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The Direct Incorporation of Organic Phase Change Materials in Thermal Storage Gypsumboard

E. Ghanbari

A Thesis

in

the Centre

for

Building Studies

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ABSTRACT

Thermal analysis of saturated long chain esters of monobasic fatty acids and their binary mixtures, proved them to be suitable energy storage materials, for space heating applications. Based on this knowledge, a series of experimental investigations was conducted to establish the blending parameters when incorporating a binary mixture of butyl stearate and butyl palmitate (as an Organic Phase Change Material) into gypsum paste, for the purpose of producing energy storage gypsum board. The function of this energy storage gypsum board is to store and save excess energy for later use (i.e. excess heat that come from solar-gain, pumps, heaters, cooking appliances, bathing, lamps, human bodies, etc.) in residential and commercial buildings as well as to regulate the desired temperature inside the living space.

Blending was carried out with the help of several types of surface active agents. Blending components such as foaming solutions, emulsifiers, starch, water and pulp paste as well as blending parameters were both prepared and optimized. Various chemical, physical and compatibility tests were conducted on the blended product.

Thermal analysis, by differential scanning calorimetry, was conducted to determine the melting and freezing points of PCM inside the board and to measure the amount of heat released or absorbed by the board during one operating cycle. Finally the blended product was examined by infra red spectrophotometry to

determine the degree to which PCM reacts with the other components.

Results indicate that not only direct incorporation of PCM in gypsum wallboard is feasible but also that the finalized products are well above ASTM and CSA standards.

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Thanks are extended to Mrs. D. Banu, research associate, for her constructive suggestions regarding the laboratory work.

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Symbol	
ANS	American National Standard
ASTM	American Society of Testing Materials
Ave	Average
BPG	Byproduct gypsum
BS	Butyl Stearate
℃	Celcius
CBS	Centre for Building Studies
CSA	Canadian Standards Association
DA	Dispersing agent
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analyzer
DSG	Desulpho-gypsum
ΔQ	Heat flux
ΔH	Heat of transition
EA	Emulsifying agent
FA	Foaming agent
f.p	Freezing point
FG	Fluoro-gypsum
FSL	Full scale load
f(T)	Temperature dependent heat transfer
GB	Gypsum board
Hf	Heat of freezing
Hm	Heat of melting
IR	Infra red

K	K-factor (conductivity)
k	DSC constant
Me	Methyl group
mp	Melting point
MW	Molecular weight
o/w	Oil in water
PCM	Phase change material
PG	Phospho-gypsum
PVA	Polyvinyl alcohol
R	R-value (Resistivity)
RH	Relative humidity
rpm	Evolution (round) per minute
S-L	Solid-liquid
S-S	Solid-solid
SSKL	Sodium salt of kraft lignin
Temp	Temperature
USG	United States Gypsum Co.
UV	Ultra violet
WFA	Westroc foaming agent
w/g	Water/gypsum ratio
Wt	Weight
XHD	Cross head

CHAPTER 1

INTRODUCTION

Price hikes of fossil fuels in the 70's, the continued depletion of fossil fuels and econo-political tensions associated with this primary source of energy have combined to push industrialized countries (US, Canada, UK, France, West Germany etc.) to initiate or accelerate their research programs toward other means of acquiring or conserving energy resources. In this context research has been concentrated mainly in areas related to solar radiation, wind energy, biomass, hydro-electricity or even hydrogen fusion. However, a necessary and more immediate concern is the more efficient use of current resources including various schemes for energy storage. Hence what is required, is a more efficient management of resources in all energy consuming sectors.

The residential and commercial buildings account for approximately 1/3 of the total consumption of energy. Therefore, research and conservation in this field is vital and, so far, has led to a number of novel measures related to thermal management schemes as well as the development of heat exchangers and advances in

insulation, air tightening and thermal storage techniques. Development and use of energy storage materials, which are the major means of utilization of solar energy, has guided researchers to extend the idea of storing energy to space conservation. By "space" we mean any place in which people live and which needs to be conditioned.

Waste energy, i.e., excess energy from comfort level, which may come from solar gains, extra heating, pumps, lamps, cooking, bathing, human bodies and any other source of energy, is presently largely wasted in buildings. This energy could be stored and saved by using suitable forms of inorganic and organic materials such as Phase-Change Materials (PCMs).

The use of building materials such as concrete, brick or other form of masonry as well as earth and water as thermal storage materials, relies upon their sensible heat capacity. By this way alone it is often not possible to obtain a suitable comfort level within a living space during the entire day. PCMs, on the other hand, depend upon their latent heat capacity for thermal storage and, within a temperature range in which the latent heat is stored or released, are able to achieve their service of storing or delivering energy within a time frame which provides comfort the entire day. However, materials such as concrete, brick or gypsum-board may be impregnated with PCM and consequently can contribute to a considerable extent to energy savings. The premise of the present work is that if PCM (binary mixture of butyl stearate and butyl palmitate) can be cost effectively incorporated into a conventional building element such as gypsum board, then the heat storage

capacity so introduced could significantly contribute to both energy conservation and thermal comfort in a building.

The main objective of this work is to establish a procedure for the direct incorporation of butyl stearate in gypsum board, so that what produced is an energy storage building component which can be used as an energy saver which may, in certain circumstances, replace plain gypsum board.

To achieve the aforementioned objective and to obtain reliable results on the function and durability ot an energy storage gypsumboard, it is of vital importance to have precise knowledge of the sources, formation, properties, characteristics, manufacturing processes and factors affecting properties of the composite. A review of the necessary literature has been made on such topics as gypsum, synthetic gypsum, surfactants, foam and foaming theory, gypsum board manufacturing and PCMs.

The appropriate PCM for the present work has been fully discussed in chapter 3. Chapter 4 deals with composites and the preparation of the components. Chapter 5, provides the development procedure for incorporating PCM into gypsum paste. This is followed in chapter 6 by a discussion related to destructive and non-destructive testing on the sample products. Chapter 7, concentrates on analytical tests such as thermal analysis by differential scanning calorimetry (DSC), IR spectrophotometric analysis, conductivity measurement and setting time evaluation. Conclusion and recommendations are given in chapter 8.

CHAPTER 2

LITERATURE REVIEW

2.1 GYPSUM

2.1.1 Introduction

Gypsum is a common rock like mineral known chemically as hydrated calcium sulphate ($CaSO_4 \cdot 2H_2O$). The mineral gypsum is widely distributed in many parts of the world and is of great economic importance. Gypsum has the unique property of giving up some of its chemically combined water and become calcined when intensely heated. It is returned to its rock-like form chemically when water is added, by forming inter-linking crystals (*crystallization*) [1]. The mixture of gypsum powder (*calcined gypsum*) and water remains plastic for a short time so that it can be easily molded or shaped as desired.

Gypsum has been used by artists and builders for several thousands years. As far back as 2000 B.C. the ancient Egyptians used gypsum for building purposes such as preparing mortar, making plaster, etc.

In the middle ages gypsum was extensively used for decorative purposes in houses, churches, graves, memorials plastic arts etc. The mechanism of hardening of gypsum was not however discovered, until 1765 by Lavoisier, who found that gypsum is a salt [2].

2.1.2 Origin

Nearly all natural deposits of gypsum have been formed by the partial or complete evaporation of sea water, brines in lagoons and other saline water. Gypsum rock consist of a white crystalline mass and it is the principal commercial form of gypsum. Other forms are alabaster, satin spar, selenite, gypsite, and gypsum sand all having minimal commercial value [1,3]. Commercial deposits of gypsum rock are found in one third of the states in the USA. In Canada, major deposits occur in Atlantic Canada, principally in Nova Scotia, Newfoundland and New Brunswick. There are also deposits on the Magdalen Islands in Quebec, along Moose River in James Bay and in Southwestern regions of Ontario, as well as in Alberta, British Colombia, the Northwest Territories and on several Arctic Islands [4].

2.1.3 Mining

Gypsum is mined in open quarries by drilling and blasting, if the deposits are situated near the surface, and it is mined underground if they occur at relatively deep levels beneath the ground surface.

2.1.4 Calcination

Typical crude natural gypsum processing and calcination is shown in Figure 2.1. Gypsum rock from the mine or quarry is crushed, sized and then heated at a temperature ranging between 120 and 160°C, to remove about three-quarters of the water of crystallization. The partially dehydrated product is then ground resulting in the well known calcined gypsum (*hemihydrate*), generally referred to as stucco in North America and Plaster of Paris or simply plaster in many other countries. The calcining process may be performed continuously using a rotary kiln or discontinuously when special retorts (*kettles*) or autoclaves are used.

Temperature control of the rotary kiln is often difficult and, as a result of this, the product usually contains considerable amounts of overheated (up to 220°C) stucco referred to as second settle. For this reason, products from rotary kilns are exposed to atmospheric air to allow the stucco to absorb water.

When the calcining process is performed in a kettle, powdered gypsum is heated between 120-160°C until 1.5 moles of combined water are expelled from each mole of ($CaSO_4 \cdot 2H_2O$) gypsum. Then the resulting product is finely grounded [2] and is then ready for use.

2.1.5 Dehydration

The thermodynamic properties of the system have been the subject of considerable theoretical and practical study. The dehydration reactions are given below:

$$CaSO_4 \cdot 2H_2O \text{ ----------> } CaSO_4 \cdot 1/2H_2O + 3/2H_2O$$

$$CaSO_4 \cdot 1/2H_2O \text{ ----------> } CaSO_4 + 1/2H_2O$$

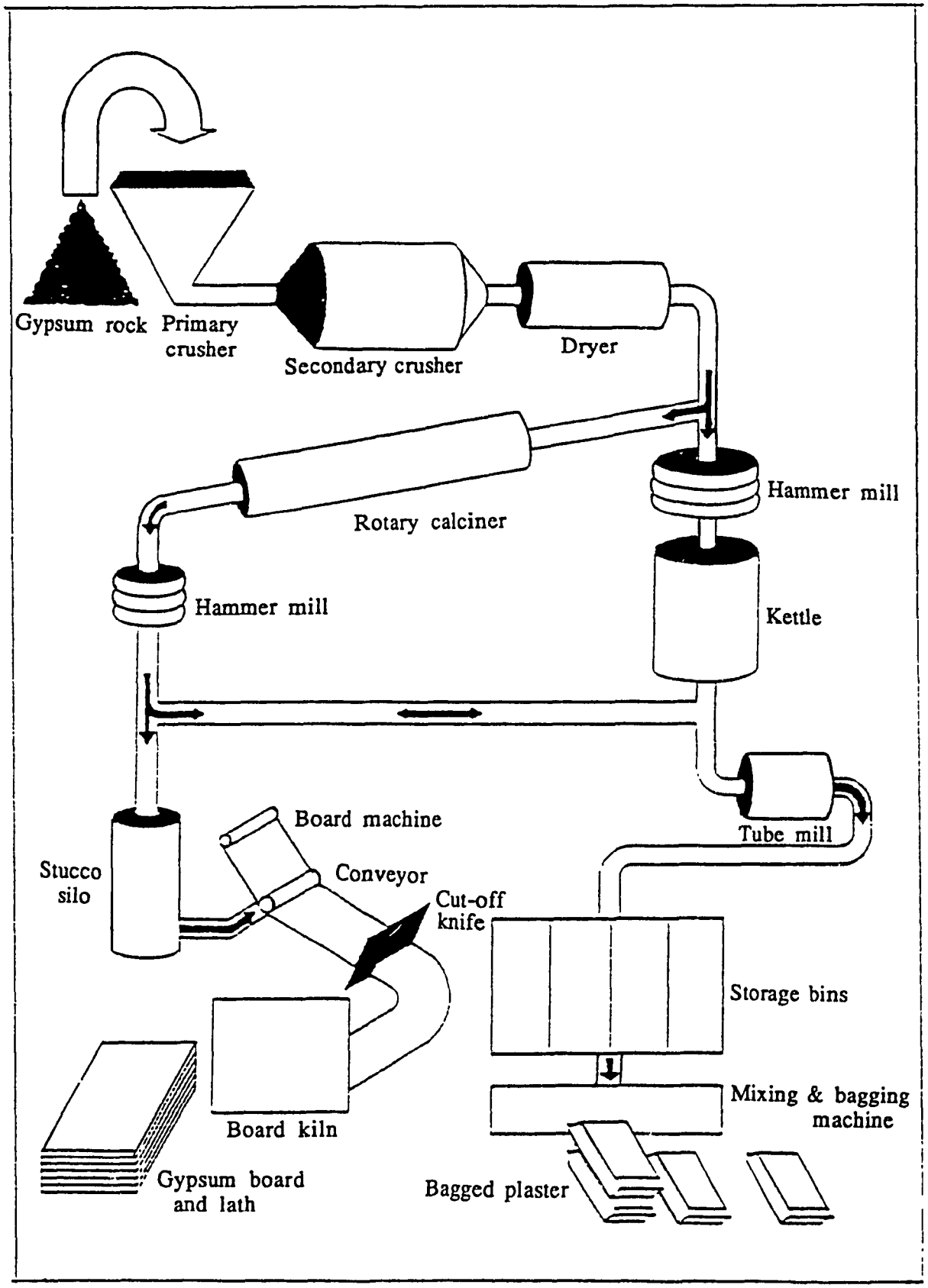


Fig. 2.1 Schematic process of gypsum production

Two forms of hemihydrate can be identified as (α) and (β). In practical terms α hemihydrate is distinguishable from β in that there is very little particle disintegration when it is mixed with water. The α form of hemihydrate requires far less mixing water to form a workable slurry. Consequently, it has the ability to produce denser and higher compressive strengths and it requires less free water than the β form for slurry preparation. The free water has to be removed after hydration is complete [3].

2.1.6 Physical properties of gypsum

Calcium sulphate dihydrate forms crystals with a density of 2.3 g/cm^3 and a hardness of 1.5 to 2 on the Moh hardness scale. The naturally occuring masses of very small crystals are white or nearly white. The compressive strength of hardened gypsum depends on the quantity of water used in the paste before setting and on the presence of additives or fillers.

