolinite, and 10 per cent by weight rubber added kaolinite samples after the freeze and microwave thaw cycles. The unconfined compression tests were performed following the instructions of ASTM D2166. The shear tests were performed according to the procedure specified in ASTM D3080.

For the water content determination by microwave oven, there is no specification in the literature. The following method is applied. A wet soil specimen is placed in a glass container and its mass is determined. It is then placed in a microwave oven, subjected to an interval of drying, and removed from the oven and reweighed. Increments of drying and reweighing are repeated until the mass becomes nearly constant. The difference between the mass of the wet and dried specimen is used as the mass of water originally contained in the specimen. The moisture content is determined using these values. The microwave oven used is a household-type microwave oven produced by Vestel model V85147. It has input power of 1000 W with 50 MHz frequency and output power of 650 W with 2450 MHz frequency.

IV. RESULTS AND DISCUSSIONS

This section includes the results of water content determination, Atterberg limits determination, direct shear tests and unconfined compression tests. In each test group, the results obtained by microwave thawing are compared with the ones obtained by conventional thawing.

4.1. Water Content Determination

The moisture (water) content, w, of a soil is defined as the ratio, expressed as a percentage, of the mass of water in the soil, w_w, to the dry mass of the soil w_s. The standard method for determining this value involves drying the soil in an oven at a temperature of 110 ± 5°C until a constant mass is achieved; in most cases overnight (16 hours) is sufficient.

Microwave oven offers a viable means for rapidly obtaining the moisture content of soils. For water content determination, 100 gr of different soils were mixed at predetermined moisture contents. Soil samples were pure kaolinite, pure bentonite, 10 per cent bentonite plus 90 per cent kaolinite, 20 per cent bentonite plus 80 per cent kaolinite, 30 per cent bentonite plus 70 per cent kaolinite, 40 per cent bentonite plus 60 per cent kaolinite. One set was dried by conventional heating, over night at 110 ± 5°C and the other set by microwave oven. For the microwave heating, samples were dried until constant weight is achieved. The distribution of water content over time was recorded. The water contents obtained by two methods were compared.

Table 4.1 summarizes the comparison of moisture contents obtained by two methods: microwave drying and conventional oven drying. Data indicate that results obtained by the standard method and by incremental drying with a microwave oven could reasonably be expected to agree within ± 1 percent for most soil types.

TABLE 4.1. Comparison of microwave and conventional oven moisture contents.

Soil type	Mass of specimen (g)	MW drying time (min)	Moisture content (per cent)		
			Conv. w_c	Micro. w_m	Difference w_m-w_c
Pure kaolinite	100	16	22.1	21.8	-0.3
10% B + 90% K	100	15	25.4	25.1	-0.3
20% B + 80% K	100	14	27.8	28.0	0.2
30% B + 70% K	100	13	29.6	28.9	-0.7
40% B + 60% K	100	13	32.7	32.1	-0.6
Pure bentonite	100	12	40.7	40.5	-0.2

Figure 4.1 is the graphical representation of water content determination test results.

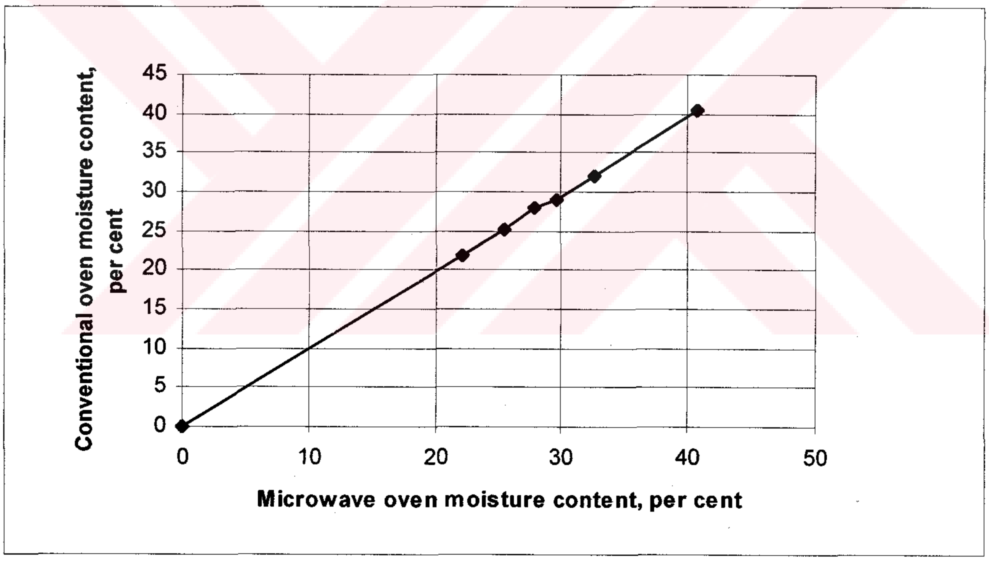


FIGURE 4.1. Comparison of microwave and conventional oven moisture contents.

The distribution of water content over time was recorded for 150 g kaolinite and bentonite samples. Samples were weighed in every two minutes until constant mass was reached. Samples' moisture content was obtained by conventional oven also. For kaolinite, the moisture content was 27.47 per cent obtained by the conventional oven and 27.18 per cent obtained by the microwave oven. For bentonite, the moisture content was 42.84 per cent and 43.17 per cent respectively. The difference is again negligible. Figure 4.2 and Table 4.2 shows this correlation.

For this case, measurement of water content by using microwave worked well. A preliminary investigation similar to the one conducted in this study is recommended for other studies.

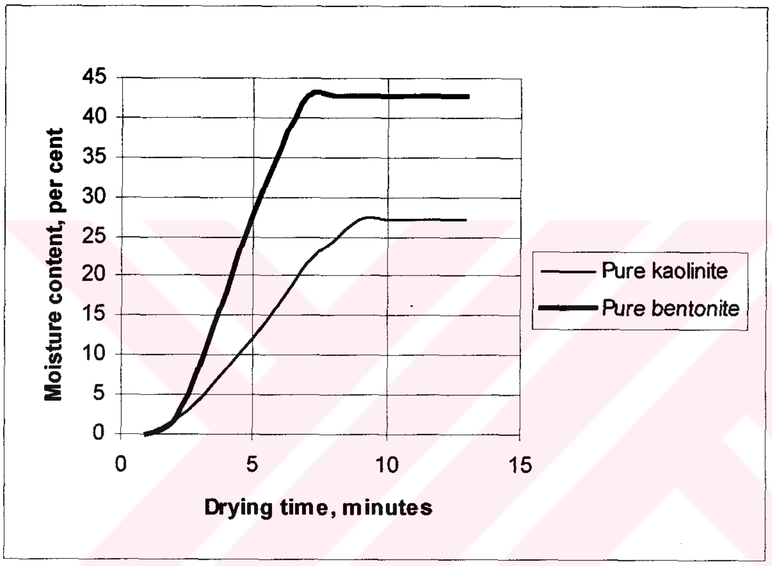


FIGURE 4.2. Moisture content vs. drying time for kaolinite and bentonite samples obtained by microwave drying.

TABLE 4.2. Distribution of moisture content over time.

	Moisture content, %	
Sample Microwave drying time (min)	Pure kaolinite	Pure bentonite
0	0.00	0.00
2	1.68	1.65
4	4.50	9.10
6	8.38	17.98
8	11.96	27.63
10	16.92	36.24
12	21.51	42.71
14	24.32	42.84
16	27.18	42.84
18	27.18	42.84
20	27.18	42.84
22	27.18	42.84
24	27.18	42.84

4.2. Direct Shear Test Results

The factors affecting the frost action in the compacted clay samples are number of cycles, duration of cycles, and the addition of rubber. Table 4.3 shows the shear strength, horizontal displacement, and strength parameters, cohesion and internal friction angle, of the samples subjected to different freeze and thaw cycles. The results obtained by microwave thawing and conventional oven thawing are compared.

As number of cycles increased from zero to five and ten, a decrease in the shear strength of the samples was measured. This reduction in shear strength is more significant for ten cycles. After ten freeze and microwave thaw cycles, pure samples' shear strength showed 5 per cent decrease and the decrease in the rubber added samples' shear strength was 7 per cent.

As duration of cycles increased from hours to days, the shear strength of the samples again decreased. Pure kaolinite samples subjected to no freeze and thaw cycle failed at 285 kPa with a horizontal displacement of 2.1 mm and 10 per cent rubber added samples failed at 290 kPa with a displacement of 2.2 mm, when subjected to 285 kPa normal stress. Pure kaolinite samples subjected to 10 daily freeze and microwave thaw cycles failed at 275 kPa horizontal stress and 2.7 mm horizontal displacement whereas the rubber added ones under the same conditions failed at 270 kPa and 2.8 mm displacement. Decrease in the shear strength was 3.5 per cent for pure samples, and 6.9 per cent for the rubber added ones.

As the number and duration of freeze and thaw cycles were increased, cohesion values of both pure kaolinite samples and rubber added kaolinite samples decreased. Figure 4.3 and Figure 4.4 shows the correlation between number of cycles and cohesion values. Cohesion of pure kaolinite subjected to no freeze and thaw cycle was 95 kPa and that of rubber added kaolinite was 97 kPa. After 10 daily cycles, cohesion of pure samples decreased to 68 kPa, by experiencing 28 per cent reduction, and that of rubber added ones to 71 kPa, by showing 27 per cent reduction. Results are shown in Table 4.4. Rubber added kaolinite samples experienced larger deformations with larger horizontal stresses and cohesion values.

Figures 4.5-4.12 shows the direct shear test results applied to pure kaolinite and 10 per cent rubber added kaolinite samples after freeze and microwave thaw cycles. Failure

TABLE 4.3.a. Comparison of direct shear test results for pure kaolinite and 10 per cent rubber added kaolinite samples subjected to freeze and thaw cycles by microwave thawing and conventional oven thawing.

Duration of cycle						2 hour freezing+7 min thawing						
Number of cycles		0				5				10			
Type of sample		pure kaolinite		kaolinite+10%rubber		pure kaolinite		kaolinite+10%rubber		pure kaolinite		kaolinite+10%rubber	
		1	2	1	2	1	2	1	2	1	2	1	2
Failure shear stress (kPa)	σ_n = 95 kPa	160	168	160	170	155	163	153	172	140	155	152	170
	σ_n = 190 kPa	215	221	232	223	210	216	249	240	192	208	208	220
	σ_n = 285 kPa	285	278	290	281	291	280	287	285	266	273	280	290
Failure horizontal displacement (mm)	σ_n = 95 kPa	1.90	1.80	2.00	1.90	2.10	2.00	2.20	2.00	2.40	2.30	2.50	2.30
	σ_n = 190 kPa	2.00	1.90	2.10	2.00	2.40	2.10	2.40	2.10	2.50	2.10	2.50	2.20
	σ_n = 285 kPa	2.10	2.00	2.20	2.10	2.60	2.20	2.60	2.20	2.70	2.30	2.60	2.30
Cohesion, c, kPa		95.0	95.0	97.3	98.0	82.7	95.0	95.7	105.0	73.3	90.0	85.3	98.0
Internal friction angle, Φ		33.3	31.0	34.4	32.0	35.6	33.0	35.2	35.0	33.6	35.0	34.0	33.0

1 Microwave thawing
2 Conventional oven thawing [18]

TABLE 4.3.b. Comparison of direct shear test results for pure kaolinite and 10 per cent rubber added kaolinite samples subjected to freeze and thaw cycles by microwave thawing and conventional oven thawing

Duration of cycle		1 day freezing+7 min thawing							
Number of cycles		5				10			
Type of sample		pure kaolinite		kaolinite+10%rubber		pure kaolinite		kaolinite+10%rubber	
		1	2	1	2	1	2	1	2
Failure shear stress (kPa)	σ_n = 95 kPa	138	145	145	155	148	145	138	140
	σ_n = 190 kPa	196	195	182	210	161	198	200	200
	σ_n = 285 kPa	271	255	256	280	275	272	270	270
Failure horizontal displacement (mm)	σ_n = 95 kPa	2.30	2.10	2.40	2.20	2.50	2.20	2.50	2.10
	σ_n = 190 kPa	2.50	2.30	2.50	2.30	2.60	2.30	2.70	2.30
	σ_n = 285 kPa	2.70	2.40	2.80	2.40	2.70	2.40	2.80	2.40
Cohesion, c, kPa		68.7	82.0	83.3	88.0	67.7	70.0	70.7	76.0
Internal friction angle, Φ		35.0	31.0	30.3	35.0	33.8	34.0	34.8	36.0

1 Microwave thawing
2 Conventional oven thawing [18]

shear strength, failure horizontal displacement, cohesion and internal friction angle values obtained after hourly and daily freeze and microwave thaw cycles are plotted.

Direct shear test results for pure kaolinite and 10 per cent rubber added kaolinite samples after freeze and microwave thaw cycles and after freeze and conventional oven thawing cycles are compared. Figures 4.13-4.28 show the comparison of failure horizontal stress, failure horizontal displacement, cohesion and internal friction angle values obtained by two methods.