The compressive strength of calcined gypsum from various sources mixed with an equal weight of water may vary from 30 to 80 Kg/cm^2 after hardening, (values are based on completely hardened products).

In the presence of 1% moisture, the compressive strength decreases to about 40% of that of the dry product because of the decreased friction between the crystals. Often, no further decrease of strength is observed when the moisture content increases over 1%. The bending strength of the hardened gypsum is usually about 50% of the compressive strength [1].

2.1.7 Chemical properties

Gypsum is relatively inert towards chemicals such as acids, alkalis and oxidizing agents.

Solubility of calcined gypsum is about 8 g/litre of water at 20°C. Calcined gypsum is rapidly converted into calcium sulphate dihydrate in presence of water as shown in the following reactions below:

$$CaSO_4 \cdot 1/2H_2O + 3/2H_2O \text{ ----------> } CaSO_4 \cdot 2H_2O$$

$$CaSO_4 + 2H_2O \text{----------> } CaSO_4 \cdot 2H_2O$$

On the other hand, calcium dihydrate when heated will proceed through following the reactions:

$$CaSO_4 \cdot 2H_2O \text{ ----------> } CaSO_4 \cdot 1/2H_2O + 3/2H_2O$$

$$CaSO_4 \cdot 2H_2O \text{ ----------> } CaSO_4 + 2H_2O$$

$$CaSO_4 \text{----------> } CaO + SO_3$$

2.1.8 Mechanism of setting

The most important property of calcined gypsum is its hardening or setting characteristics when mixed with water. The setting is based on two processes:

i) The chemical process of combining with water to form calcium sulphate dihydrate and;

ii) The physical process of crystallization of the dihydrate from the aqueous solution.

Calcined gypsum and precipitation of calcium sulphate dihydrate, which is in the form of fine fibrous crystals, are responsible for the formation of a solid mass.

In a large excess of water, both the hydration and the crystallization processes occur but a hard mass is not formed since the fibrous crystals cannot interlock. On the other hand, if the quantity of water is too low, the hydration process is not complete and an inhomogeneous, hardened product results. Usually about 40 to 50 grams of water per 100 grams of calcined gypsum are required to obtain an optimally hardened product.

The time needed to obtain a solid mass (*setting time*) from which no water can be liberated by pressing with a finger, is 4-5 minutes. The total hardening time is much longer and increases until the last traces of free water are evaporated. Based on necessity, the process of crystallization, and thus the setting, can be accelerated or retarded by using small quantities of accelerators or retarders [11].

2.1.9 Uses [1,3,12]

The major fields of use of calcined gypsum are;

i) The building industry (which is the largest user of calcined gypsum) for such uses as:
   - Plaster for interior coatings on lath, bricks, concrete, and other materials;
   - Prefabricated building products like bricks, wallboards, wallpanels and floor tiles;
   - Sound and heat insulators when mixed with a porous material such as vermiculite and perlite;

ii) As a retarder in the manufacture of portland cement;

iii) As a filler in paint and paper manufacture;

iv) As a soil conditioner in agriculture;

v) In the production of ammonium and calcium sulfate;

vi) For the hardening of the water in the beverage industry;

vii) In the pharmaceutical industries.

2.2 SYNTHETIC GYPSUM

Byproduct gypsum (BPG) i.e., phosphogypsum, fluorogypsum, titanogypsum, desulphogypsum, are all the chemical end products of industrial processing plants consisting primarily of calcium sulphate dihydrate ($CaSO_4 \cdot 2H_2O$). The production of BPG in United States in 1982 is shown in Table 2.1.

Table 2.1
Byproduct gypsum produced in the United States
(millions of metric tons) [7].

Kind	Quantity
Phosphogypsum	30
Fluorogypsum	2
Titanogypsum	1
Desulphogypsum	3
Other (from citric acid, boric acid, and other organic acid production)	0.7
Total	36.7

A brief review of BPGs is useful to obtain an insight on gypsum products.

2.2.1 Phosphogypsum

Phosphogypsum (PG) is a byproduct of phosphoric acid production. For every ton of phosphoric acid produced, approximately 4.5-5.5 tons of PG is generated. Consequently, long term projections predict that over $1x10^9$ tons will be stockpiled in the United States by the year 2000. Acidulating phosphate rock involves digesting a slurry of phosphate rock with sulfuric acid and separating the resulting phosphoric acid from the solid reaction products by filtration. The filter cake contains calcium sulfate, the degree of hydration of which depends on the operating acid concentration and temperature. Typical reaction temperatures for the dihydrate process range from 57-71°C and results in P_2O_5 concentrations ranging from 28 to 32% [10]. The reaction is shown below:

$$Ca_3(PO_4)_2 + 3\ H_2SO_4 \text{ --------> } 3\ CaSO_4 + 2\ H_3PO_4 \cdot 2H_2O \qquad [13]$$

The hemihydrate process was initiated in Japan and subsequently was adopted in Europe and it is used to a limited extent in the United States. This process produces higher quantity material by controlling the conditions to get $CaSO_4$ in a form of hemihydrate. The reaction temperature for this process ranges from 76-79°C.

$$Ca_3(PO_4)_2 + 3\ H_2SO_4 \text{ --------> } 3CaSO_4 \cdot 1/2H_2O + 2H_3PO_4 \qquad [13]$$

Material characterization of Mobil chemical company PG has shown that its byproduct gray color PG consist of the following components [10].

CaO	32.5	%
SO_3	44.00	%
P_2O_5	0.65	%
F_2	1.2	%
SiO_2	0.5	%
Fe_2O_3	0.1	%
Al_2O_3	0.1	%
H_2O (crystallization water)	19.00	%
Others	1.95	%

Beside this composition, studies have indicated that a potential radiation hazard exists in the use of PG produced from sedimentary phosphate rock [4, 9] which contain radium (Ra^{226}) and uranium (U^{238}) contamination [5, 8]. Some of the major contaminants as well as their harmful effects are summarized in Table 2.2.

2.2.2 Fluorogypsum

Fluorogypsum (FG) is a byproduct of the manufacture of hydrofluric acid, through the chemical reaction shown below.

$$CaF_2 + H_2SO_4 \text{ ---------> } CaSO_4 + 2HF \qquad [13]$$

Cooperative research programs have been conducted to determine the suitability of using waste FG obtained from a plant in Ontario (Allied Chemical Canada Ltd, Amherstburg, Ontario Plant at St-Lawrence cement Inc) [4].

Table 2.2
Harmful constituents of phosphogypsum [10].

Contami-nant	Form	Characteristics	Harmful effect
P_2O_5	1) H_3PO_4 in the voids between solid particles.	Water soluble	Increases corrosion.
	2) Ca slat occluded in dihydrate crystals.	Citrate soluble sp. gr. 2.32,	For gypsum board;decrea-ses setting time,strength and adhesion.
	3) Unreacted rock	Insoluble large particles; sp. gr. 2.74	For cement (retarder): decreases strength.
Uranium	1) Uranium phosphate dissolved in the acid and present in the voids.	Water soluble	For road construction: decreases strength, radioactive
	2) Unattacked rock	sp. gr. = 2.74	
Radium	1) $RaSO_4$ suspended in the acid in the voids.	Separate heavy particles concen-trated in fine fractions	Radioactive & produces "radon" gas
	2) $RaSO_4$ precipita-ted with gypsum.		
	3) Unreacted rock.		

2.2.3 Desulphogypsum (DSG)

The use of lime or limestone to desulphurize stack gases from utility or industrial plants burning high sulphur fuel results in the production of large amounts of waste gypsum in the form of sludge called flow-gas DSG. It may present a disposal problem if profitable uses are not developed [12]. The reaction which takes place in the process is given below:

$$CaO + SO_3 \text{ ----------> } CaSO_4 \qquad [13]$$

DSG is obtained as a moist, finely divided powder from the desulphurization of combustion gases in power plants. Of all the numerous processes, desulfurization with lime stone ($CaSO_3$), calcium hydroxide $Ca(OH)_2$ or lime (CaO) has proven to be the best and is extensively used. The end product after desulfurization is a mixture of calcium sulfite and calcium sulfate or, after oxidation with oxygen from the air, calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$), so called flue gas gypsum. Reactions taking place in the desulfurization process can be represented by the following equations as shown below:

$$SO_2 + CaCO_3 + 1/2H_2O \text{ ----------> } CaSO_3 \cdot 1/2H_2O + CO_2$$
$$CaSO_4 \cdot 1/2H_2O + SO_2 + H_2O \text{ -------> } Ca(HSO_3)_2 + 1/2H_2O \qquad \}[14]$$
$$Ca(HSO_3)_2 + O_2 + 2H_2O \text{ ----------> } CaSO_4 \cdot 2H_2O + H_2SO_4$$
$$CaO + SO_3 \text{ ----------> } CaSO_4 \qquad [13]$$

2.2.4 Use of BPG

In spite of many deleterious contaminants in the BPG and without thorough purification of the resulting waste byproducts, many industrialized countries like Japan, USA, France, United Kingdom, are using BPG mixed with or completely replacing natural gypsum. For example

i) The production of BPG briquets for use as a set retarder in the cement industry by the American Cyanamid Co. and Lemco Inc in 1980 [7].

ii) The use of over 60,000 tons of BPG with a gypsum rock raw material feed for the production of wallboard by The United States Gypsum Co's (USG), in 1982 [7]. The utilization of BPG demonstrates how improved technology can be used to recycle a solid waste or sludge material.

iii) Tampa Electric Company's BPG (180,000 metric tons in 1985) was purified and used for the production of wallboard, and briquets.

iv) In Japan, the technology of utilization of BPG developed rapidly and various types of BPG are now fully accepted for use in cement, wallboard and plaster.

v) In 1981, Western Europe produced 23 million tons of BPG of which only 871 tons was PG. Out of a total 25 million tons consumed 2.5 million metric ton was BPG.

vi) In France, BPG is used in the form of cellular (foamed) gypsum plaster for pouring floors and walls in place and as prefabricated blocks.[7]

vii) Construction blocks for floor and wall construction and partition blocks for interior use are in common use in North Africa, the Mediterranean Countries and the Middle East.

2.3 SURFACTANTS

2.3.1 Introduction

The term surfactant, derived from the term surface-active-agent, technically defines a group of compounds which, when dissolved in water or other solvent, orient themselves at the liquid interface with other surfaces and effect a modification of the liquid properties at that surface [17].

This modification generally is evidenced by increased solubilization, foaming, emulsification, wetting, dispersing or penetration power of the solution.

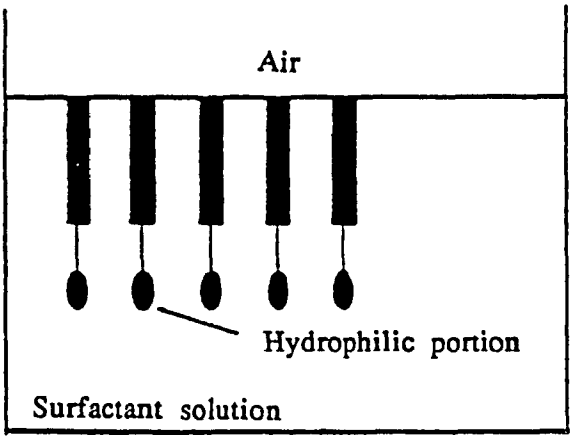
Molecularly, all surfactants have a common structure, being composed of a long hydrocarbon chain which is oil-soluble and a terminal group, which is water-soluble. This end group confers water solubility on the entire molecule. The molecule may be depicted structurally as shown in Fig. 2.2a.

Surfactants achieve their unique effects in solutions because of the orientation of these molecules. At the solution interface with a second phase, the oil soluble (*hydrophobic*) portion is thrust outward and the water soluble (*hydrophilic*) group downward into the solution (Fig. 2.2b). Close packing of molecules can be achieved with this geometry, leading to a higher concentration of surfactant at the interface than in the bulk solution. A marked reduction in surface tension results which promotes improved penetration and oil

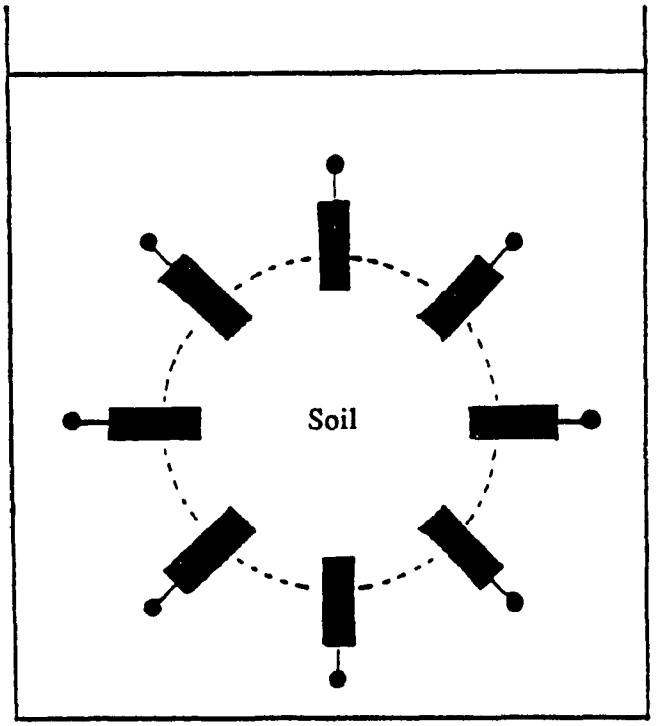
emulsification. Thus the surfactant wets particulate matter so that, upon application of mechanical energy (hand, machine), the particle is removed easily and dispersed in solution [17]. In solvents such as water the surfactant molecules are distributed in such a manner that their concentration at the interfaces is higher than in the inner regions of the solution. This behavior is attributable to their amphiphilic structure (*hydrophilic-hydrophobic*).

At the phase borders, an orientating alignment of the surfactant molecules occurs. This results in a change of system properties, i.e a lowering of interfacial tension between water and the adjacent phase, a change of wetting properties as well as formation of electrical double layers at the interfaces. Within the solution surfactant micelles [Fig.2.2c] form, provided a certain surfactant concentration is exceeded [16,17].

(a)



(b)



(c)

Fig. 2.2 (a) Diagrammatic representation of hydrophobic group (at left) hydrophilic group (at right). (b) Oreintation of surfactant molecules at air/solution interface.(c) Micelles of surfactant disolved in a solution.

2.3.2 Raw Materials

Starting materials for surfactants generally belong to one of the following raw material families:

- Fat derived chemical raw materials based on vegetable and animal fats and oils.
- Petrochemical raw materials based on crude oil and natural gas.[16]

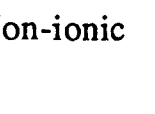
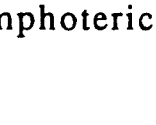
2.3.3 Classification

Chemically, surfactants may be classified as anionic, cationic and non-ionic. Anionics and cationics ionize in aqueous solution, while non-ionics do not, although a wide range of polarity does exist. Anionic surfactants are the least expensive and constitute the largest group of surfactants used commercially. Commercially available surfactants are not uniform substances but are mixtures of homologous substances, e.g. having chain lengths C_8-C_{18}. Table 2.3 gives some examples of major types of surfactants.

The most general use for the sulfated fats and oils are as emulsifiers. Monobasic esters of oleic acid, such as butyl oleate are sulfated also producing wetting and rewetting agents. On the other hand, sulfated alcohols have been shown to have very good foaming ability with high foam stability. Alkylarylsulfonates act as wetting agents and general surfactants. Sodiumdioctylsulfosuccinate acts as a superior wetting agent to that of sulfoethylesters and amides are good dispersing agents [15].

Table 2.3

Examples of surfactants [16,17].

Type of surfactant	Hydrophobic group	Hydrophilic group	Chemical name
Anionic	$C_{12}H_{25}O\text{-}SO_3^-$	SO_3Na	Sodium dodecyl
	$R\text{-}C_6H_4$ ($R = C_{10\text{-}13}$)	SO_3Na	Alkyl sulfate benzene sulfonate
Cationic		Cl^-	Tetra alkyl amonium chloride
Non-ionic		$R = C_{6\text{-}16}$	Fatty alcohol poly ethylene glycol ether
	$R\text{-}C_6H_4\text{-}O\text{-}(CH_2\text{-}CH_2\text{-}O)_nH$	$R = C_{8\text{-}12}$	Alkylphenolpoly ethylene glycol ether
		$n = 5\text{-}10$ $R = 11\text{-}17$ $n = 1\text{-}2$ $m = 1$	Fatty acid alkanolamides
Amphoteric		$R = 12$	Dimethyldodecyl ammonium proprionate

2.3.4 Application of surfactants

In all industrial processes that take place at interfaces, surfactants are useful because work processes may be simplified, accelerated or economized. Surfactants are applied in a number of different building or construction materials e.g:

i) As dispersants for the preparation of bitumen emulsions in road construction;

ii) In the utiiization of polymer dispersions;

iii) As additives to plasters and cement coatings and in wood impregnation;

iv) In the manufacturing of concrete and mortars to improve wetting of the solid constituents such that the workability of the mixture is increased

v) Combinations of low-foaming surfactants and defoamers play a role in the liquification of cement. (Cement liquifiers are intended to reduce the quantity of makeup water). Suitable liquifiers are phenol formaldehyde-bisulfide and melamine formaldehyde-bisulfite condensation products.

vi) To prevent frost damage in concrete, so called air entrainment additives are utilized. These are, for the most part, foaming surfactants such as alkylsulfates, lignosulfonates or protein degradation products. Also applicable for the formation of air porous concrete are nonylphenolpolyglycolethers. They are utilized in plaster, paint and coatings and especially in the production of GB. [16]

2.4 FOAM AND FOAMING THEORY

2.4.1 Introduction

Since foam is a primary constituent of gypsum board, an understanding of the theory of foam and foaming is essential for the proper control of quality and density of the product. The term "*foam*" as defined by ASTM, implies a mass of bubbles formed on liquids by agitation. A foaming agent (FA) is a material that increases the stability of a suspension of gas bubbles (*foam*) in a liquid medium.

The formation of foam or foaming may be controlled by either antifoaming or foam stabilizing agents, depending on whether a particular industrial process requires, as in the former case, a reduction of foam formation or, as in the latter case, foam stabilization.

In the production of GB it is common practice in the wallboard industry to incorporate air bubbles in the core in order to decrease the density of the gypsum core. This is normally achieved by the addition of a pregenerated foam to the stucco mix along with other additives, immediately prior to core formation. This foam is normally generated, by incorporating air, in an aqueous solution of foaming agent, into a foam generator [18]. In this way, solid cellular structures are manufactured by dispersing a gaseous phase within a fluid mass which subsequently becomes solid. The fluid may be a homogeneous hot melt which solidifies on cooling or a liquid which polymerizes chemically. It may also be a heterogeneous slurry of fine particles and liquid, as in the case of GB, which stiffens and sets to a solid [19].

2.4.2 Foam Production

Foam may be produced by either chemical or mechanical means, depending on the process used to produce the gas voids.

Chemically formed solid foams result from a gas evolving from a chemical reaction within the initially fluid medium. The gas forms bubbles which are then trapped within the slurry. The resultant foaming products are then allowed to set. With chemical foaming methods, the homogeneity of the cellular structure depends on the rate and degree of completion of the chemical reaction. This, in turn, varies with the temperature, pH, water hardness, the relative amount of the reactants and other process variables.

Mechanically formed foams are made by introducing the gaseous phase from an external source into the fluid mass before it solidifies, and may be generated "*in situ*" or preformed. The density and stability of the resulting foam depends on the amount of FA, its chemical composition, the intensity and duration of mixing, as well as the physical and chemical nature of the solids being dispersed.

Extensive research on the application of chemically generated foams to the production of CB has been carried out [2]. For example slurries of calcined gypsum may be foamed by incorporating aluminum sulfate and calcium carbonate in the slurry which releases CO_2 gas in the presence of the water in. Another method is by the catalytic decomposition of hydrogen peroxide (H_2O_2) to oxygen by means of a manganese dioxide catalyst [17]. But chemical methods have not received wide acceptance in the GB industry due to quality problems associated to each method. The mechanical preformed foam

blending method lends itself readily to continuous operations and is the method most applicable to GB industry [19].

2.4.3 Foam generating system in the manufacturing process

In the manufacturing process the foam generating system is normally made up of two connected systems, i.e. the batching system and the generating system. The function of the batching system is to prepare an aqueous dilute solution of foaming agent from the FA concentrate . The percent concentration by weight of the solution can be varied according to mixing requirements.

The batching system consists mainly of one mixing tank and two batching tanks and 2 or 3 mixers, depending on the rate of production. Finally, a supply line leads to the generating system. The generating system combines the dilute aqueous solution of FA with air in fixed proportions to form a highly stable foam of the required density. The resulting foam is then metered into the gypsum slurry as it is formed at a continuously controlled flow rate. The system consists mainly of a metering pump which continuously meters out a constant flow rate from one of the batching tanks and feeds it into two centrifugal pumps connected in series. Air is fed into the first pump and foam is conveyed into the pin mixer at the board line.

2.4.3.1 Characteristics of generating system

i) The system should be capable of producing stable foam in a density range of 0.05 to 0.15 g/cm^3 at various solution flow rates.

ii) The air bubbles within the generated foam are to be less than 0.4 mm in diameter [19].

iii) The system should be capable of producing the above densities by utilizing a foaming agent solution of no greater than 1% concentration by weight (For economic reasons).

2.4.3.2 Physical characteristics of the foams

All foams have essentially identical structures: a honey-combed arrangement of gas bubbles separated by thin liquid walls (*lamellae*). Such a foam is a dispersion of gas in liquid and is a thermodynamically unstable two phase system. The disintegration of a foam progresses by the breakdown of foam lamellae, which is considered to be the consequence of statistical fluctuations in the thickness of the wall. The low foam stability of aqueous solutions of short chain fatty acids and fatty alcohols is due to this mechanism [15].

To stabilise the foam (i.e. to prevent disintegration) use is made of the addition of a small amount of FA (which are essentially long chain fatty acid salts and alcohols). More persistant foams are formed when solutions of colloidal materials are foamed.

However, foam stability is the result of the interaction of several of the following factors including:

i) Surface tension: For a foam to be stable, the surface tensions of the solution must be considerably less than that of the pure solvent

ii) Viscosity: A foam is stable if the gas bubbles do not coalesce, but remain separated by thin liquid walls. If the viscosity of

these liquid partitions is sufficiently high, foam persistence is enhanced.

iii) Surface area; liquid films of small surface area are more stable than those of large areas. In general, the smaller the bubble, the greater the persistence.

iv) Temperature: Foam persistence generally increases with decreasing temperature, probably owing to the combined effects of increased viscosity in the liquid film and decreased gas pressure within the bubbles [19].

v) Concentration: For highly surface active FAs there exists an optimum concentration, i.e. one which yields the greatest foam persistence (foam generated at higher concentration is less stable). Surfactants are characterized by a marked reduction in surface tension over a very narrow concentration range.

2.5 GYPSUM BOARD MANUFACTURING

2.5.1 Introduction

The manufacture of GB originated in the USA in the early 1900's. The earliest GB was known to the trade as a base for plastering. In the 1920's, GB in large sheets and with improved surface finish, was brought to market, permitting direct application of decorative finishes without plastering. By far the larger proportion, of GB (85%) now goes to such "*dry wall*" construction [20].

GB is most commonly made in a width of (125 or 120 cm) and in lengths of 244-488 cm. Depending on the intended application, it is made in thicknesses of 6.4 mm (1/4"), 9.5 mm (3/8"), 12.7 mm (1/2") and 15.9 mm(5/8"), the 12.7 mm thickness being the most commonly used among them.

2.5.2 Board Manufacturing

GB products are made in panels or slabs consisting of a noncombustible gypsum core which is surfaced and edged with covering material specifically designed for various uses with respect to performance, application, location and appearance [1]. GB products, produced for specific uses in building construction. are:

i) Wallboards: These are products with prefinished surfaces or with surfaces suitable for paint finishing after appropriate fastener and joint treatment.

ii) Sheathing boards: These are used in exterior frame walls for wind bracing, as a base for finish materials and for increased fire resistance.

iii) Backing Boards, provide a base for adhesive application of wallboards in multiply construction and of acoustical tiles in suspended ceilings.

iv) Form boards, serve as both permanent forms and finished ceilings in poured gypsum concrete roof decks.

Board products are made in different sizes, thicknesses and edge finishes, typically with square, tongue and groove or beveled edges. Fig 2.3 shows some typical edge designs.

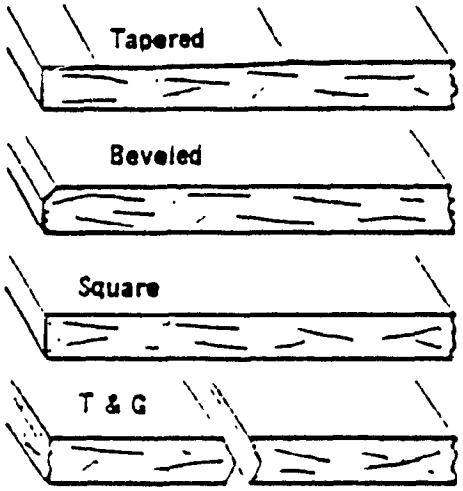


Fig 2.3 Typical edge design for gypsum board products [1].

GB is manufactured by a high speed continuous method. Minor solid ingredients are metered volumetrically into the main stucco stream which, in turn, is usually metered by a variable speed auger conveyor. The stucco, water and separately generated foam are introduced through separate ports into a horizontal, high energy pin mixer.

Thorough blending is accomplished even though the residence time in the mixer is of the order of only 2 seconds. The slurry is discharged and spread between two continuous paper sheets and completely enveloped. In the 3 to 4 minutes required for the GB to move down the belt conveyor, the core material sets and is firm enough to be cut to the desire length by a rotary cutter. The sheets are then inverted and fed into a multi deck kiln dryer. The dryer is usually heated directly, using two to four heat zones where free water is evaporated. Finally, boards not requiring additional finishes, may be end trimmed and automatically bundled and stacked for shipment. Others may receive special backings such as aluminum foil, decorative vinyl paper or organic texture coating. Prior to shipment, boards are conveyed to a special location to be treated [3,4].

2.5.3 Gypsumboards

The main types of gypsumboard are:

- Regular gypsumboard;
- Predecorated gypsumboard;
- Foil backed gypsumboard;
- Fire-resistant gypsumboard; and
- Water-resistant gypsumboards.

2.5.3.1 Regular gypsumboard

Regular gypsumboard is surfaced on the back with a gray, liner paper and on the face and along the longitudinal edges, with a calendered manila paper which provides a smooth, even finish suitable for decorating. In construction, wallboards are nailed, screwed or adhesively bonded to supporting wood or metal studs or to furring.

2.5.3.2 Pre-decorated gypsumboard

This board is essentially regular GB with a factory-applied decorative finish on the face of the board. The common finish materials are paper and vinyl, furnished in a variety of colours, patterns and simulated wood finishes.

2.5.3.3 Foil-backed gypsumboard

Foil-backed board is regular GB with a bright-finish aluminum foil bonded to the back. The foil backing serves as a vapor barrier; its use eliminates the need for a separate vapor barrier in an exterior wall assembly.

2.5.3.4 Fire-resistant gypsumboard

Fire-resistant gypsumboard is similar in exterior covering, appearance, edge treatment and installation to regular GB, but it has a core specially formulated with additives and glass-fibers for greater strength.