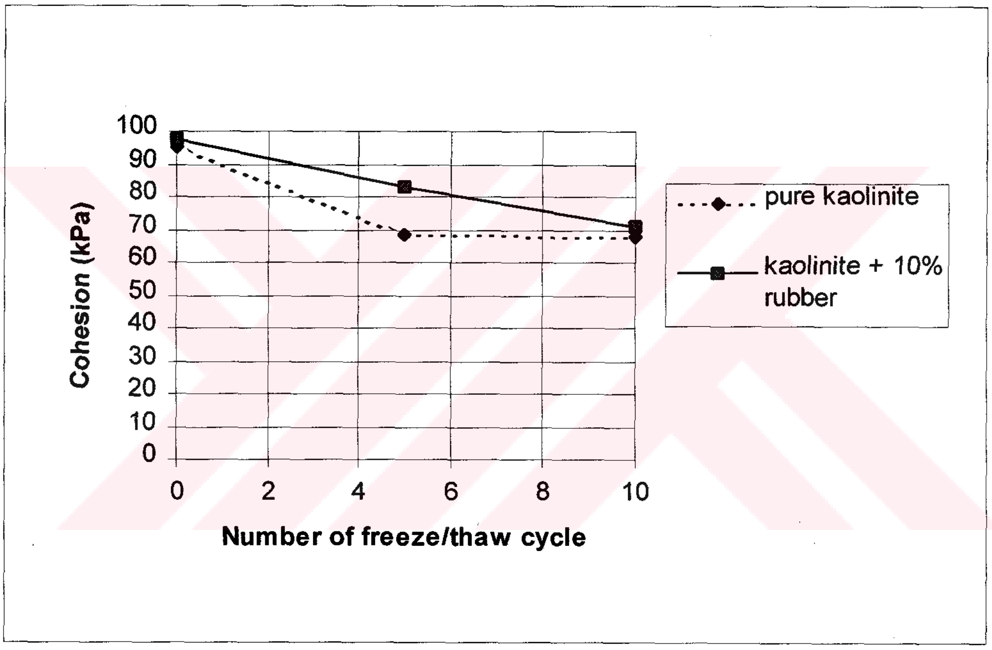


FIGURE 4.3. Cohesion values obtained from direct shear tests versus number of freeze and thaw cycles (daily cycles).

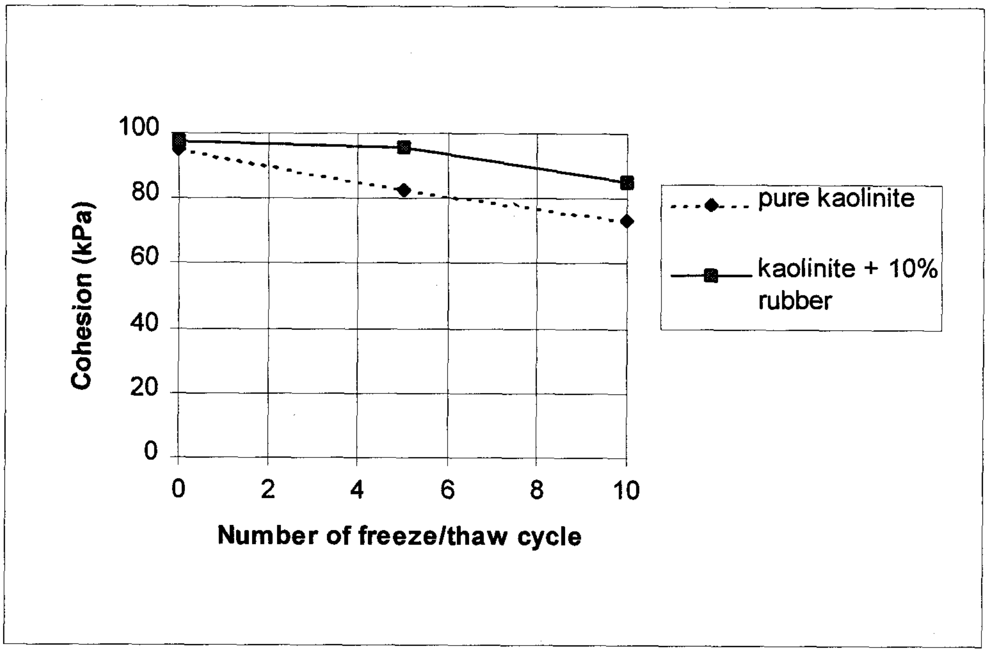


FIGURE 4.4. Cohesion values obtained from direct shear tests versus number of freeze and thaw cycles (hourly cycles).

TABLE 4.4. Comparison of cohesion with respect to number and duration of cycle.

	Cohesion (kPa)	
Number and duration of cycle	pure kaolinite	kaolinite + 10% rubber
0	95.0	97.3
5 x (2 hour freezing+ 7 min microwave thawing)	82.7	95.7
10 x (2 hour freezing+ 7 min microwave thawing)	73.3	85.3
5 x (1 day freezing+ 7 min microwave thawing)	68.7	83.3
10 x (1 day freezing+ 7 min microwave thawing)	67.7	70.7

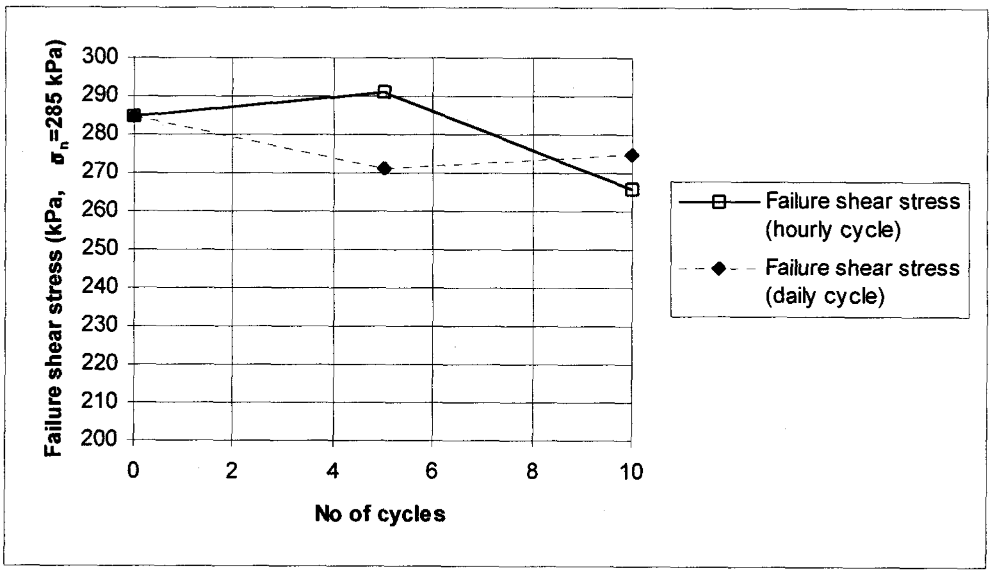


FIGURE 4.5. Comparison of failure shear stresses for pure kaolinite obtained by direct shear tests.

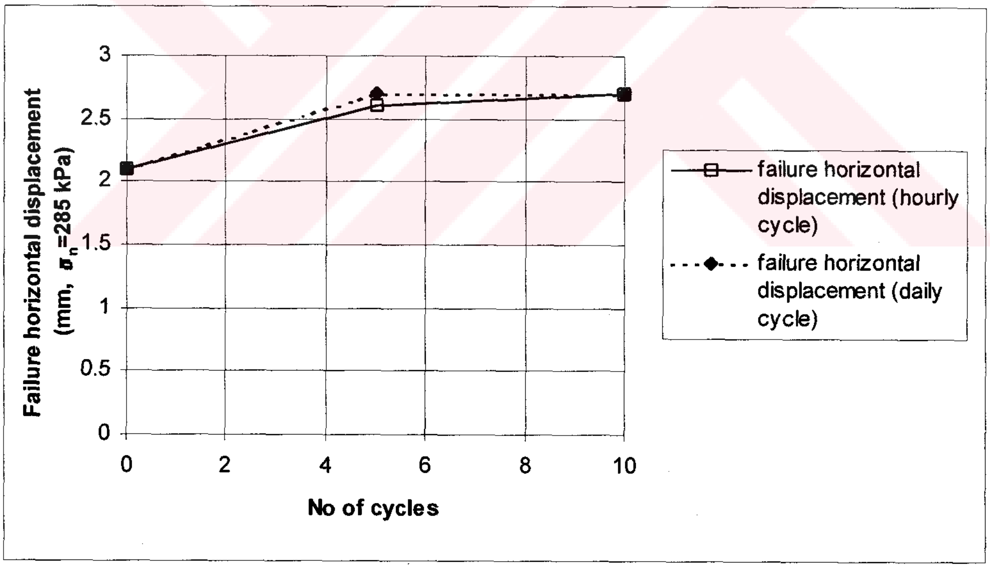


FIGURE 4.6. Comparison of failure horizontal displacements for pure kaolinite obtained by direct shear tests.

The shear strength of pure kaolinite was 285 kPa before application of freeze thaw cycles. After five daily cycles, one day freezing and seven minutes microwave thawing, it decreased to 271 kPa. Application of five more daily cycles did not make any significant change. For the kaolinite samples subjected to hourly cycles, two hours freezing and seven minute thawing, shear strength increased to 291 kPa after five cycles and decreased to 266 kPa after ten cycles (Figure 4.5). Kaolinite samples experienced a horizontal displacement of 2.7 mm after subjected to both ten daily and hourly freeze and microwave thaw cycles.

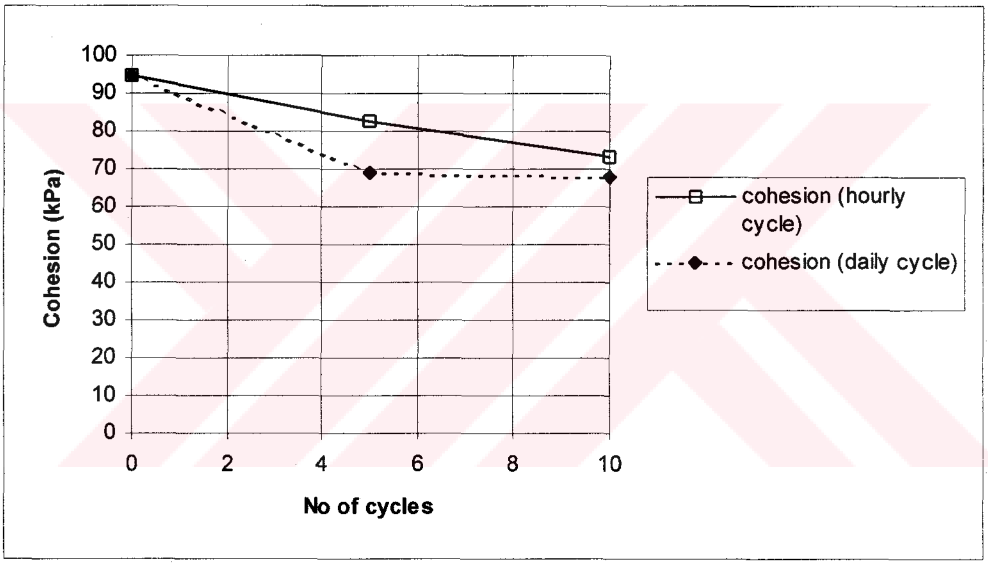


FIGURE 4.7. Comparison of cohesion values for pure kaolinite obtained by direct shear tests.

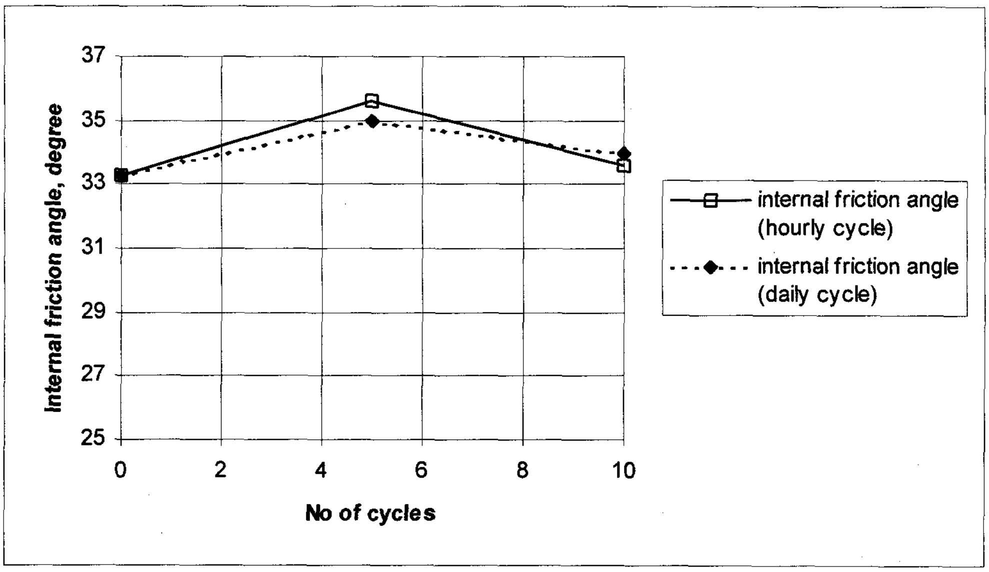


FIGURE 4.8. Comparison of internal friction angles for pure kaolinite obtained by direct shear tests.

Cohesion of pure kaolinite was 95 kPa before application of freeze microwave thaw cycles. It decreased to 83 kPa after five hourly cycles were applied and to 69 kPa after five daily cycles were applied. Cohesion of pure kaolinite decreased to 73 kPa after ten hourly cycles were applied and to 68 kPa after ten daily cycles were applied. The reduction in cohesion was 28.7 per cent at most (Figure 4.7). The internal friction angle was 33 degrees before application of cycles. It increased to 34 degrees after ten cycles were applied which is not of much importance (Figure 4.8).