2.5.3.5 Special types of gypsumboard

Special types of gypsumboard are produced by some manufacturers for areas such as bathrooms, kitchens and utility rooms. Water resistance is achieved by using a multi-layered covering of chemically treated paper and a gypsum core formulated with asphaltic additives. However, gypsum's inherent qualities such as incombustibility, fire resistance and relatively light weight make it highly useful in a variety of other building products such as partition products, roofing materials, ceiling boards, plaster products, etc. In the proposed production of energy storing GB it is assumed that the manufacturing procedure shall be the same as standard GB

production except that an extra port is added for metering the quantity of PCM introduced to the pin mixer.

2.6 PHASE CHANGE MATERIALS (PCM's)

2.6.1 Introduction

In the past, materials have been used for reasons of strength, durability, availability, design and cost. But there has been little consideration for the flow of energy through them. Furthermore the energy crisis of the 70's, characterized by shortages and price escalations, brought about the need for alternative energy sources, (solar, biomass, geothermal, wind, etc.) better energy management and the serious emergence of conservation.

The ability to store energy efficiently has been the key to success in many of mankind's industrial ventures. So energy research has been a very active field for the past few decades and fruits of this effort are being increasingly integrated into our daily lives [21,22]. Until fairly recently research in thermal energy storage lagged that of the other areas of energy research. Now much of the emphasis of this research has been to develop materials which by the nature of their phase-change process are well suited for storing thermal energy [23]. It has included the use of active solar systems with energy storage, including the use of passive solar systems moreover, instead of designing buildings for direct gain, which usually includes massive components made of concrete, brick, etc. (to absorb the solar heat) it is possible to use well designed phase change thermal storage materials to store the same amount of heat in much less massive

components [27]. However, a variety of building materials and techniques have been used to conserve heat or cool buildings and thereby reduce energy cost. Included among them are structural elements which incorporate PCMs. However, nowadays heat storage is considered more and more to be a serious option for saving energy and research on PCMs, and system is quite active and should lead to a number of interesting developments.

2.6.2 Phase Change Materials (PCM's)

Physico-chemical phase changes, such as melting and dehydration reversibly absorb large amounts of thermal energy when the material changes its form [24].

Extensive research carried out on phase change materials has shown that, during heating, a large amount of energy is absorbed by these materials in transition between phases and that this same energy can be released during the cooling cycle. The transitions of phase can be from solid to liquid or from one solid state to another. This phase transition shows a relatively high heat storage capacity within a narrow transition temperature range. The heat storage capacity of the PCM within the range where the phase transition occurs will be higher than sensible heat storage for the same volume.

Various PCMs have been investigated in the range of-20 to +140°C [28]. They can be used directly for either cooling or heating purposes and can roughly be divided into two groups:

i) Organic PCMs (e.g. paraffin waxes, fatty acid and esters, etc.);

ii) Inorganic PCMs (e.g. salt hydrates).

2.6.2.1 Organic PCM

Many organic phase change material groups have been studied. Polyhydric alcohols [28], paraffins and hydrocarbons [26], several fatty acids [21,26], certain organometallic compounds and crosslinked polyethylenes [24]. Among these groups certain promising materials have been categorized according to specific applications. As has been mentioned, two types of transitions may take place in PCMs due to their physico-chemical characteristics, i.e. solid to solid (S-S) or solid to liquid (S-L) transitions.

S-S PCMs are crystalline solids that undergo changes in their structures at temperatures well below their melting points. These materials remain solid throughout the range of their service temperature [25].

Although S-S PCMs offer the advantages of less stringent container requirements and a greater design flexibility, the heat of transition for S-S transformation of these materials is lower than for S-L transition.

These transition materials have a very small transition range or transition temperature (like naphthalene and salt hydrates) and quite often show supercooling. The materials which have a wider melting range, say from 10 to 20°C (e.g. waxes), show a second peak (Fig. 2.4) which is usually a S-S transition for both low and high temperature PCMs [28]. With the paraffins, the main difficulties encountered are due to their large volume change during phase transition and to their low thermal conductivity [21].

2.6.2.2 Inorganic PCM (salt hydrates)

Although all the salt hydrates are potentially attractive (e.g. $Na_2SO_4 \cdot 10H_2O$) because of their large latent heat and high energy density, they have several disadvantages. It has been shown that after repeated cycling, incongruent melting and phase separation often occur with sodium sulfate decahydrate ($Na_2SO_4 \cdot 10H_2O$), calcium chloride hexahydrate, sodium phosphate dodecahydrate and sodium thiosulphate pentahydrate [26].

Among salt hydrates, Glauber's salt ($Na_2SO_4 \cdot 10H_2O$) is one of the most extensively studied PCMs for solar energy storage, because of its low price, suitable phase change temperature, high latent heat, and availability as a suitable nucleating agent (borax). As has been mentioned previously, the major problem associated with Glauber's salt is segregation due to incongruent melting and super cooling. When $Na_2SO_4 \cdot 10H_2O$ is heated above 32°C, its transformation temperature, the water of crystallization can no longer remain bound to the solid lattice and a forward reaction takes place [22] i.e:

$$Na_2SO_4 \cdot 10H_2O \text{ ----------> } Na_2SO_4 + 10H_2O$$

About 15% (wt) of the mass of Glauber's salt forms insoluble, anhydrous Na_2SO_4 crystals in a saturated Na_2SO_4 solution upon melting [29]. The anhydrous crystals precipitate, because of their higher density in relation to the saturated solution. During the cooling cycle, the upper part reforms $Na_2SO_4 \cdot 10H_2O$ crystals, blocking diffusion of water to the inner part of the precipitate and

prevents them from rehydrating. Therefore, the result is a loss of thermal storage efficiency from cycle to cycle [29]. However, these problems have led to renewed attention for organic PCMs.

2.6.3 Incorporation of PCM

The change in form requires that these PCMs be housed in some kind of durable and leak proof container. Solid state PCMs may in some cases be more readily incorporated into structures and components than can S-L PCMs. However, if properly selected, S-L PCMs can function very well in porous materials such as gypsum and concrete because, even in this liquid state, the PCM will be retained in the structure of the host material by virtue of surface tension. The S-S PCMs may be melted and then cast into the component or composite structure [25].

It would be very desirable to achieve the same large energy storage density in a solid state PCM which did not change its form during the transition and therefore require a less secure containment. However, many solids undergo reversible phase changes in the solid state and very few have a sufficiently energetic transformation to be of potential practical use in thermal energy storage [24].

However, encapsulation of PCMs in spheres, tubes, trays or cans has been the method of study and commercialization until recently. Encapsulation has particular advantages in passive designs such as in Trombe walls or partitions where storage is needed. It also is applicable to air type solar collectors. In the case of porous structures like concrete, brick, gypsum board, etc., direct incorporation or

immersion of the preformed porous structure in the molten PCM is presently under laboratory investigation. Development of a bulk storage design for active solar storage is also under investigation.

2.6.4 Function of PCM

The simplest way of storing thermal energy is as sensible heat, where a material like concrete, rock, water absorbs energy by increasing its temperature without undergoing any change of phase. However thermal energy storage by phase change energy (latent heat) of a suitable material has the advantages of higher energy density (Fig 2.5), giving considerably smaller volumes and relatively isothermal behavior [29]. It is possible, in principle, to use any reversible change with high heat of absorption or release, but S-L phase changes are generally preferred, for space heating and domestic hot water needs.

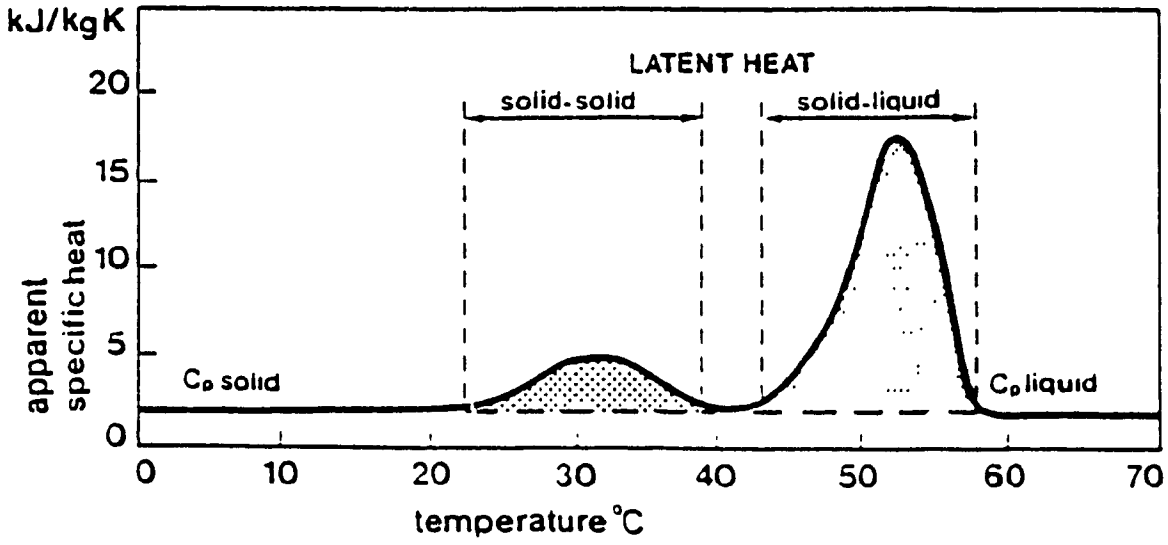


Fig.2.4, DTA curve of Shell paraffin wax.52/54 showing a S-S & S-L phase transition [28].

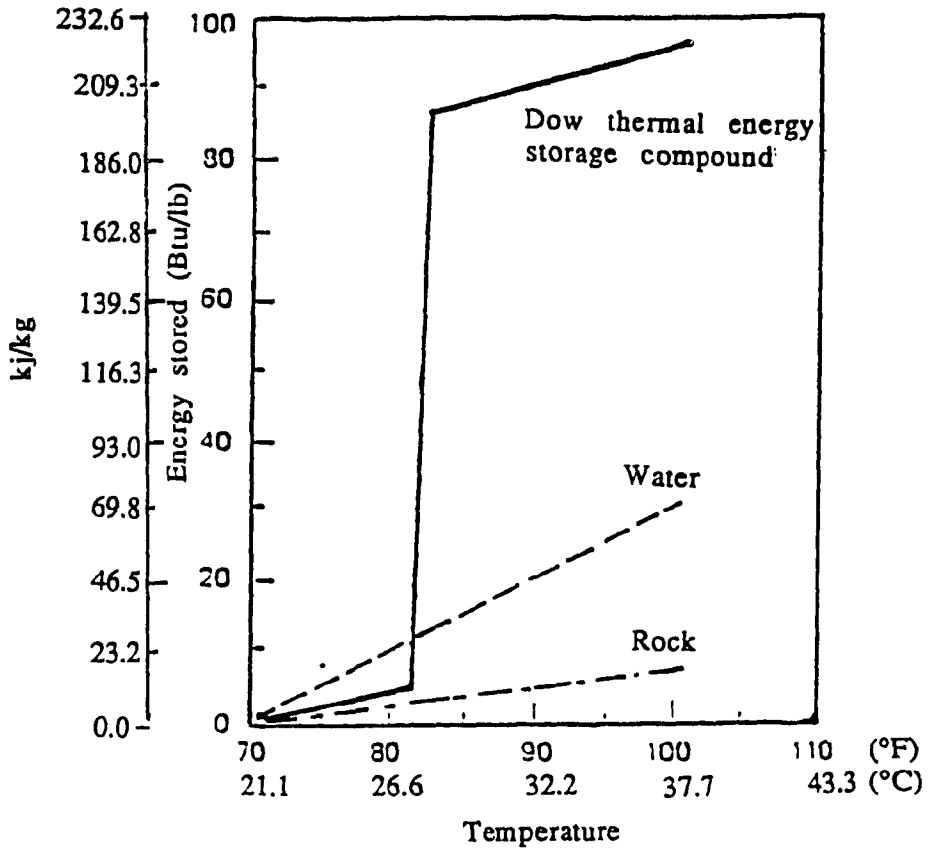


Fig.2.5, Comparison of Storage Materials [10].

There is a great deal of interest in phase change thermal energy storage systems due to their inherent ability to store large amounts of heat and to release it to the surrounding environment as temperatures drop below predetermined levels. These systems are of particular interest in the architectural and building trades where climate control and concommitant energy consumption is one of the principle considerations in building design and material selection [32].

By incorporating PCMs into building materials, energy in excess of that necessary to maintain comfort conditions is inherently absorbed and subsequently released when the surrounding environment drops below the comfort range. Thus in winter months, PCMs incorporated

into structural elements in the walls or floors of buildings and the like, can absorb solar energy as well as excess internal space energy during daytime hours and release it to the interior at night as temperatures drop. In the summer months the same PCM, due to its thermostatic character, conserves coolness by absorbing energy.

Materials with melting points in the range of 18-65°C can be used for passive structural and solar heating units and the higher melting materials are particularly aimed at applications with high performance collectors in solar-cooling and industrial processes [26].

2.6.5 Characteristics of PCM

An appropriate PCM should meet the following requirements:

i) The transition of phase should be within a temperature range over which the complete system can operate both for charging and discharging energy.

ii) The effective thermal conductivity for charging and discharging should be sufficient.

iii) The stability of the storage material is important to ensure that the heat storage capacity does not show a significant decrease over the required life time.

iv) Container materials have to be available which do not interact with the storage materials and which are strong enough to prevent leakage problems over their life time.

v) The flammability and toxicity should meet the legal requirements and the PCM should have little or no odour.

vi) Small temperature difference between storage and heat transfer media.

vii) High rate of heat transfer between storage material and heat transfer medium.

viii) Low cost of PCM and low capital cost.

ix) Efficient use of storage volume.

x) Low cost of operation and maintenance [28].