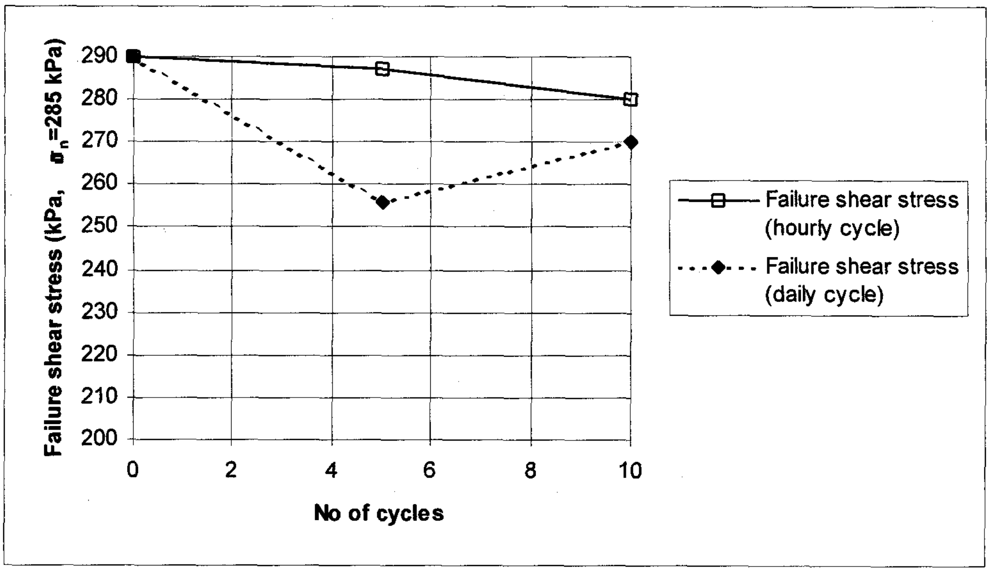


FIGURE 4.9. Comparison of failure shear stresses for 10 % rubber added kaolinite obtained by direct shear tests.

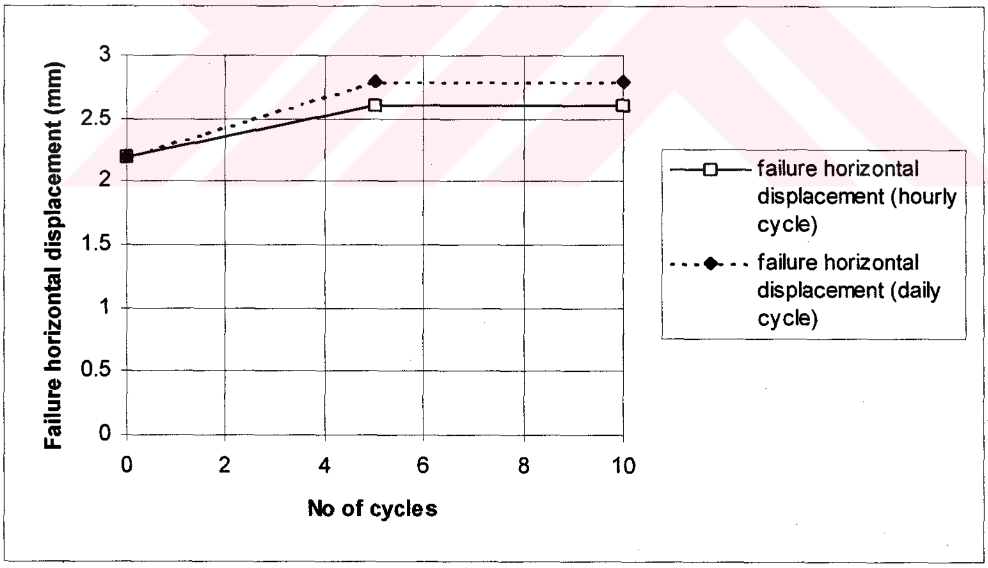


FIGURE 4.10. Comparison of failure horizontal displacements for10 % rubber added kaolinite obtained by direct shear tests.

The shear strength of ten per cent rubber added kaolinite was 290 kPa before application of freeze thaw cycles. Application of five hourly cycles, two hours freezing and seven minutes microwave thawing, did not make any significant change in shear strength. After five daily cycles were applied, shear strength decreased to 256 kPa, and it increased to 270 kPa by application of five more cycles. Reduction in shear strength was 6.9 per cent. After application of cycles, samples experienced more ductile failure. The horizontal displacement at failure was 2.8 mm after ten daily cycles (Figures 4.9, 4.10).

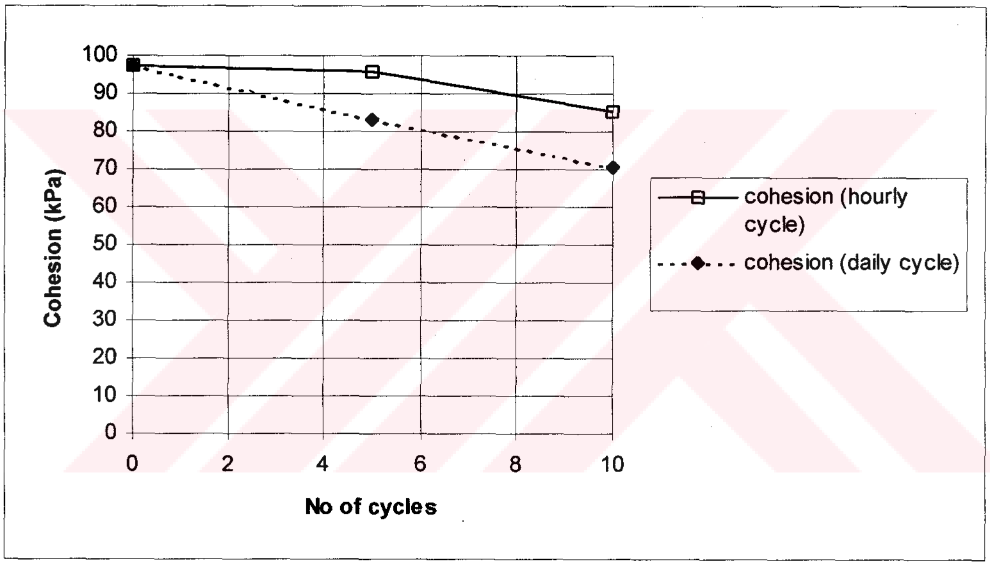


FIGURE 4.11. Comparison of cohesion values for 10 % rubber added kaolinite obtained by direct shear tests.

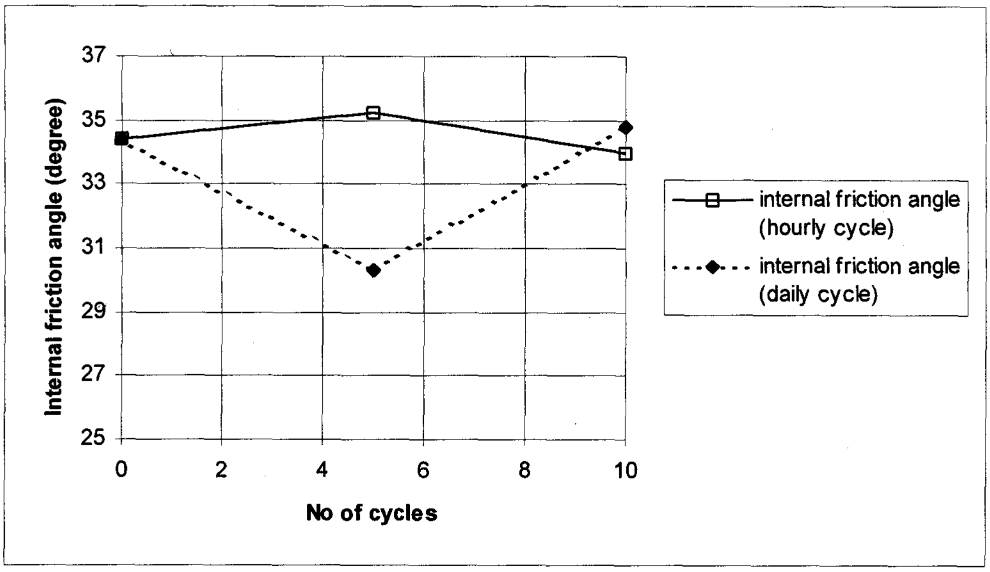


FIGURE 4.12. Comparison of internal friction angles for 10 % rubber added kaolinite obtained by direct shear tests.

Cohesion of ten per cent rubber added kaolinite was 97 kPa before application of freeze thaw cycles. It decreased to 96 kPa after five hourly cycles were applied and to 85 kPa after ten hourly cycles were applied. The application of daily cycles affected the cohesion of ten per cent rubber added samples more significantly with respect to hourly cycles. Cohesion decreased to 83 kPa after five daily cycles and to 71 kPa after ten daily cycles. Application of freeze and microwave thaw cycles did not change the internal friction angle. The only change observed in five daily cycles was a decrease in internal friction angle by four degrees.

As the direct shear test results obtained after the application of freeze and microwave thaw compared with the ones obtained after the application of freeze and conventional oven thaw, the following points can be stated.

As shear strength of pure kaolinite samples subjected to hourly cycles are compared, it is observed that shear strength decreased from 291 kPa to 266 kPa as number of cycles increased from five to ten in the microwave thawing case and 280 kPa to 273 kPa in conventional oven thawing case (Figure 4.13). In daily cycles, the shear strength increased after pure kaolinite samples were subjected to five cycles in both cases, microwave oven thawing and conventional oven thawing. Application of five more cycles created the same behavior in both methods, the shear strength decreased by 3.5 per cent in microwave thawing and by 2.2 per cent in conventional oven thawing (Figure 4.14).

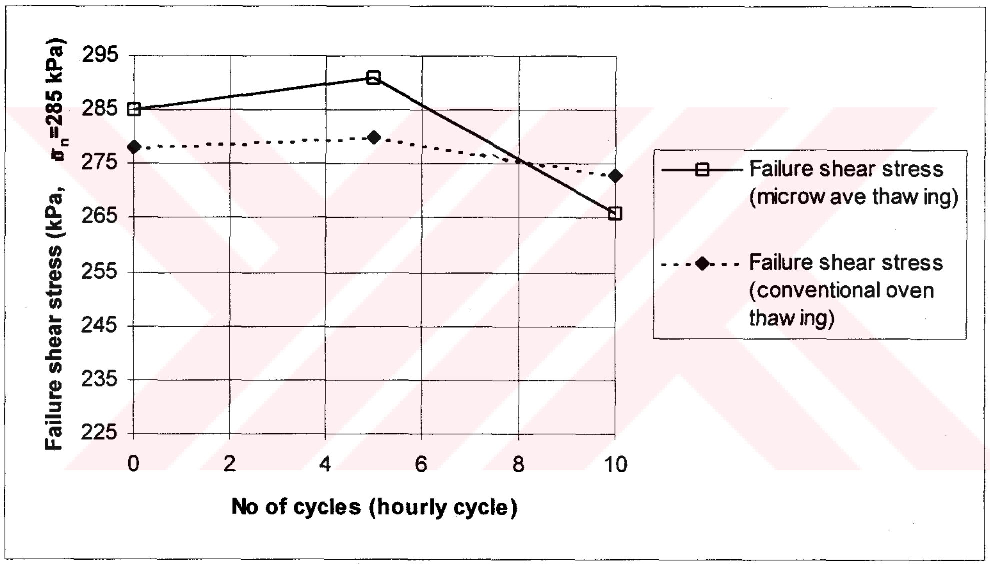


FIGURE 4.13. Comparison of failure shear stresses for pure kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

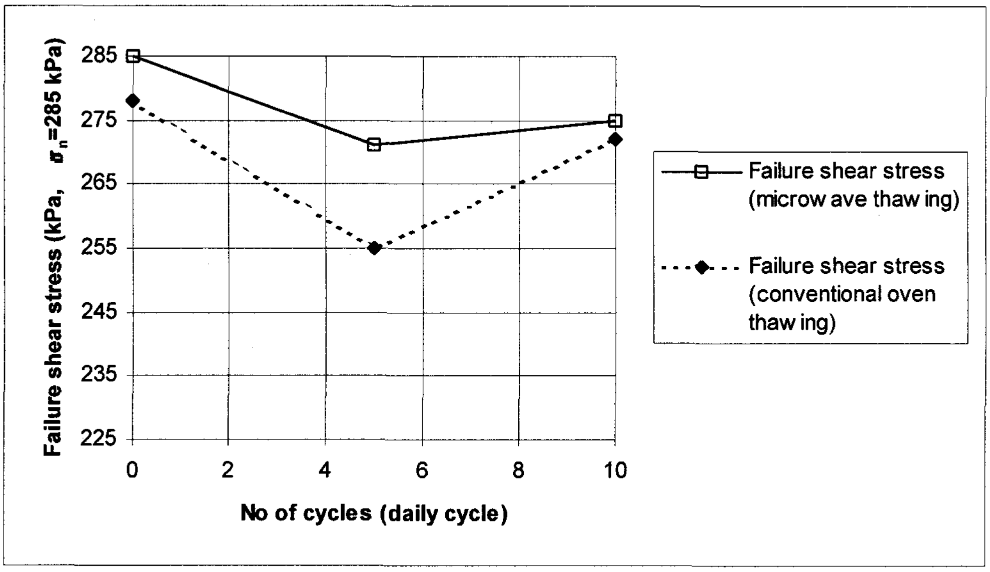


FIGURE 4.14. Comparison of failure shear stresses for pure kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

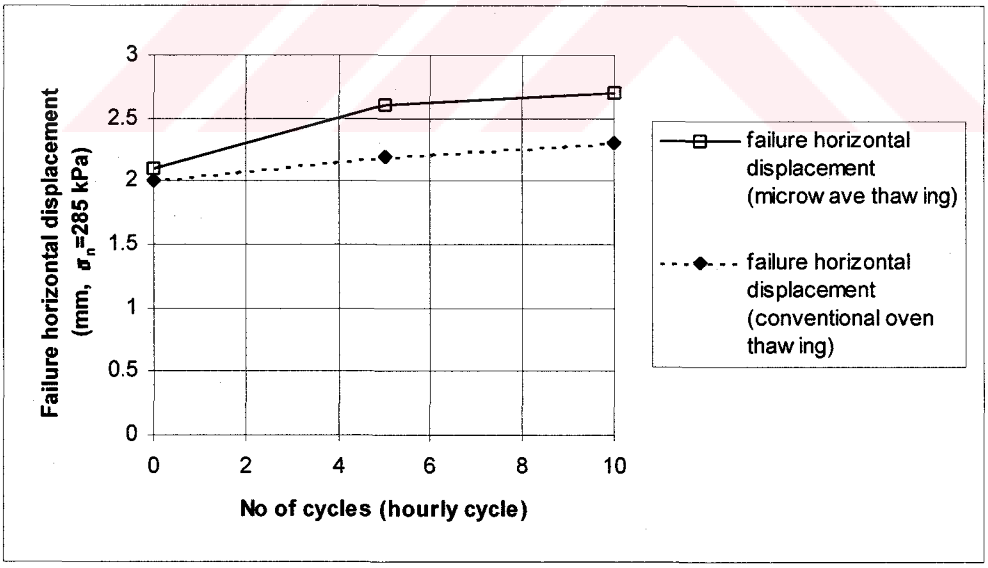


FIGURE 4.15. Comparison of failure horizontal displacements for pure kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

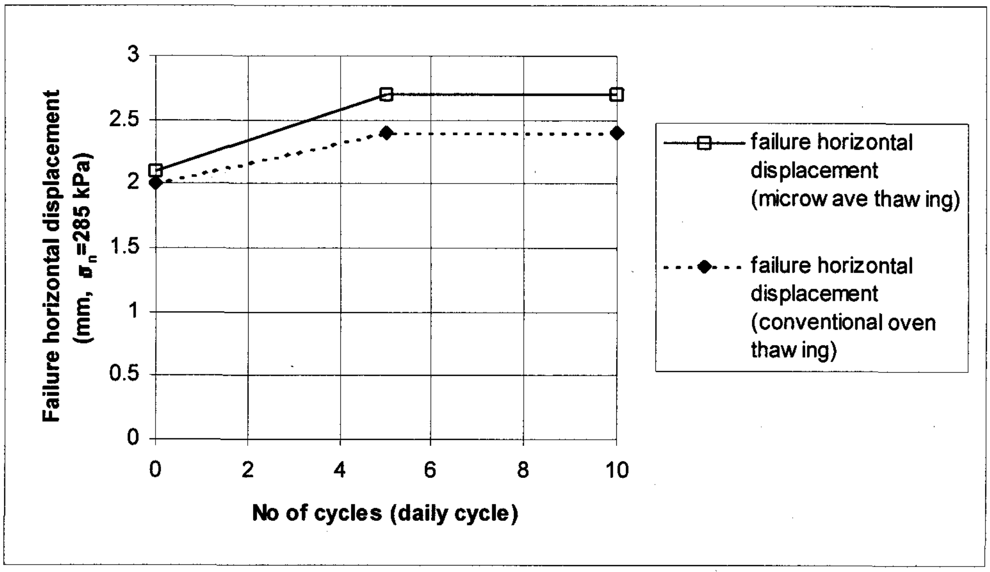


FIGURE 4.16. Comparison of failure horizontal displacements for pure kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

As it is seen from Figures 4.15 and 4.16, pure kaolinite samples subjected to freeze and microwave thawing cycles experienced more ductile failure than the ones subjected to freeze and conventional oven thaw cycles. After ten daily freeze and microwave thaw cycles were applied, the horizontal displacement at failure was 2.7 mm and after ten freeze and conventional oven thaw cycle it was 2.4 mm.

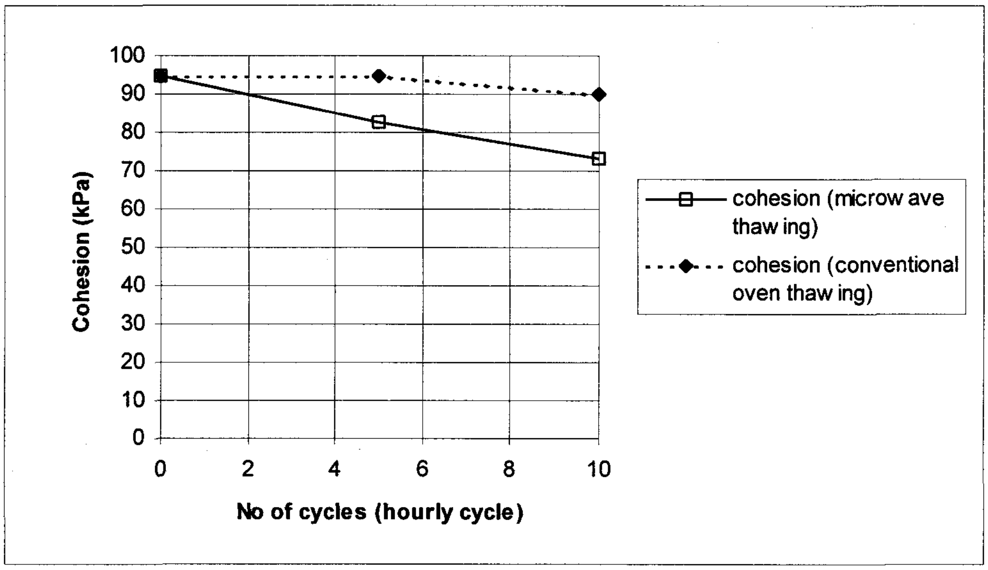


FIGURE 4.17. Comparison of cohesion values for pure kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

In both hourly and daily cycles, cohesion of pure kaolinite in microwave thawing case was greater than the cohesion obtained after conventional oven thawing. As number of cycles increased from zero to five and five to ten, cohesion showed a decreasing trend. This reduction in cohesion was 28.7 per cent after ten daily freeze and microwave thaw cycles, and 26.3 per cent after ten freeze and conventional oven thaw cycles (Figure 4.17, 4.18).

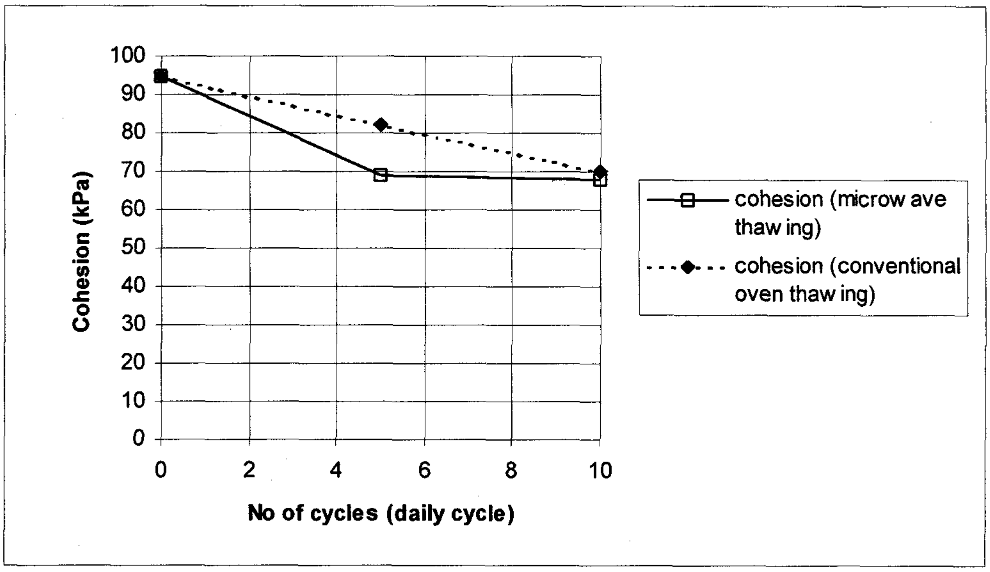


FIGURE 4.18. Comparison of cohesion values for pure kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

In conventional oven thawing case, internal friction angle showed an increasing trend as number of cycles increased. In microwave oven thawing case, after five cycles, internal friction angle increased, application of five more cycles decreased the internal friction angle. These increases and decreases are within the range of ± 4 degrees (Figure 4.19, Figure 4.20).

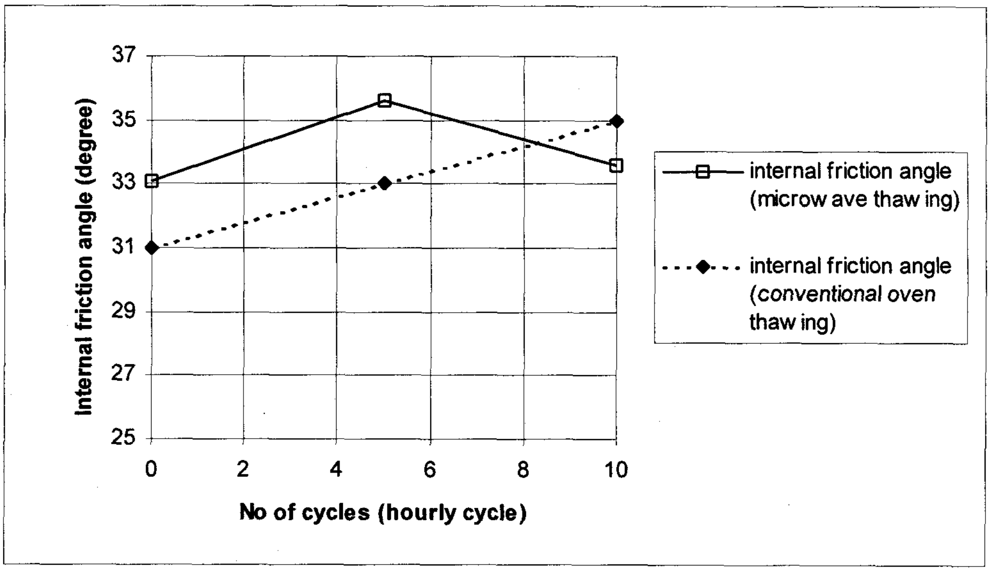


FIGURE 4.19. Comparison of internal friction angle values for pure kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

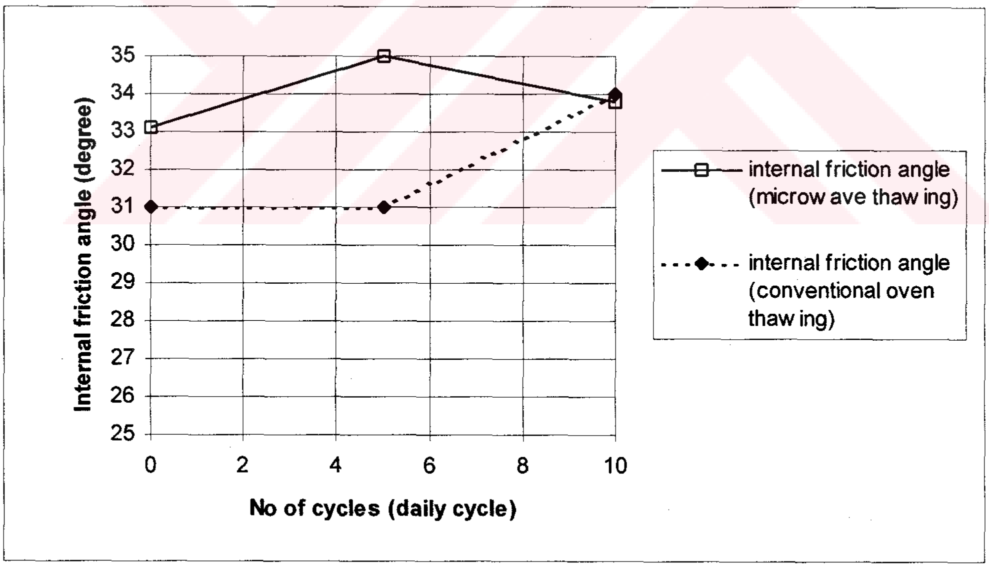


FIGURE 4.20. Comparison of internal friction angle values for pure kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

Figures 4.21-4.28 are the graphical presentation of direct shear test results applied to ten per cent rubber added kaolinite samples. Results obtained after freeze and microwave thaw cycles were compared with the ones obtained after freeze and conventional oven thaw cycles.

Shear stress of ten per cent rubber added kaolinite samples increased as number of cycles increased from zero to five and five to ten in conventional oven thawing case. In microwave oven thawing case, increasing the number of cycles created opposite effect in both hourly and daily cycles (Figures 4.21, 4.22).