2.6.6 Raw Materials for PCM

Fats, oils, and waxes belong to the group of naturally occuring compounds called lipids. Lipids are those constituents of animals and plants which are soluble in organic solvents but insoluble in water. The lipids, which yield fatty acids and alcohols on hydrolysis with aqueous base (saponified), are referred to as simple lipids. These can be further divided into two classes:

i) fats and oils which, upon hydrolysis, yield long-chain fatty acids and glycerol and;

ii) Waxes which, upon hydrolysis, yield long-chain fatty acids and long-chain alcohols [33].

Fats and oils are the most important lipids found in nature. At the present time, the world consumption of fats and oils is estimated at 50-55 million tones per year which reflects both their nutritional and industrial importance.

2.6.6.1 Natural sources of fats and oils

Fats and oils come from animal and vegetable sources:

i) Animal fat in the form of tallow is obtained from cattle, sheep and goats, as well as in the form of lard from hogs.

ii) vegetable oils, which are chiefly present in seeds and nuts of plants such as soya bean, ground nuts, coconuts, palm kernel, sesame and mustard seed, are important sources of edible oils; cotton seed, lin seed and castor seed yield non-edible oils.

iii) Marine oils, which are obtained from sardines, herrings, salmon, whales, dolphins, seals, etc.

2.6.6.2 Production of fats and oils

Animal fats are recovered from selected animal material by rendering, whereas vegetable and marine oils are processed by solvent extraction. These methods are followed by refining in both cases. The hydrolysis of fats and oils gives long chain fatty acids [34].

2.6.6.3 Fatty Acids

A number of higher molecular weight (MW) carboxylic acids (fatty acids) occur in nature in the form of esters with glycerol or higher MW monohydric alcohols. They are generally straight chain fatty acids and contain an even number of carbon atoms. As has been mentioned, fatty acids are prepared by hydrolysis of fats and oils which are the triesters of glycerol. Still higher MW fatty acids are obtained from waxes in which they are present as esters of higher MW alcohols. Table 2.4 shows the percentage composition of fatty acids in natural fats and oils. Stearic acid (C_{17} H_{35} COOH) and palmitic acid (C_{15} H_{31} COOH) are produced mainly from tallow and other low cost animal by-products which are colourless waxy solids.

Fatty acids are commercially important products. Fatty acids have applications in many important industrial products, as shown in

Table 2.5. These chemicals are obtained primarily from naturally occuring animal fats and vegetable oils [34]. Fatty acids, because of their thermal properties, meet the thermodynamic and kinetic criteria for low cost energy storage materials for use in composite building components. They have a low melting point range with high latent heat of melting per unit mass. They have a small volume change (*expansion/contraction*) during phase transition, show optimum nucleating and crystal growth and, because of this, exhibit no super cooling during freezing cycles. Being saturated, they show excellent chemical stability and are non-toxic, non-corrosive and have low vapour pressure at room temperature. Thermal properties of a mixture of stearic acid and palmitic acid varies with the weight percentage in the binary mixture. Figures 2.6 to 2.11 show the relative dependency regarding percentage composition of each component in the mixture.

Table 2.4

Composition (%) of some Common Fats and Oils [33]

Fat or Oil	Fatty Acid				
	Myristic acid	Palmitic acid	Stearic acid	Oleic acid	Linoleic acid
OILS:					
Olive oil	-	6-10	1-4	83-84	4-7
Peanut oil	-	6-9	2-5	50-60	20-30
Groundnut oil	-	6-14	2-7	46-72	13-38
Cottonseed oil	1-2	17-29	1-4	13-44	33-58
Mustard oil	-	1-3	1-3	8-40	10-29
Coconut oil	1-2	17-29	1-4	13-44	33-58
Sunflower oil	-	2-10	1-6	7-42	55-80
Soybean oil	-	7-12	2-6	20-50	36-65
FATS:					
Beef tallow	2-6	24-32	15-25	37-43	2-3
Butter fat	7-12	23-30	8-13	30-40	4-5
Human fat	3-6	24-26	5-8	40-45	8-10
Lard	1-2	25-30	12-18	40-50	5-7
MARINE OILS:					
Whale	5-10	10-20	2-5	33-40	-
Fish	6-8	10-25	1-3	-	-

Table 2.5

Approximate % end use of Fatty acids [35]

Application field	Percentage
Fatty Alcohols	12
Soaps	9
Emulsion Polymerization	5.5
Dimer and Trimer	5.5
Lubricating Grease	5
Specialty Household Cleaners	5
Plastic Additive	4
Food Additives	4
Cosmetics	4
Downstream Acids	4
Ore Floatation	3.5
Paint and Varnish	3.5
Other Uses	35
	100

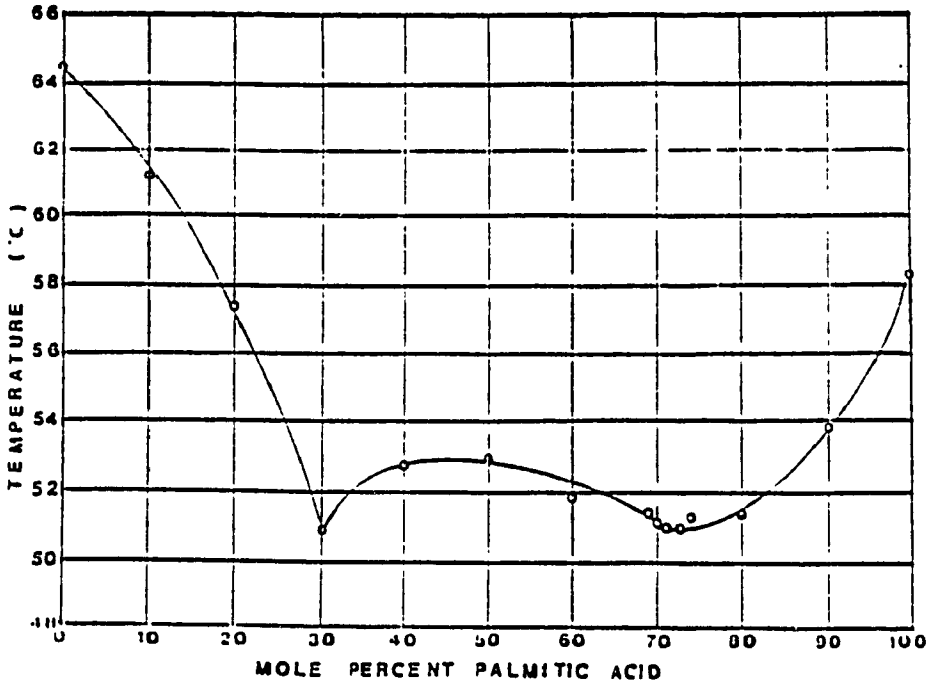


Fig.2.6 Melting points of binary system palmitic/stearic acids [35].

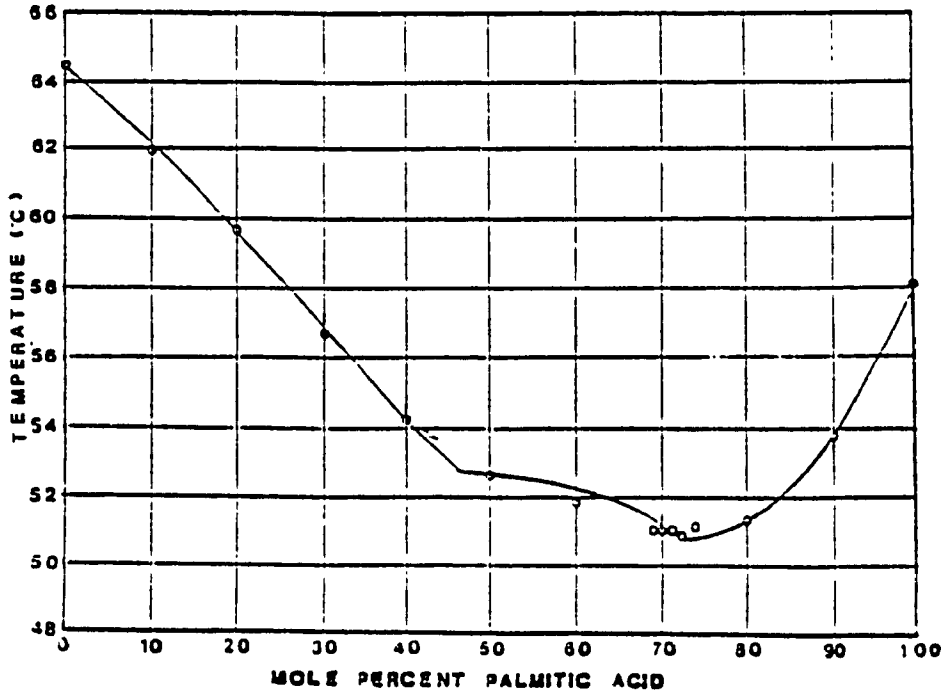


Fig.2.7 Freezing points of binary system palmitic/stearic acids [35].

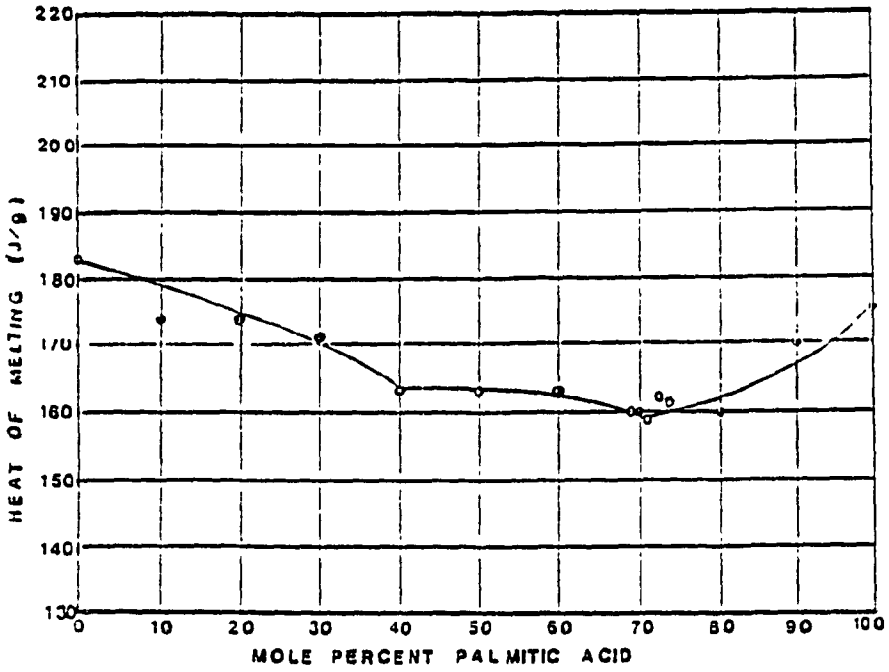


Fig.2.8 Heat of melting of binary system palmitic/stearic acids [35].

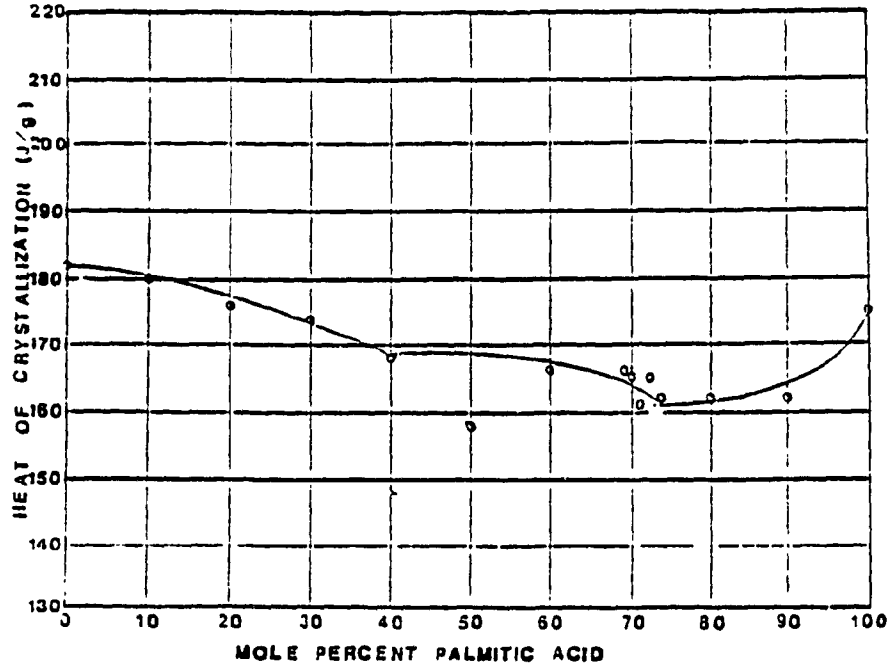


Fig.2.9 Heat of crystallization of binary system pal/stearic acids [35].

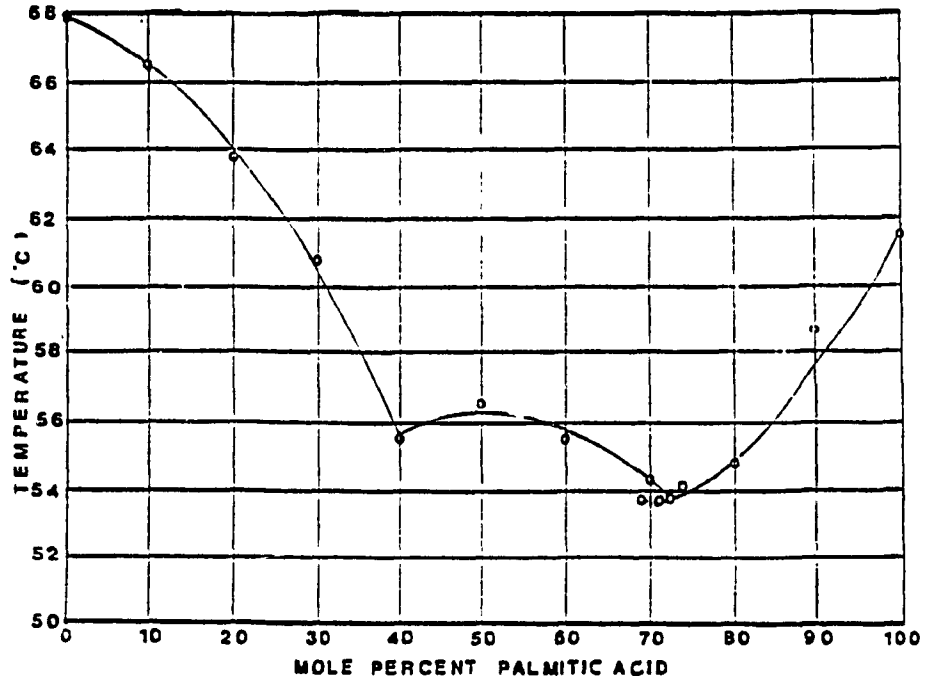


Fig.2.10 Temperature for maximum endothermic heat flow of binary system palmitic/stearic acids [35].