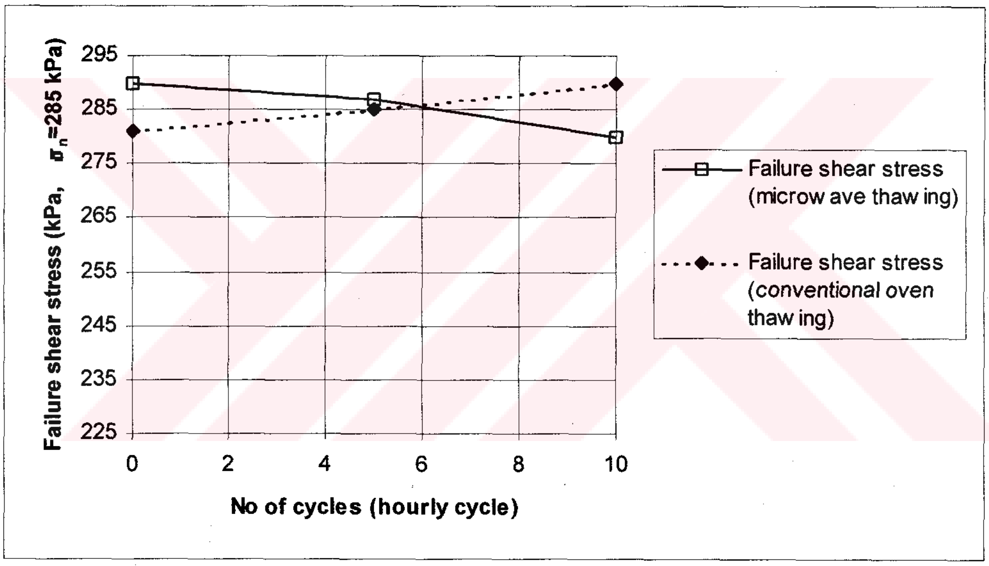


FIGURE 4.21. Comparison of failure shear stresses for 10 % rubber added kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

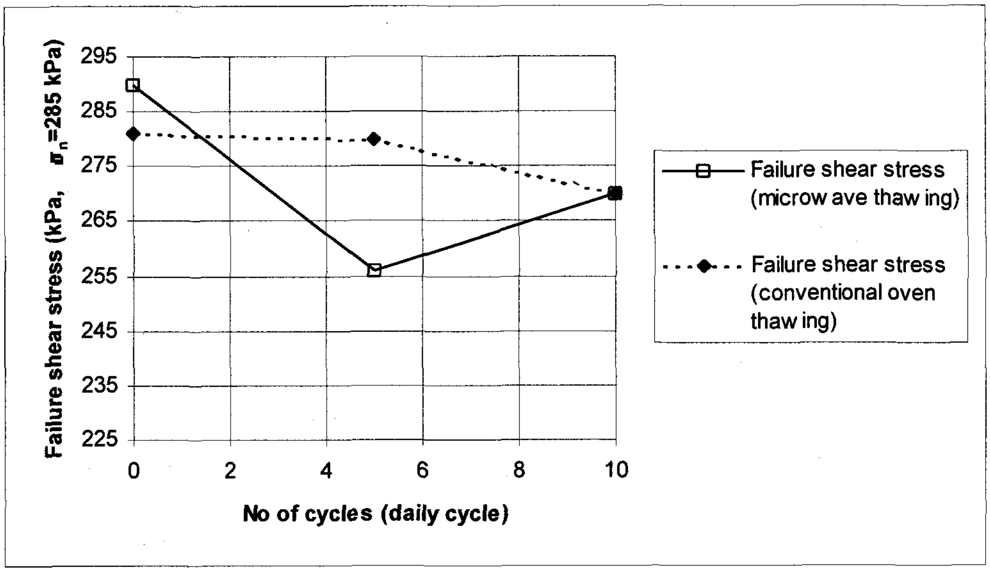


FIGURE 4.22. Comparison of failure shear stresses for 10 % rubber added kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

Ten per cent rubber added samples subjected to microwave oven thaw cycles experienced more ductile failure than the ones subjected to conventional oven thaw cycles. As number of cycles increased samples underwent larger displacements. Failure horizontal displacement was 2.8 mm after samples were subjected to ten daily freeze and microwave oven thaw cycles and 2.4 mm after samples were subjected to ten daily freeze and conventional oven thaw cycles (Figures 4.23, 4.24).

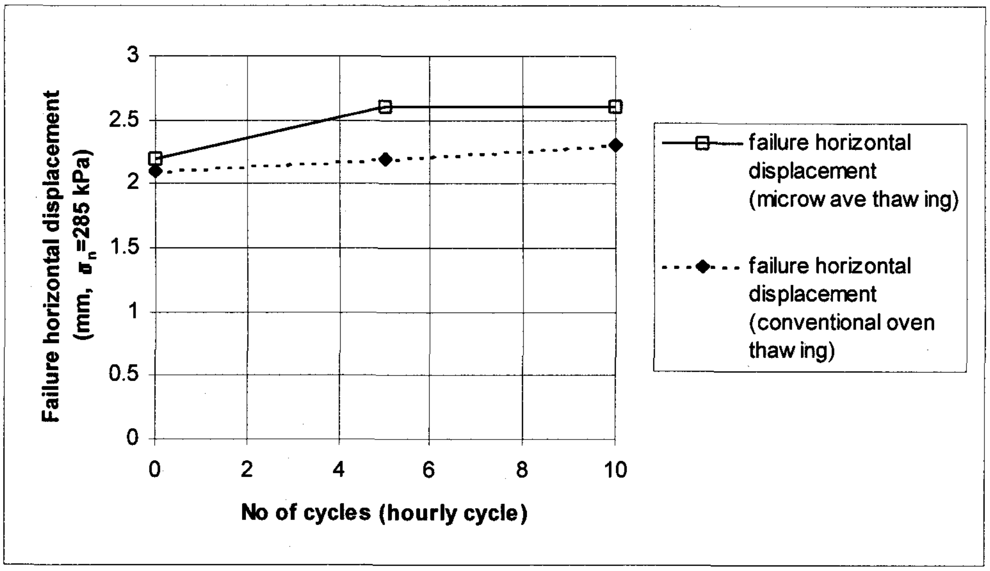


FIGURE 4.23. Comparison of failure horizontal displacements for 10 % rubber added kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

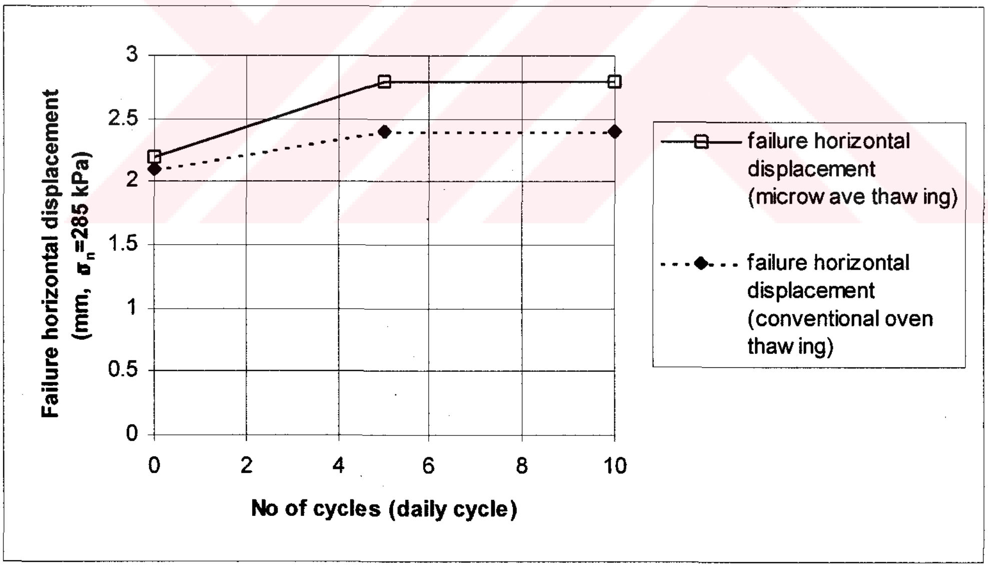


FIGURE 4.24. Comparison of failure horizontal displacements for 10 % rubber added kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

Cohesion of ten per cent rubber added samples subjected to microwave oven thaw cycles was smaller than the ones subjected to conventional oven thaw cycles. As number of cycles increased samples' cohesion decreased. This decrease was 27.3 per cent after samples were subjected to ten daily freeze and microwave oven thaw cycles and 22.4 per cent after samples were subjected to ten daily freeze and conventional oven thaw cycles (Figures 4.25, 4.26).

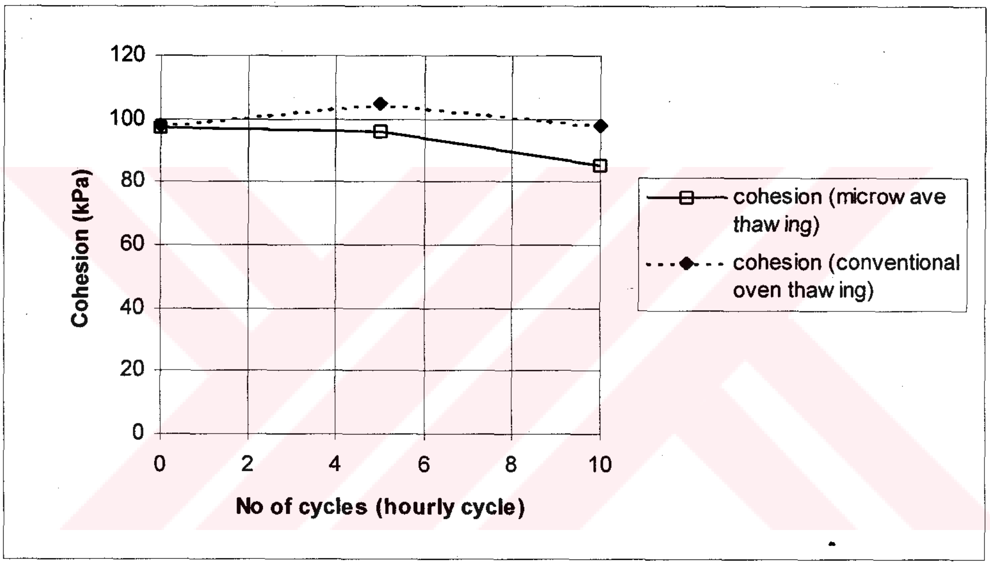


FIGURE 4.25. Comparison of cohesion values for 10 % rubber added kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

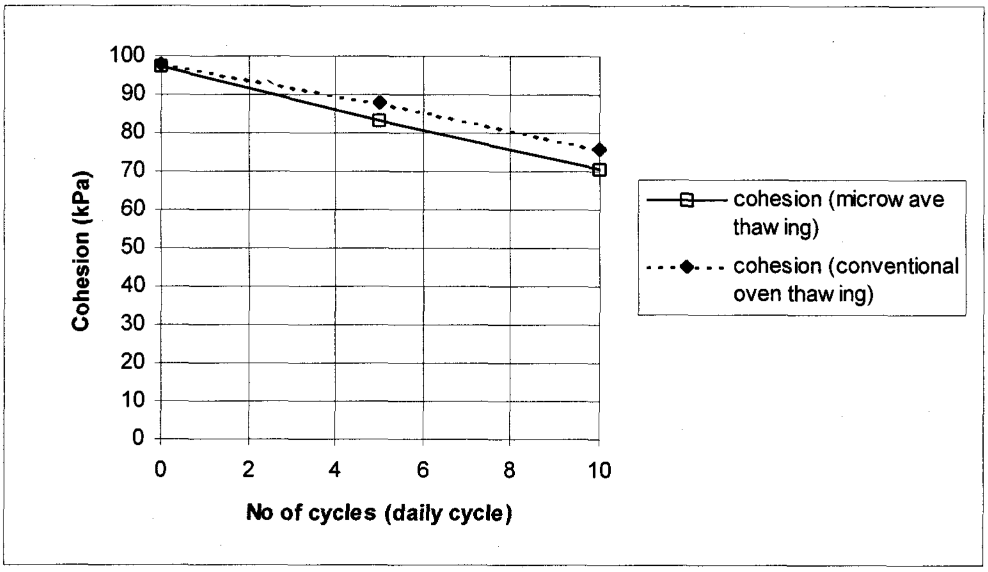


FIGURE 4.26. Comparison of cohesion values for 10 % rubber added kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

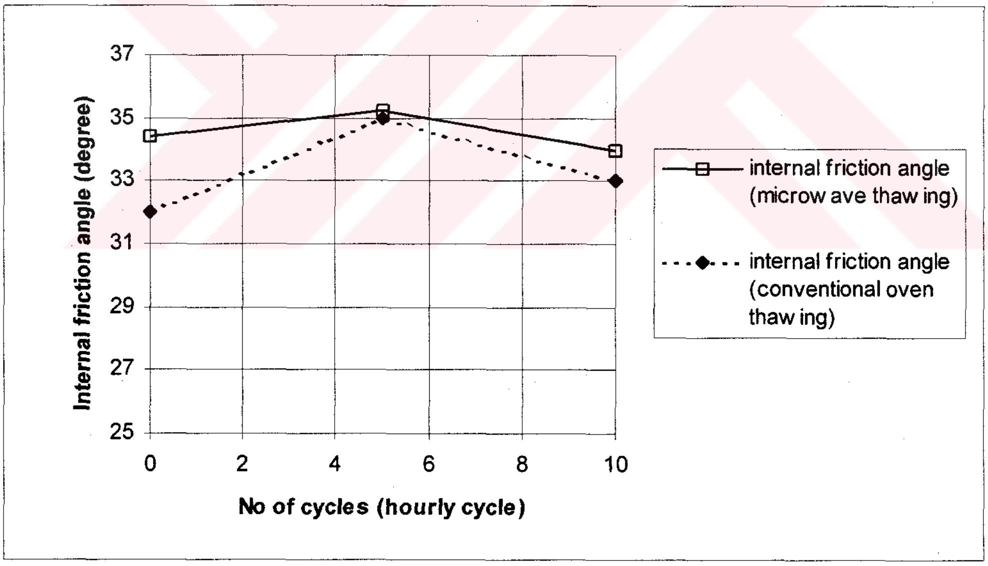


FIGURE 4.27. Comparison of internal friction angle values for 10 % rubber added kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle obtained by direct shear tests.

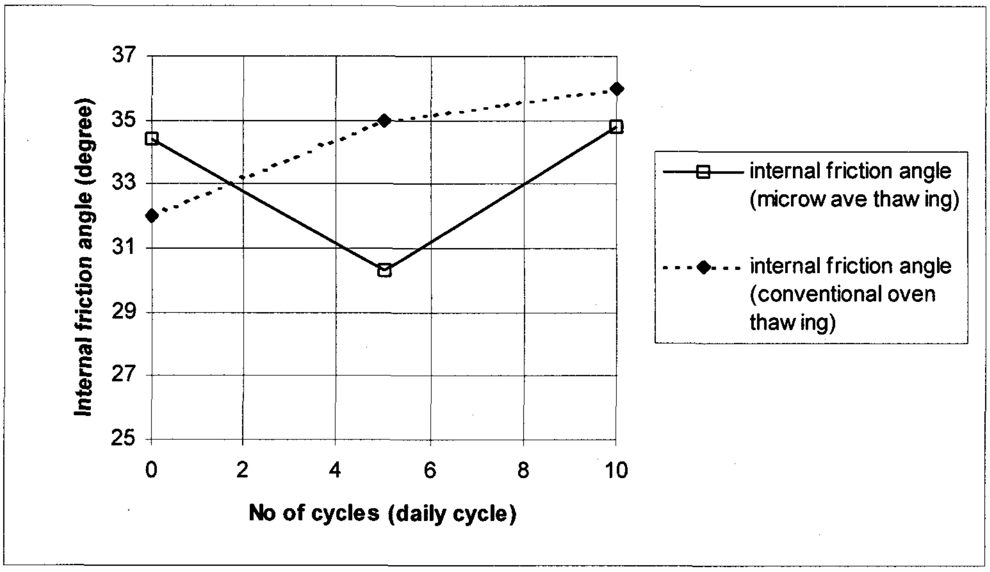


FIGURE 4.28. Comparison of internal friction angle values for 10 % rubber added kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle obtained by direct shear tests.

Failure shear stresses obtained by direct shear tests for pure kaolinite samples which were subjected to zero, five and ten freeze and microwave/conventional oven thaw cycles are compared in Figures 4.29 and 4.30. Pure kaolinite samples subjected to freeze and microwave thaw hourly cycles experienced larger decrease in shear strength after ten cycles with respect to the ones subjected to freeze and conventional oven thaw hourly cycles. For the daily cycles, major decrease in shear strength occurred after the application of five cycles . Samples subjected to five more daily cycles did not experience any significant change in shear strength in both microwave and conventional oven thawing methods (Figures 4.31, 4.32).

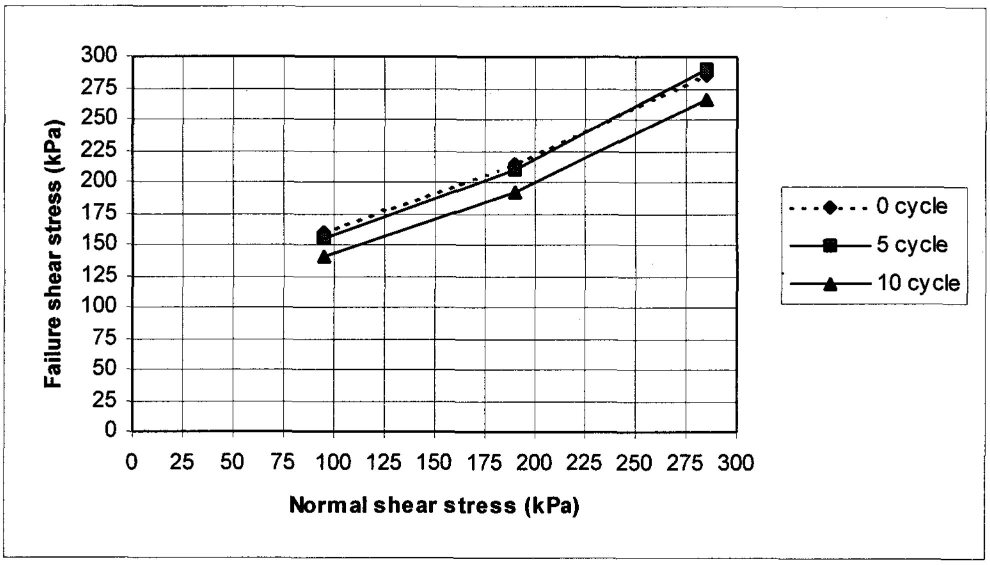


FIGURE 4.29. Failure shear stress of pure kaolinite subjected to freeze and microwave thaw hourly cycles.

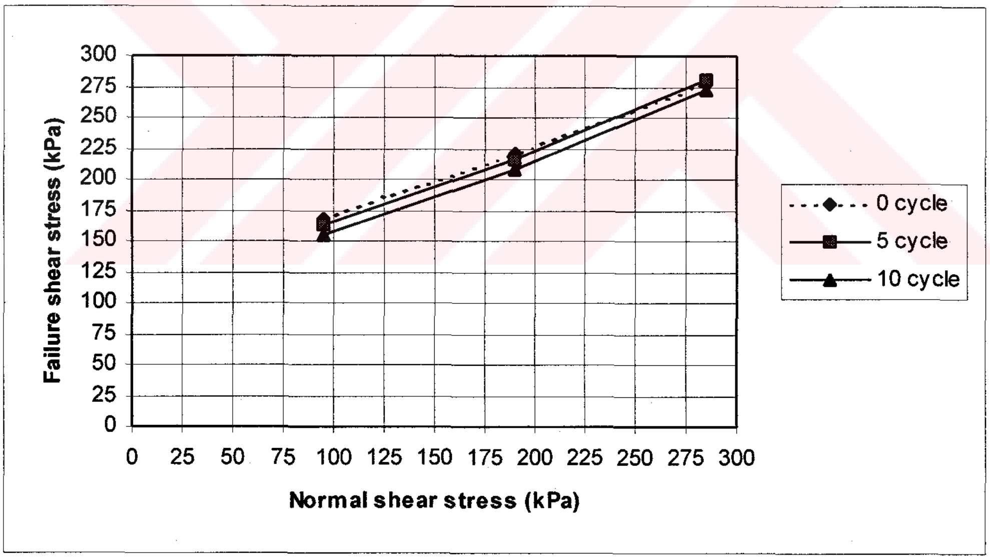


FIGURE 4.30. Failure shear stress of pure kaolinite subjected to freeze and conventional oven thaw hourly cycles.

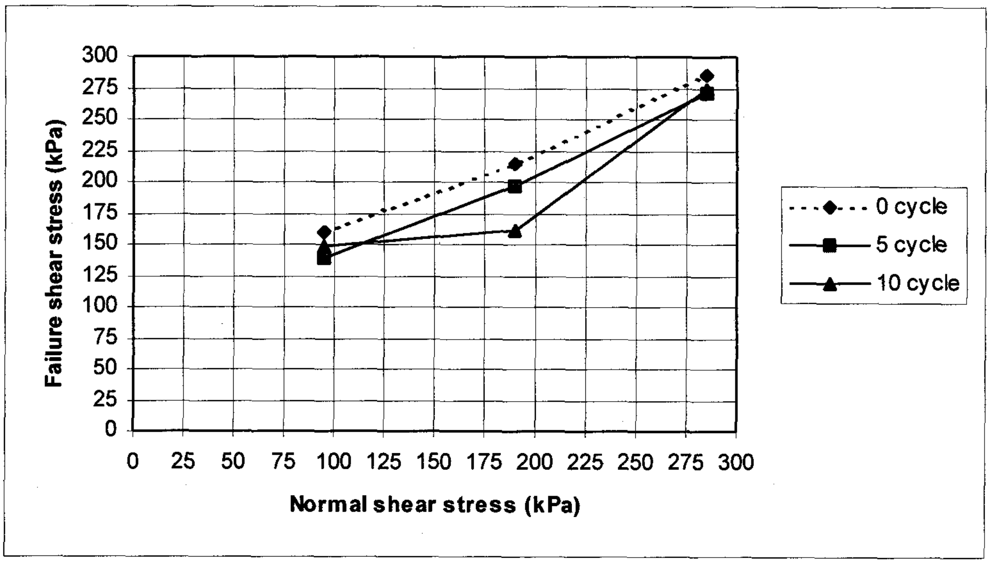


FIGURE 4.31. Failure shear stress of pure kaolinite subjected to freeze and microwave thaw daily cycles.

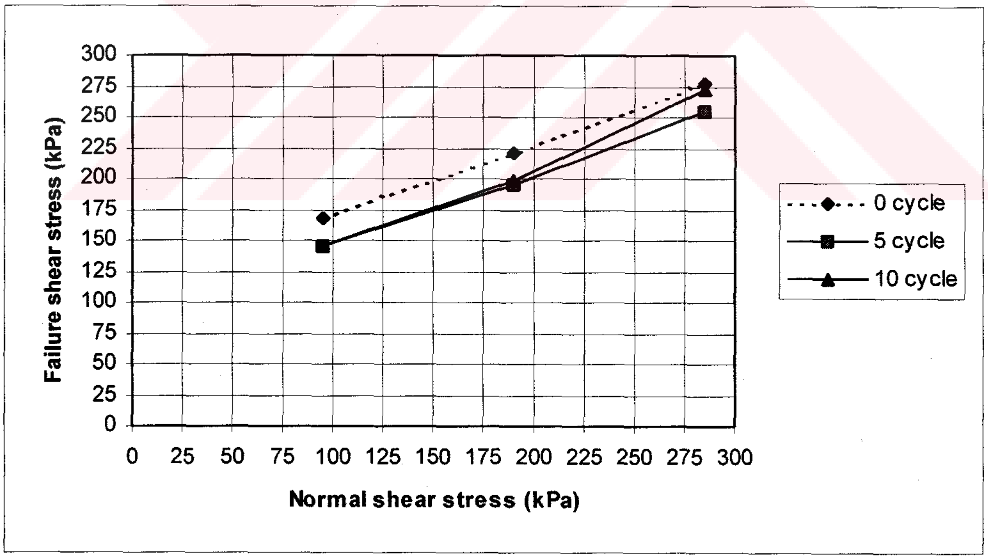


FIGURE 4.32. Failure shear stress of pure kaolinite subjected to freeze and conventional oven thaw daily cycles.

As direct shear test results performed on pure kaolinite and 10 per cent rubber added kaolinite samples obtained by conventional heating methods compared with the ones obtained by microwave heating method the following results are obtained: In both methods, as number and duration of cycles increased, the shear strength and cohesion values decreased. The decrease in cohesion was more significant in microwave heating method. Also the samples experienced larger deformations when subjected to microwave heating during thawing period.

4.3. Unconfined Compression Test Results

After the application of hourly and daily freeze and microwave thaw cycles, unconfined compression tests were conducted on pure kaolinite and 10 per cent rubber added kaolinite samples in order to determine their unconfined compressive strength and their stress strain behavior. The effect of number and duration of cycles on the strength characteristics were investigated. The results were compared with those obtained after the samples were subjected to freeze and conventional oven thaw cycles and are summarized in Table 4.5. The following results are obtained after freeze and microwave thaw cycles.

After five hourly freeze and microwave thaw cycles, the unconfined compressive strength of pure kaolinite samples increased by 29 per cent. Application of five more hourly cycle didn't change the unconfined compressive strength value. In daily cycles, it decreased by 15 per cent after five cycles, and 24 per cent after ten cycles (Figure 4.33). Strain at failure showed a decreasing trend in both hourly and daily cycles (Figure 4.34).

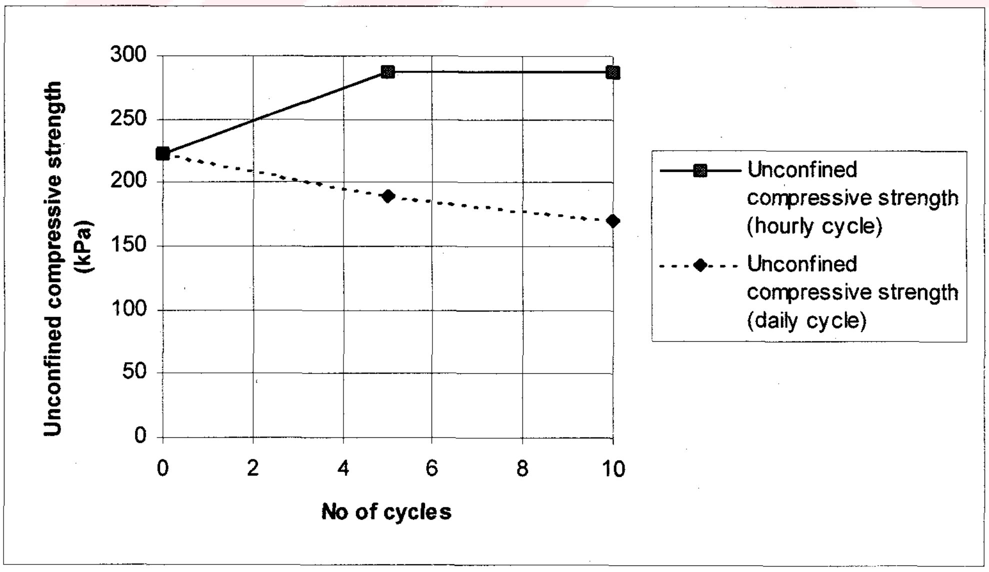


FIGURE 4.33. Comparison of unconfined compressive strength values for pure kaolinite samples subjected to hourly and daily cycles obtained by unconfined compression tests.

Table 4.5. Unconfined Compression Test results.