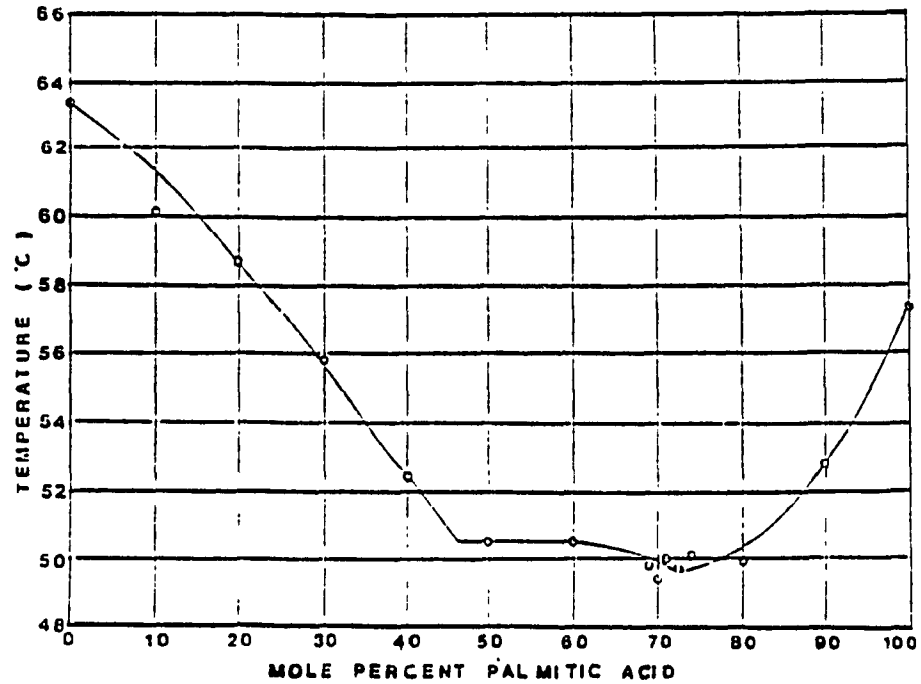


Fig.2.11 Temperature for maximum exothermic heat flow of binary system palmitic/stearic acids [35].

CHAPTER 3

SELECTION OF AN APPROPRIATE PCM FOR USE IN STORAGE WALLBOARDS

3.1 INTRODUCTION

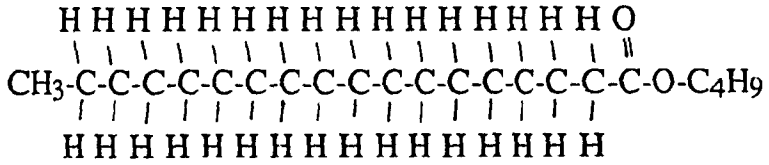
Finding the right PCM for a specific use is rather a difficult task. To get a suitable PCM, many lengthy sets of tests and verification must be done. However, one such procedure has been performed at the Centre for Building Studies (CBS) of Concordia University.

The CBS is working on the incorporation of a suitable PCM into porous structural building components to produce energy storing building elements for use in passive solar systems. The project was initiated in 1982; with special interest being placed on S-L organic PCMs. The CBS has established the suitability of 14 PCMs (Table 3.1) which melt in or near the comfort zone. From these 14 candidates, and based on a series of characteristic evaluations, fatty acid esters were chosen (Table 3.2) as suitable candidates for incorporation in

GB products. Further investigation related to the specific thermal, physical, chemical, kinetic and economic requirements narrowed the choice to two candidates which could be diffused into regular wallboard to provide a useful passive thermal storage matreialds. The first candidate is a mixture of 45 mole per cent capric acid with 55 mole percent of lauric acid; the second is a commercial product with 49 mole per cent butyl stearate and 48 mole per cent butyl palmitate [36]. In this study, in addition to the above criteria, additional requisite incorporation characteristic have also been considered.

3.2 BUTYL STEARATE

The raw material for butyl stearate is a mixture of stearic and palmitic acids. Chemically, butyl stearate is an ester of stearic acid with the following molecular formula;



In this work, butyl stearate refers to a binary mixture of 49% butyl stearate and 48% butyl palmitate, a commercial product (Emery 2325) obtained from Emery Industries Ltd. Canada.

The studies performed at the CBS have shown that BS can be successfully incorporated in many different porous building materials.

Table 3.1
Organic phase change materials studied at CBS [37].

Chemical Composition	Characteristic	Trade name
FATTY ACID ESTERS		
Butyl stearate	Saponification no.170	Uniflex Bys-CP
Isopropyl stearate	Ester 99%	Unimate IPS
Me-12 hydroxy stearate	Saponification no.178	Cenvax ME
Isopropyl Palmitate	Ester 99%	Unimate IPP
Dimethyl sebacate	Saponification no.483	Uniflex DMS
Vinyl stearate	-	-
ETHOXYLATED ALCOHOLS	grafted side chains with:	
Alcohols C_{11}-C_{15} MW 850-920; Ave.860	15 ethoxy units	Tergitol 15S15
Alcohols C_{11}-C_{15} MW 1020-1150; Ave.1080	20 ethoxy units	Tergitol 15S20
Alcohols C_{11}-C_{15} MW 1440-1640; Ave.1520	30 ethoxy units	Tergitol 15S30
ALKYL-PHENOL		
Ethoxylatednonylphenol MW 1900-2100; Ave.1980	grafted side chain with 40 ethoxy units.	Tergitol NP 40
SULPHUR COMPOUNDS		
Octadecyl thioglycolate	assay 95%	-
Dilauryl thio-dipropionate	assay 99%	Evanstab 12
Octadecyl 3-mercapto propionate	assay 95%	Evangard-18MP

MW = Molecular weight; Me = Methyl; Ave = Average

Table 3.2

Thermal characteristics of the FA esters [37]

Fatty acid esters	Heating		Cooling	
	m.p.[°C]	ΔHm [J/g]	f.p.[°C]	ΔHf[J/g]
Isopropyl palmitate	11	100	11	95
Isopropyl stearate	14	142	18	140
Butyl stearate	19	141	21	136
Dimethyl sebacate	21	135	Super cools	120
Vinyl stearate	27	122	29	122
Me-12 hydroxy stearate	43	126	42	120

mp = Melting point fp = freezing point Me = Methyl

ΔHm = Heat of melting ΔHf = Heat of freezing

The aim of the present work is to study the feasibility of incorporating BS as an organic PCM directly in the mixing stage during the production of GB.

The following sets of criteria regarding the selection of BS for storage wallboard has been adapted from a review of the report entitled "Energy Storing Wallboard" [38].

3.3 THERMAL PROPERTIES

i) Transition Temperature

The phase transition temperature of this material is such that its melting point is complete at the upper limit shown below. In this case, BS phase transition temperature is in the region of 20°C so that, charging and discharging of heat can be accomplished in the 17-23°C which is in the comfort level range. As can be seen from Fig 3.1 the fusion and crystallization temperatures are within 4°C of each other which prevents excursion outside the comfort zone.

ii) Latent heat

The BS should have the highest possible latent heat of fusion, preferably in the17-23°C range, so that it meets the thermal requirements of buildings, with the least amount of BS (since the greater the heat of fusion, the less the amount of BS is required)

iii) Heat Transfer

One of the very important factors influencing the selection of BS is its ability to transfer heat from within the matrix in which it is incorporated. With respect to the wallboard, the problem of heat transfer has been reduced by the fact that wallboard combines the functions of reservoir, conduit, heat exchanger, distributor and collector[38]. However, the conductivity and thermal diffusivity of gypsum and BS must be considered together.

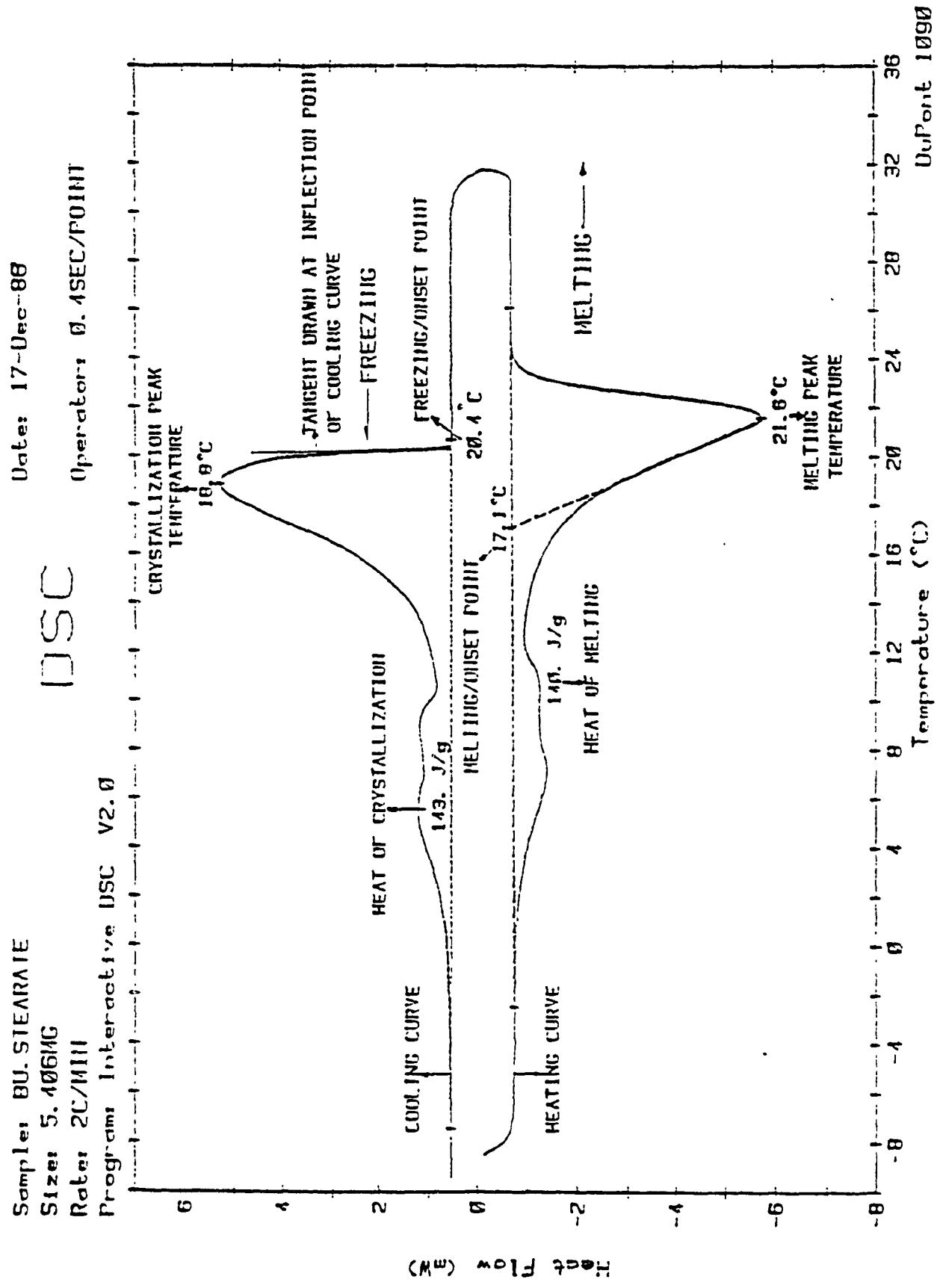


Fig.3.1 DSC Thermogram of heating/cooling cycle of butyl stearate.

3.4 PHYSICAL PROPERTIES

i) Phase Stability

As the BS in matrix changes from one phase to the other the composition of the two phases become identical under all operating conditions and equilibrium is maintained. Therefore, the melting point of the components is congruent and the constituents do not segregate during the phase change process.

ii) Vapour pressure

In order to have a low vapour pressure at room temperature the PCM must have a high boiling point, desirably over 200°C. (b.p of BS is 230°C)

iii) Volumetric changes

The expansion-contraction during L-S phase transition is a matter that must be considered such that, volumetric changes are not detrimental to the integrity of the composite. In this respect BS contracts upon freezing and leaves sufficient place within the voids present in the gypsum matrix for expansion to occur.

iv) Density

To maximize the heat storage per unit volume, it is desirable to select a PCM with the greatest possible density. This consideration is important since it affects the cost of the final product.

3.5 KINETIC PROPERTIES

i) Supercooling

Some materials remain in the liquid state even when the temperature drops below their freezing points. If this happens, the latent heat is not released at the required temperature and

consequently their prime function is negated. butyl stearate is a suitable candidate in this respect since, as an organic compound it does not show a tendency toward supercooling.

ii) Crystallization Rate

The rate of crystallization of PCM is controlled by the kinetics of incorporation of the molecules in the crystal lattice and by the rate of heat transfer. Both of these processes must proceed at a rate that ensures the exchange of heat within an acceptable period of time for effective temperature conditioning.