UNCONFINED COMPRESSIVE STRENGTH, kPa STRAIN AT FAILURE, %									
PURE KAOLINITE									
0 cycle		5 cycle				10 cycle			
		2 hour freezing+7 min thawing		1 day freezing+7 min thawing		2 hour freezing+7 min thawing		1 day freezing+7 min thawing	
1	2	1	2	1	2	1	2	1	2
223	200	287	188	189	140	287	180	170	130
7.25	8.50	5.20	8.75	6.10	10.25	5.60	9.00	6.50	10.50
KAOLINITE + 10% RUBBER									
0 cycle		5 cycle				10 cycle			
		2 hour freezing+7 min thawing		1 day freezing+7 min thawing		2 hour freezing+7 min thawing		1 day freezing+7 min thawing	
1	2	1	2	1	2	1	2	1	2
201	190	199	170	148	180	211	180	139	110
9.50	8.25	10.00	9.25	8.20	10.50	5.80	9.50	7.80	9.75

1 Microwave thawing

2 Conventional oven thawing [18]

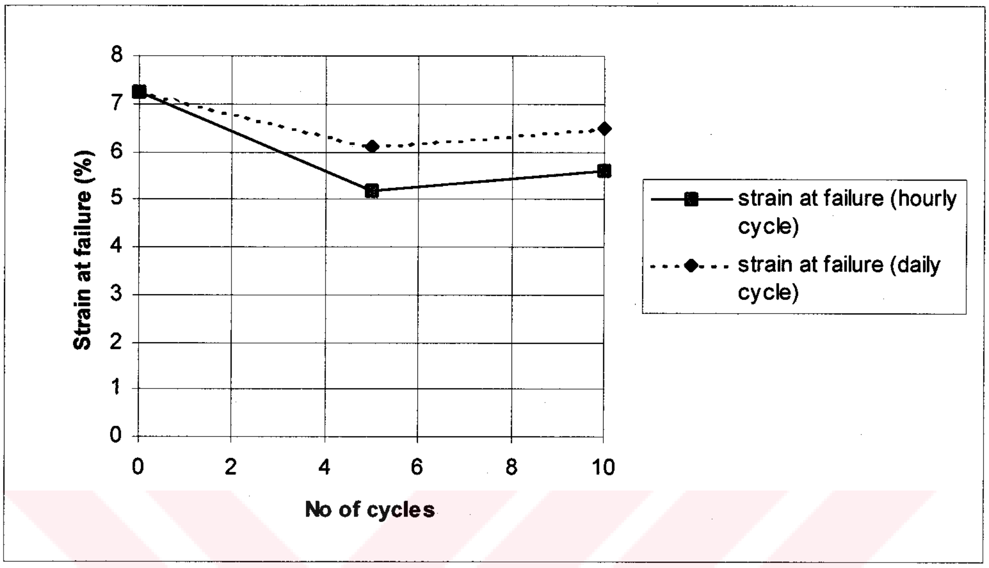


FIGURE 4.34. Comparison of strain at failure values for pure kaolinite samples subjected to hourly and daily cycles obtained by unconfined compression tests.

Unconfined compressive strength of ten per cent rubber added kaolinite samples decreased after hourly and daily freeze and microwave thaw cycles (Figure 4.35). Strain at failure has also showed a decreasing trend after freeze and microwave thaw cycles. Decreasing rate is 40 per cent for ten hourly cycles and 18 per cent for ten daily cycles (Figure 4.36).

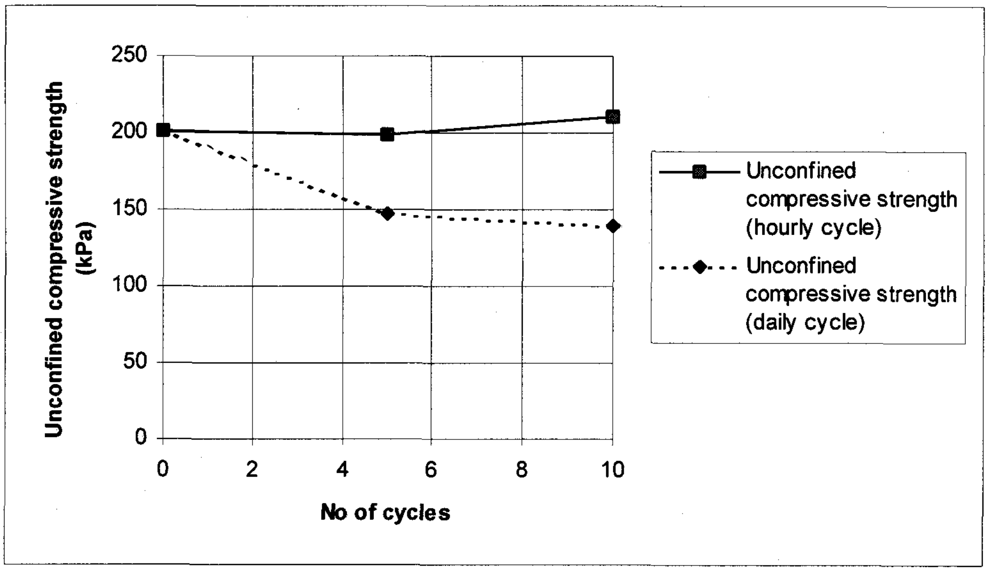


FIGURE 4.35. Comparison of unconfined compressive strength values for 10 per cent rubber added kaolinite samples subjected to hourly and daily cycles obtained by unconfined compression tests.

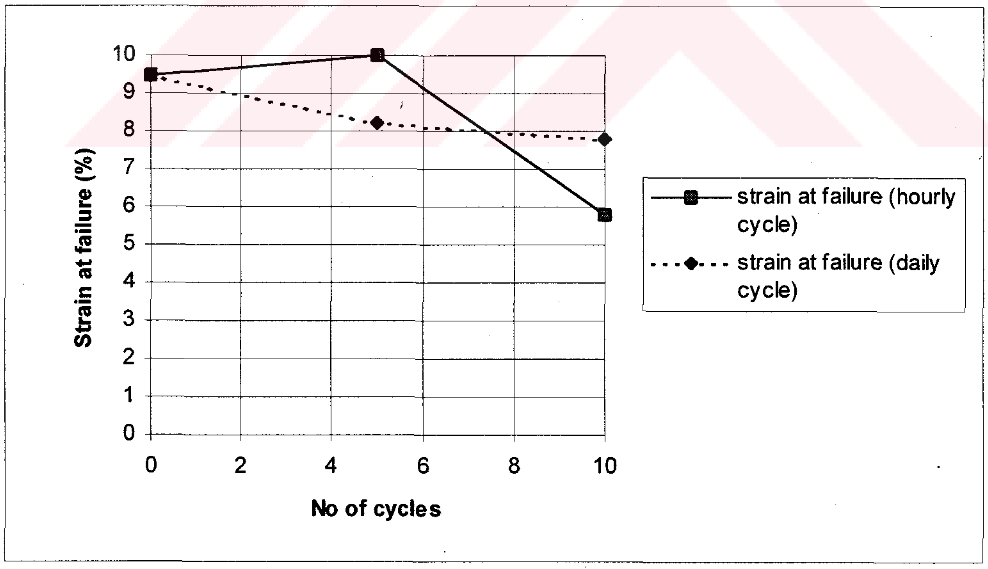


FIGURE 4.36. Comparison of strain at failure values for 10 per cent rubber added kaolinite samples subjected to hourly and daily cycles obtained by unconfined compression tests.

As the unconfined compression test results obtained by microwave thawing compared with the ones obtained by conventional oven thawing, the following points are concluded.

For pure kaolinite samples, in hourly cycles, unconfined compressive strength decreased as number of cycles increased when samples were thawed in conventional oven. It increased when microwave oven was used in thawing period. The results are plotted in Figure 4.37. In daily cycles, unconfined compressive strength decreased in both cases (Figure 4.38). Decrease in unconfined compressive strength is 24 per cent for microwave thawing and 35 per cent for conventional oven thawing when samples were subjected to ten cycles.

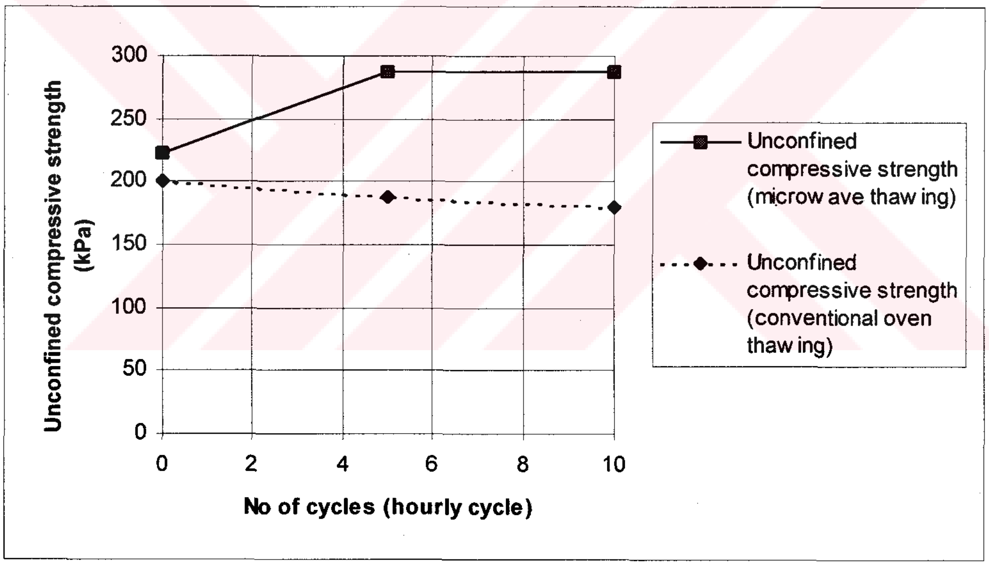


FIGURE 4.37. Comparison of unconfined compressive strength values for pure kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle.

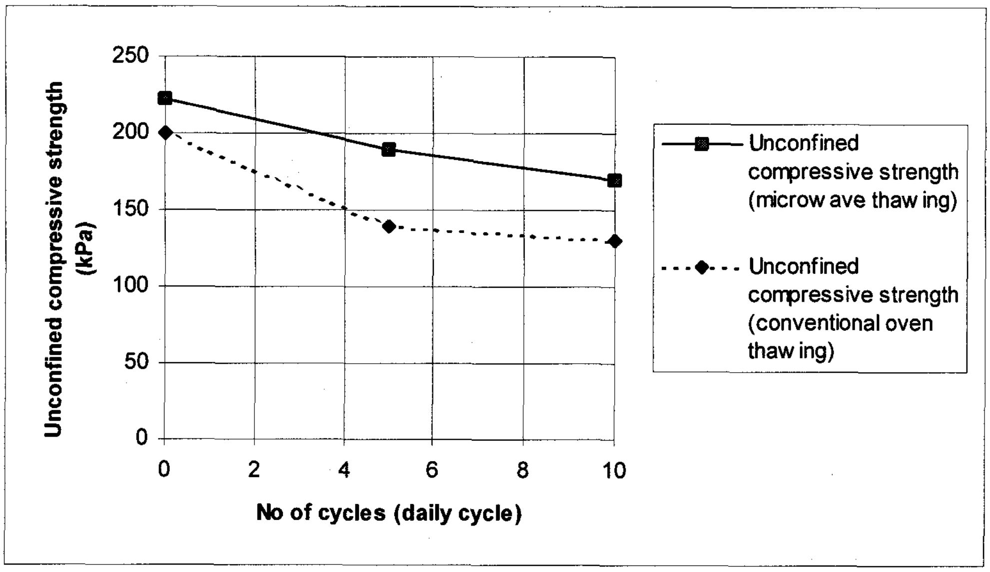


FIGURE 4.38. Comparison of unconfined compressive strength values for pure kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle.

Strain at failure for pure kaolinite samples increased as number of cycles increased in both hourly and daily cycles when conventional heating method was used in thawing period. It was decreased when microwave oven was used in thawing period (Figure 4.39, Figure 4.40).

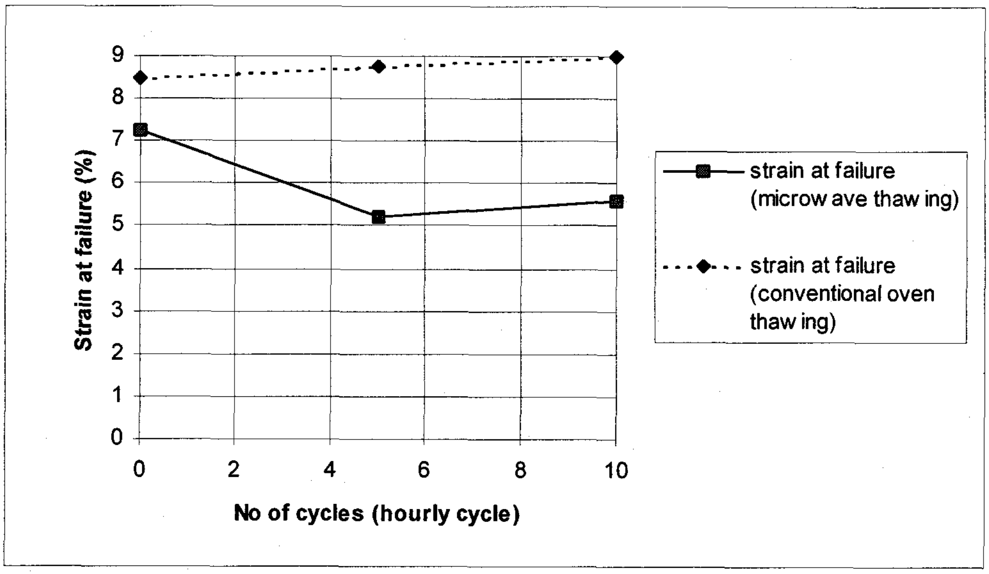


FIGURE 4.39. Comparison of strain at failure values for pure kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle.