3.6 CHEMICAL PROPERTIES

i) Chemical Stability

To ensure that the material has a long life, chemical reactions and thermal decomposition must be limited to an acceptably low rate (see chapter 7.2, IR analysis).

ii) Compatibility

The PCM used for incorporation must be inert toward other components of the composite system and the materials with which it comes into contact. These include building materials, wiring, piping, paints, wall covering, adhesives, and others (see section 6.3 compatibility tests).

iii) Non Toxicity

The PCM and its combination there of, must be non-toxic.

iv) Fire and explosion Safety

The incorporation of PCM into a building component or material must not constitute or cause any hazardous problems; fire, fume, explosion etc (see chapter 6.2.4 fire tests)

v) Elimination of Nuisance

The PCM selected must not cause any nuisance due to allergic reaction (*vaporization*) or create unpleasant odours.

vi) Availability

The source materials for selected PCM must be sufficiently abundant and readily available.

CHAPTER 4

COMPOSITE MATERIALS

4.1 INTRODUCTION

A composite material is a material system composed of a mixture of two or more constituents (phases) differing in form or material composition and essentially insoluble in each other.

In principle, composites can be constructed of any combination of two or more materials, whether metallic, organic or inorganic. Although the possible material combinations in composites are virtually unlimited, the form of the constituents is more restricted. The major form of the constituents used in composite materials are fibers, particulates, flakes, layers, fillers and matrices.

The matrix is the constituent which forms the main body of the composites, serving to enclose the composite and give it its bulk form; the fiber, particulates, layers and fillers are the structural constituents which determine the internal structure of the composite. Generally, but not always, they are the additive phase. In both homogenous and gradient composites the constituents can be arranged in either an oriented or random fashion. Several

classification systems have been used [39], one of them is based on the form of the structural constituents. This gives five general classes of composites as follows (Fig 4.1):

i) Fiber composites composed of fibers within a matrix.

ii) Flake composites composed of flat flakes within a matrix.

iii) Particulate composites composed of particulates within a matrix.

iv) Filled (or skeletal) composites composed of a continuous skeletal matrix filled by a second phase.

v) Laminar composites composed of layers of laminar constituents.

4.2 PERFORMANCE OF COMPOSITE MATERIALS

From the nature and morphology of composites it is evident that the behavior and properties of composites are determined by:

i) The materials of which the constituents are composed.

ii) The form and structural arrangement of the constituents.

iii) The interaction between the constituents.

The first factor is the most important since it is obvious that the intrinsic properties of the materials of which the constituents are composed are of critical importance. They largely determine the general order or range of properties which the composite will display. Although the interaction of constituents results in a new set of properties, these combined properties in the composite derive from those of the individual materials. Structural and geometrical characteristics of the constituents also make important contributions

to the composite properties. The shape and size of the individual constituents, their structural arrangement and distributions and the

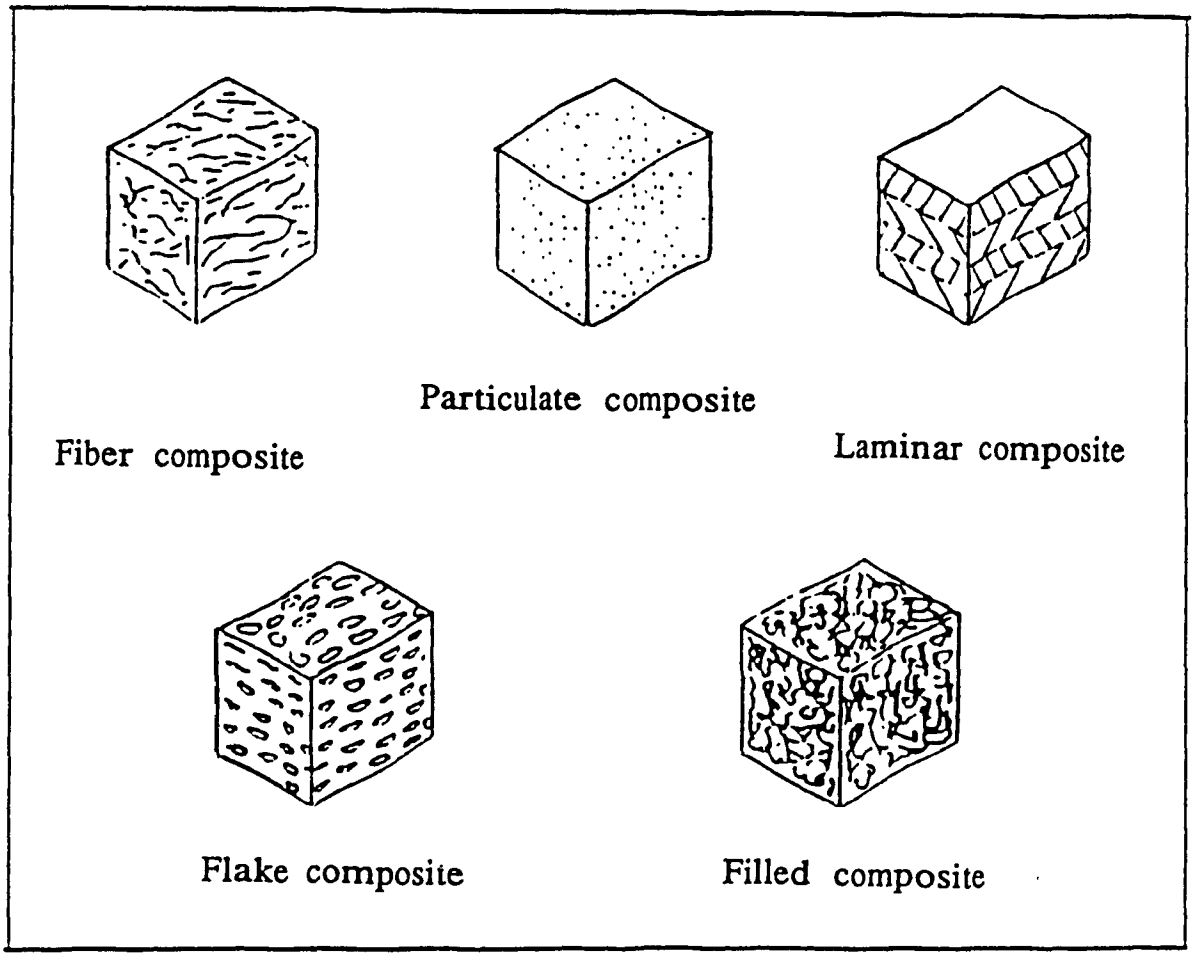


Fig. 4.1 Classes of composites [39].

Although various additives or modifiers may be used in the core, they ordinarily amount to not more than 2% of the total by weight. However, the quality of the finished board and efficiency of its manufacture are overwhelmingly dependent on the qualities of the stucco supplied to the board machine.

Total impurities in the stucco, including naturally occuring inert mineral substances, should not exceed 12% [20]. The stucco should be ground so that no more than 5% is retained on a (149 μ) standard sieve. The fineness of the stucco, in terms of Blaine surface area, should be at least 7000 cm^2/g.

BPG has often been considered as a starting material for GB stucco. However, as it was mentioned in section 2.2, special process steps, including rigorous control of residual phosphates, are required in order to make a usable stucco from BPG. The added process costs can be justified only in the situations where good mineral gypsum is not available.

4.3.1.2 *Starch*

Starch is widely distributed in the plant world and each variety produces a starch of slightly different properties, ranging from size of granules to proportions of the various fractions present [40]. Chemically, starch is a carbohydrate, more particularly, a high M W polysaccharide, having the empirical formula $(C_6H_{10}O_5)_n$, where n is large but not accurately known. Polysaccharides are polymers of sugar and their hydroxyl groups enable them to form pastes with water. Starch itself is considered to be made up of two molecular constituents; amylose, a straight chain molecule (I), and amylopectin, a branched structured molecule (II). Their proportion, and the

relative amount of each are important contributions to the overall performance of the composite.

Of far-reaching importance in the performance and use of composites are the effects produced by the combination and or interaction of the constituents.

Since composites are mixtures or combinations of constituents that differ either in material or form, the properties of their combination must always be different. The basic principle underlying the design, development and use of composites is that, the use of different constituents to obtain combinations of properties and or property values are different from those of the individual constituents by themselves

4.3 COMPONENTS OF STORAGE WALLBOARD

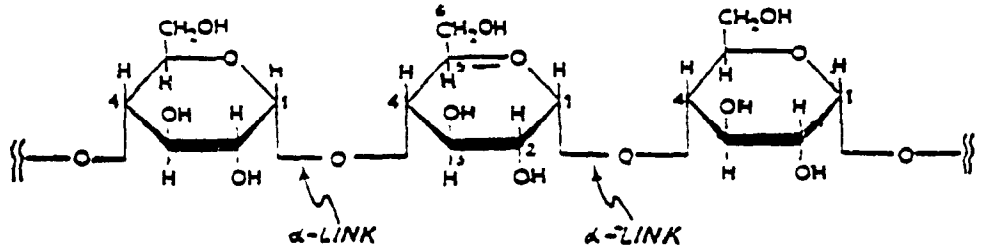
Composite energy storing board actually belongs to the particulate class of composite. It may also be put into the filled or fiber categories of composites because of the presence of pulp fibers in the board as one of its components. However, composite energy storing wallboard is composed of the following constituents which will be briefly reviewed.

4.3.1 Dry Components

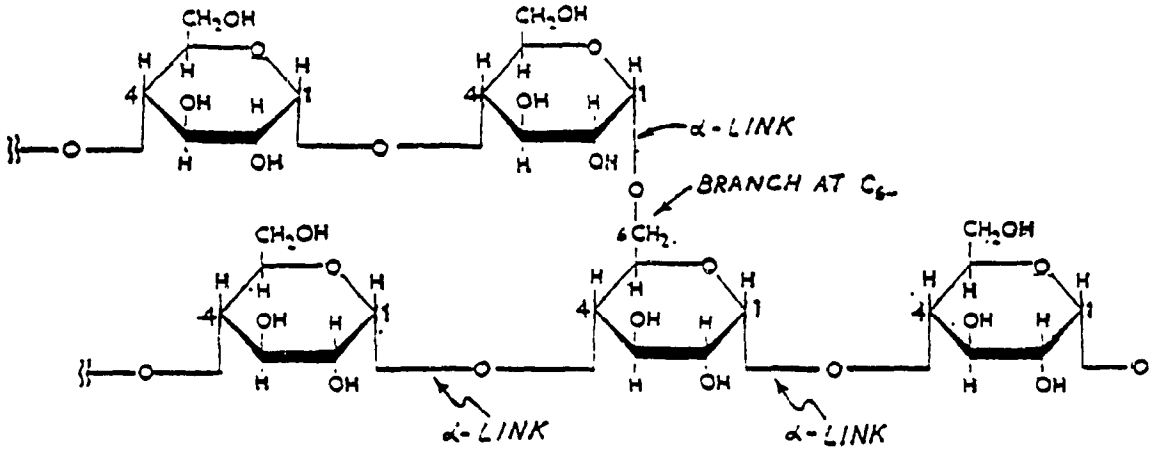
4.3.1.1 *Stucco (plaster of Paris)*

The process of manufacturing natural and BPG has previously been discussed. In GB, stucco acts as the matrix, and makes up the core of the board while other components are responsible for specific functions in the composite.

number of repeating groups per starch molecule is presumed to vary widely between starches of different origin [41].



I-Amylose [43]



II-Amylopectin [43]

Unmodified starch is insoluble in cold water and absorbs up to about 30% water without appreciable swelling. Although a sticky dough or paste can be made by simply mixing starch and cold water, such an adhesive has little adhesive value. When the temperature is raised, however, the granules absorb more water and finally burst, forming a gelatinous colloidal gel which displays strong adhesion to many substrates, especially to cellulosic materials. It is hygroscopic in nature and has a moisture content between 10 and 20%.

The use of starch in adhesives or semi-adhesive applications is an age old practice; it is used on an enormous scale in almost all countries and has ability to give strong adhesion in low concentrations in water.

However, in this study, modified starch derived from maize or from other grains were added in amount of 0.7% (wt) to gypsum slurry for board making to reinforce the bond between the coverpaper and the core of the board.

4.3.1.3 *Lignogel (lignosulfonate)*

Lignogel is a complex derivative of lignin obtained from sulfite waste liquor a by-product of the pulp and paper industry. It is marketed as a stabilizer for asphalt emulsions, a modifier for latices, a binder for printing ink, and as a dispersing agent [49]. As a dispersing agent it accelerates the wetting process of particles to be dispersed and is used in amounts up to 0.1% (wt) to increase the fluidity of the gypsum slurry, i.e., to reduce the amount of mixing water required.

4.3.1.4 *Waste newspaper*

Waste paper fiber can be utilized not only for reuse in printing papers, but also in such necessities as toilet tissue, paper towels, boxboard, chip board and an infinite number of other paper products [42]. Waste newspaper, after mechanical disintegration, can be used as filler for boards and building papers. In wallboard manufacturing, waste newspaper is employed in amounts ranging from 0.5 - 1% (wt).

Paper fiber contributes to the strength of the board and reduces core brittleness.

4.3.1.5 *Coverpaper*

The coverpaper for wallboard production is a multiply papercover sheet which is comprised of cellulosic and mineral fibers. The multiply sheets are made by separately dispersing a mass of cellulosic fibers (includes ligno-cellulosic fibers) and mineral fibers (includes rock, wool, slagwool, spun or drawn glass fibers) in water, combining the two dispersions of fibers in desired proportions and finally forming the combined fiber into a web of paper on a papermaking cylinder machine [44].

The special paperboard used for the coversheets is made at a caliper of about 0.51 mm and a weight of about 340 g/m^2. The paper must be sized so that it is readily wettable by the stucco slurry, yet resist through saturation which would cause surface staining. If the sheet is too tight "*paper blows*" will occur in the kiln (oven).

The coverpaper will sandwich the gypsum core and act as a mold (container). It doubles the flexural strength of the board especially when the applied load is perpendicular to the fiber direction of the paper board.