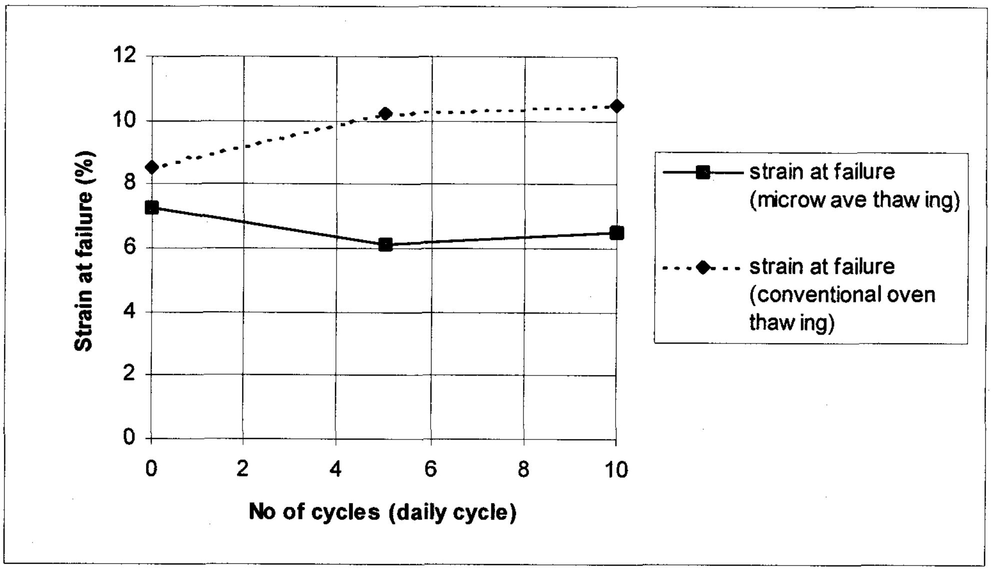


FIGURE 4.40. Comparison of strain at failure values for pure kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle.

For ten per cent rubber added kaolinite samples, in both methods, (microwave oven and conventional oven thawing), unconfined compressive strength decreased as number of cycles increased for both hourly and daily cycles. Figure 4.41 and Figure 4.42 show this relationship. Strain at failure decreased 18 per cent when microwave was used in thawing period and increased 18 per cent when conventional oven was used in thawing period after ten per cent rubber added kaolinite samples were subjected to 10 daily cycles (Figure 4.43, Figure 4.44).

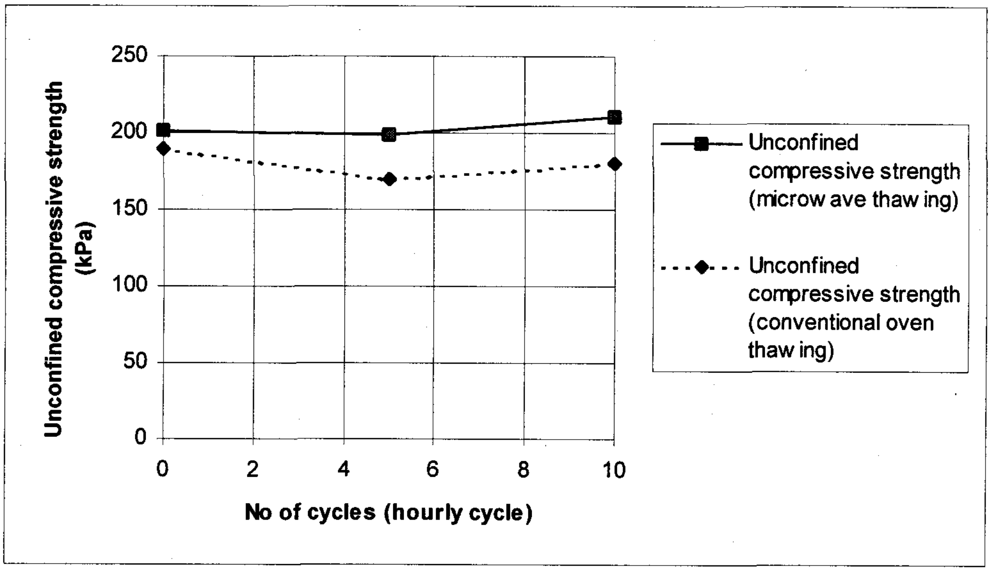


FIGURE 4.41. Comparison of unconfined compressive strength values for 10 per cent rubber added kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle.

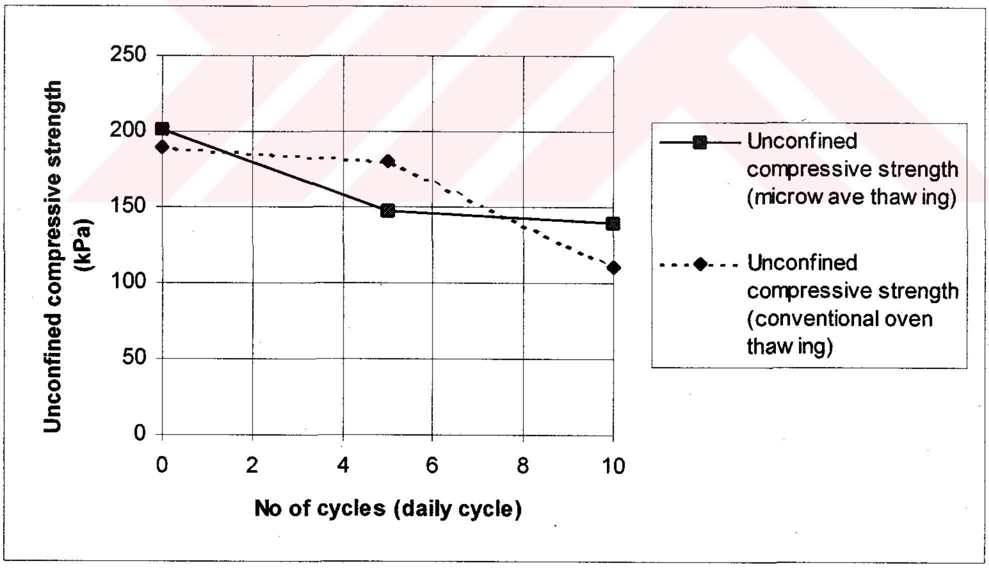


FIGURE 4.42. Comparison of unconfined compressive strength values for 10 per cent rubber added kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle.

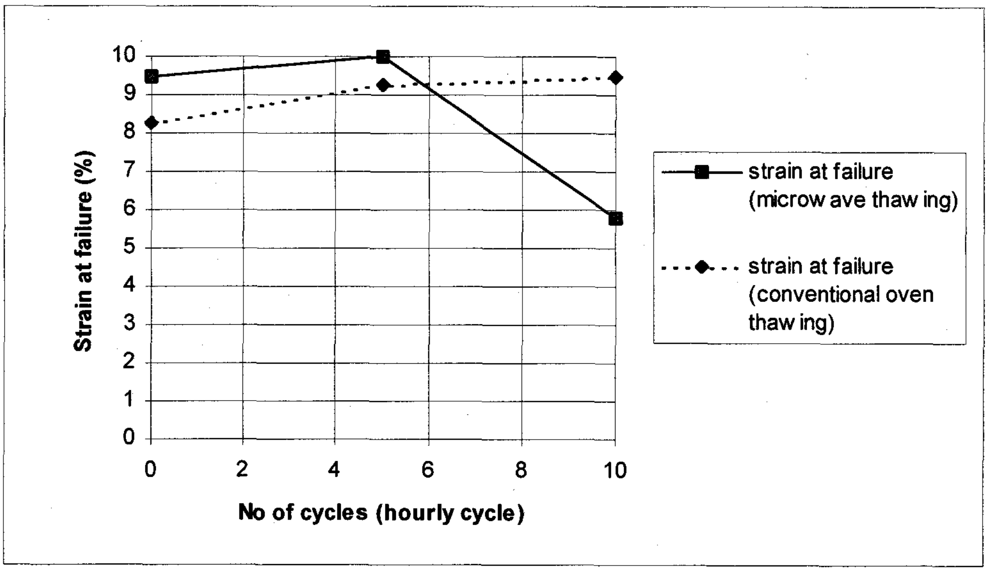


FIGURE 4.43. Comparison of strain at failure values for ten per cent rubber added kaolinite samples subjected to microwave thawing hourly cycle and conventional oven heating hourly cycle.

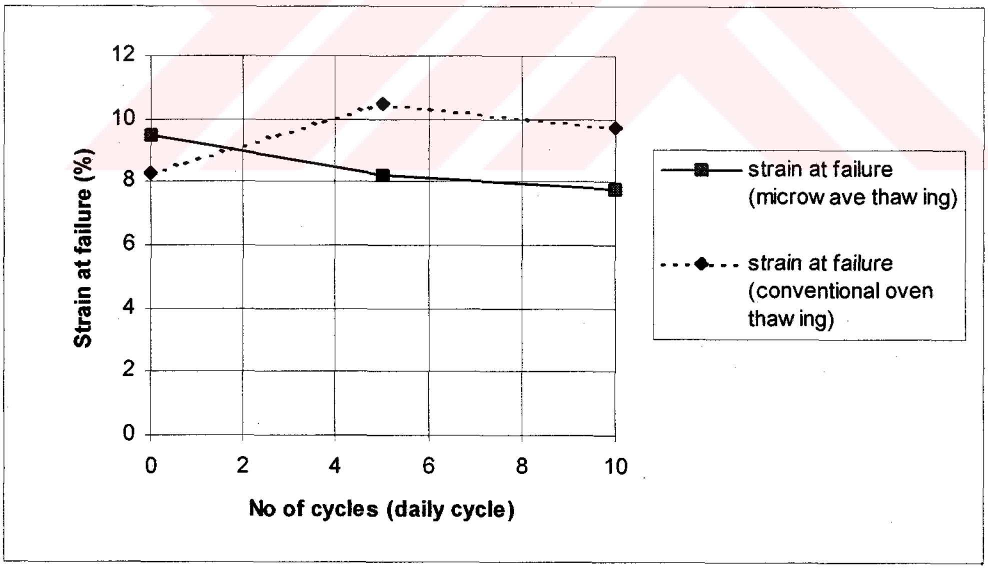


FIGURE 4.44. Comparison of strain at failure values for ten per cent rubber added kaolinite samples subjected to microwave thawing daily cycle and conventional oven heating daily cycle.

V. CONCLUSIONS

In an age of continued pollution, excessive refuse production, and contaminated water supplies, the design criteria for landfill liners and covers must be reassessed. Landfill liners and covers are intended to retain refuse and prevent surface and ground water infiltration to and from the containment cell. These covers and liners commonly include a clay component which may not function as designed if subjected to a number of extreme conditions such as chemical attack, erosion, desiccation, differential settlement, and freeze/thaw effects. For liner and cover design the increase in hydraulic conductivity due to freeze and thaw is generally studied. However, for the clay layer used for cover at the slopes of disposal area, the change in shear strength properties become important due to freeze and thaw effects.

Many studies have been conducted to determine the effect of freeze and thaw cycling on the strength of clayey soils especially in highway engineering applications. The major problem in freezing and thawing studies is the extremely long testing times. Any method that will shorten the freeze thaw test time will be beneficial. The use of microwave energy for processing materials has been the subject of a large amount of exploratory research. The aim of using microwave energy is to decrease the duration of freeze and thaw cycle by half as compared to conventional heating method and to see whether if microwave energy affects the strength and structure of compacted clay with respect to conventional heating in freeze and thaw cycles.

Direct shear tests and unconfined compression tests were conducted on compacted kaolinite samples. Water content determination tests were conducted on compacted kaolinite and bentonite samples. The effect of number of cycling and duration of freeze and microwave thaw cycling were investigated and compared with the results obtained by conventional oven thawing. Zero, five and 10 cycles were applied for duration of one hour, one day freezing and 5-10 minutes of microwave thawing.

The test results obtained are limited to the type of microwave and power levels used in this study. The conclusions may be generalized only if comparable power levels are used. The following statements can be concluded according to the results obtained by the present experimental study:

1. Data indicates that water content determination test results obtained by the standard method and by incremental drying with a microwave oven could reasonably be expected to agree within ± 1 per cent for most soil types, therefore microwave oven is practical way for rapidly obtaining moisture content of soils.
2. Direct shear test results revealed that shear strength obtained by microwave thawing is greater than the ones obtained by conventional oven thawing whereas the strain is greater in microwave thawing in contrast to the results of unconfined compression tests. The information obtained by direct shear tests is not sufficient to predict and compare the cohesion and internal friction angles of clay samples in the case of microwave thawing and conventional oven thawing but in general cohesion values were lower and internal friction angle values were slightly higher than those values obtained from conventional tests.
3. Unconfined compression tests results show that the strength of clay samples subjected to freeze and microwave thaw cycling is greater than the ones obtained by the conventional thawing method. Samples experienced smaller strain when subjected to freeze and microwave thaw cycling with respect to those obtained from conventional oven thawing.

Microwave energy can be used alternatively for restricted applications. In freeze and thaw cycling of clay liners, use of microwave energy created the same effects as conventional heating method; changing the parameters such as increasing the number of cycles and duration of cycles lead to the same behavior in strength characteristics with only one difference: It lead to the higher strength values.

A preliminary investigation on microwave oven type, power level and soil type is recommended for each geotechnical engineering application. The results obtained from microwave testing must be compared to those of obtained from conventional testing. Once it is confirmed that the both techniques are comparable, then the microwave may be used safely to decrease cycle duration.

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