4.3.2 Wet Components

4.3.2.1 *Water*

Gypsum has the unique property of giving up some of its chemically combined water and powdering when intensely heated (*calcination*). Then it is restored chemically to the original rock like form when water is added by forming interlocking crystals

(*crystallization*). The mixture of calcined gypsum powder and water remain plastic for a short time and can be easily shaped or molded. Exploiting these properties of gypsum is possible only by using the proper amount of water for hydration reaction and formability of the paste. However, even when fluidizing agents are used, board stucco, as compared to other cementitious materials, requires a relatively large amount of water to make a formable slurry. A typical ratio is 75 parts water per 100 parts stucco (i.e. w/g ratio is 0.75). Of these 75 parts water, about 18 parts (*combined water*) are taken up as the stucco ($CaSO_4.1/2H_2O$) hydrates to gypsum ($CaSO_4.2H_2O$), leaving 57 parts (*free water*) to be evaporated [20].

4.3.2.2 *Foaming agent solution*

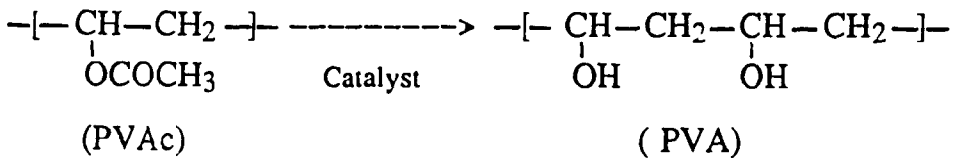
The void volume left by evaporation of water reduces the apparent core density but still lighter weights are desired for ease of application and economy in shipping and handling.

Weight reduction may be accomplished by addition of light aggregates such as expanded perlite, vermiculite or polystyrene, but more often by the incorporation of a separately generated aqueous fluid foam (see chapter 2.4). The most widely used foaming agent is a special potassium rosin soap. Alkali metal salts of alkylarene-sulfonic acids are also frequently employed. Usage ranges from 2.5 to 4.8 g/m^2 of GB on the basis of 1.27 cm thickness. In this study a foaming agent which was provided by Westroc Industries (*Westroc Foaming Agent* (WFA)) has been used.

4.3.2.3 Dispersing and emulisifing agent

i) *Poly Vinyl Alcohol (PVA)*

PVA is a completely synthetic, water soluble polymer of vinylalcohol, often containing a proportion of acetate groups. PVA is a high polymer that has no isolatable monomer. The formation of the monomer is a transient stage in the preparation of the polymer from poly (vinyl acetate)(PVAc) [41] as shown below;



The extent to which the acetate groups are replaced by hydroxyl (-OH) groups is described as the percentage of hydrolysis and the properties of PVA depend very much on this factor. Accordingly, the solubility may vary from almost complete insolubility in water and solubility in certain organic solvents to the opposite extreme. For example, with 70% acetate groups PVA is insoluble in water and with less than 3% it is soluble in hot and swellable in cold water.

PVA is a safe and nonhazardous material. It offers a combination of excellent film-forming and binder characteristics. As an adhesive, PVA is used in paperboard, industrial product for bonding paper, textiles, wood, and for general purpose household adhesives. As an stabilizer, it is used in emulsion and suspension polymerization. In this study the PVA solution is used for its emulsion stabilizing and film forming properties.

ii) *Lignin*

Lignin is a byproduct of the pulp and paper industry. It has been shown that lignin, which comprises 17-33% of wood, is a complex aromatic polymer and has a role in cementing the polysaccharide components in cell walls both chemically and physically, thus increasing the mechanical strength of wood as a composite material and its decay resistance toward microorganism [47].

Commercially, lignin and other components are removed in the production of woodpulp used in vast quantities for the manufacture of paper. The spent liquors contain degraded lignin salts [40].

Although in natural wood the lignin may well have essentially infinite molecular weight, isolated lignins or lignins recovered from commercial pulping operations, such as kraft lignins or lignin sulfonates, are broadly polydisperse materials with molecular weight ranges from C_9 monomers and dimers to oligomers and polymers of undetermined degree of polymerization [46].

The structure of lignin is complex and is still a matter of models. Despite the effort that has been expended in research on lignin the structure remains unknown. However, the suggested structure for a soft wood or coniferlignin in its native state is as shown in Fig. 4.2 [48].

The great mass of extracted lignin continues to pile up. Paper industries are faced with disposal problems of a magnitude not faced by other industries. What should be done with the solutions of lignin? Despite the attention that has been given to the problem, it

remains yet to be resolved. Instead of being a source of useful aromatic compounds it continuous to be a disposal problem.

It has, however, the potential of becoming a major source of polymer-based products for different industries, including the construction industry, for such items as building board adhesives, binders, coatings, foam insulation, etc. [48]. Most lignin product applications are based on technical lignin (*lignosulfonate & Kraft lignin*). Lignin macromolecules in solution systems act as emulsifying agents, stabilizers and dispersants. Based on its surface tension properties lignin, has been used to incorporate the butyl stearate into the gypsum slurry during wallboard production.

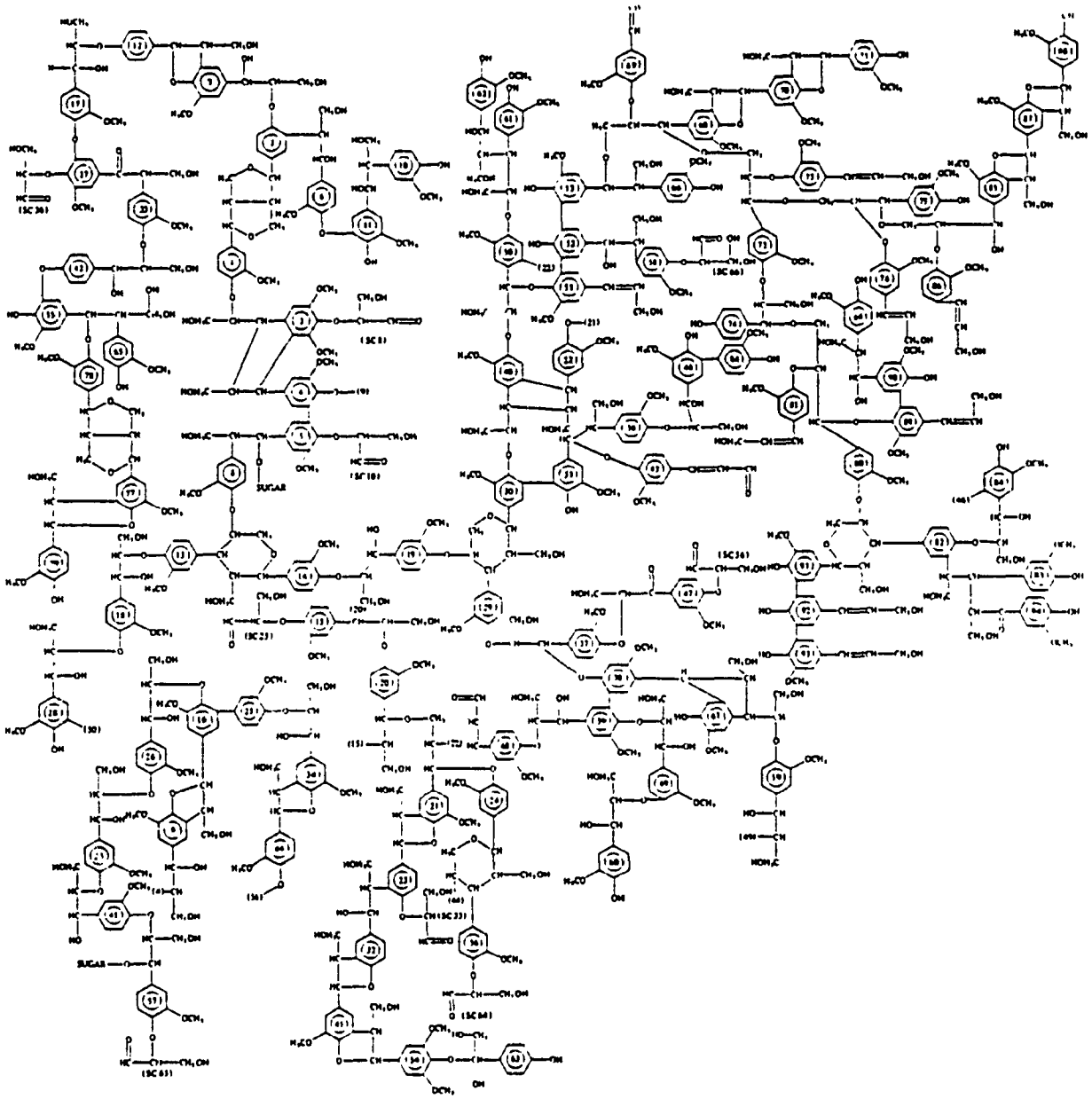


Fig.4.2 Softwood lignin model designed by computerized evaluation [48].

4.4 COMPONENT PREPARATION

4.4.1 Dry Mix

A dry mix was prepared by weighing specific amounts of stucco, starch and lignosol (wetting agent). A specific amount of accelerator is prepared in order to permit sufficient time to thoroughly mix and distribute the blend manually, prior to the initiation of setting process.

After weighing, all dry components were introduced into a round bottom container and were initially mixed manually for about two minutes and then mechanically rotated by motorized mixer (Canlab model RZR-50) to obtain a well homogenized dry mix suitable for slurry preparation.

4.4.2 Pulp Preparation

Paper fibers were introduced into the core of the board, to reduce its brittleness and to increase its bending strength. A required amount of paper fibers in the form of waste newspaper was weighed in combination with requisite amounts of starch, water and potassium sulfate. The components were blended using a Waring commercial blender (model 33BL79) for 30 minutes to obtain a well homogenized pulp paste which is suitable for use in the slurry preparation.

4.4.3 Foam preparation

To provide sufficient voids within the matrix and to reduce the density of the GB to permit the incorporation of PCMs, a suitable foam was prepared using WFA. One gram in 100 cc solution of

foaming agent was prepared by weighing 10 grams of WFA and diluting it to 1000 cc using distilled water, of which 140 cc was introduced into the blender (7011 model 33BL73). Foaming proceeded at high speed for 1.5 minutes after which the foam was ready for use in the slurry. A discription of the methods used to optimize the foam density is given in chapter five (see section 4).

4.4.4 Emusifiers

i) *WFA Solution*

solutions of 0.85, 1, 1.25 and 1.5%(wt) WFA were prepared in distilled water and used as emulsifiers to facilitate the dispersion of the PCM into the slurry. The 1.5%(wt). solution gave optimum results.

ii) *PVA Solution*

Solutions of 1, 1.5, 2.5 & 5%(wt) concentration of PVA were prepared in distilled water at 50°C to be used as an emulsifier. The optimum in this case was 2.5% (wt). solution with respect to the ease of emulsification and slurry workability.

iii) *Lignin Solution*

Dispersion solutions of 0.5, 1, 1.5 and 2%(wt) concentration of sodium salt of kraft lignin (SSKL) were prepared for use as an emulsifier. Based on numerous trials, the 1.5% (wt). solution of SSKL gave the optimum emulsification and workability results.

CHAPTER 5

METHODS OF PCM INCORPORATION

5.1 INTRODUCTION

The incorporation of PCMs into building materials has been a matter of considerable research in recent years [25,31,32,36]. They may be incorporated, usually, in one of the following ways:

- Addition of small self contained capsules of PCM when blending the components of the building material.
- Immersion of the building materials (e.g. bricks, tiles, boards, blocks, etc.) into a bath of liquid PCM.
- Direct incorporation of PCM with other components which make up the building materials, during the manufacturing process.

As has already been mentioned, work on developing an immersion type product is complete and the present study is concerned principally with the direct incorporation technique. As a starting point, test specimens were made using the immersion technique such that a comparison could be made with specimens made using the direct incorporation technique.

5.2 IMMERSION TECHNIQUE

This technique involves immersing prefabricated and prefinished wallboard samples of nominal size, 20.32 x 20.32 x 1.27 cm, in a bath of liquid PCM with proper control of immersion time and bath temperature. For these experiments, specimens were prepared from GB and immersed in liquid PCM having a temperature of 78-81°C. Immersion time and temperature varied as a function of percentage absorption and density required for each specimen series. The relationship between PCM absorption in relation to both immersion time and PCM temperature is given in Figs. 5.1 and 5.2 and Tables 5.1 and 5.2.

It can be seen from this data that the rate of absorption of PCM within a specified time interval (55±5s) increases with an increase in PCM temperature. This process continues up to the saturation point of the GB, i.e. the point where the voids present in the gypsum matrix are completely filled. This point was determined by successive weighing of the GB until no weight increase was observed. In the case of Butyl Stearate optimum conditions were achieved at a 26-28% weight gain (using an immersion time of 55±5 seconds and a PCM temperature of 78-81°C.

Beyond a 28%(wt) gain , the PCM is exuded from matrix. Further increase in the density of the wallboard give rise to problems in respect to transportation, installation and cost.

It should be pointed out that immersion time is also affected by other factors such as the temperature, type and thickness of the GB being impregnated. In this particular instance, however, the

immersion operation should take place at a temperature of 78 - 81°C and within an immersion time of 55 ± 5 seconds.

Table 5.1
Percentage weight gain of PCM in GB specimens as a function of immersion temperature.

No.	Initial Wt.(g)	Final Wt (g)	Time (s)	Temp. (°C)	% Wt. gain	Gain in Wt. (g)
1	342.40	418.07	55 ± 5	25	22.10	75.67
2	336.00	414.50	"	35	23.36	78.50
3	328.60	409.67	"	45	24.67	81.07
4	331.80	418.07	"	65	26.00	86.27
5	334.50	425.95	"	75	27.34	91.45
6	337.00	433.62	"	85	28.67	96.62
7	340.00	442.00	"	95	30.00	102.00

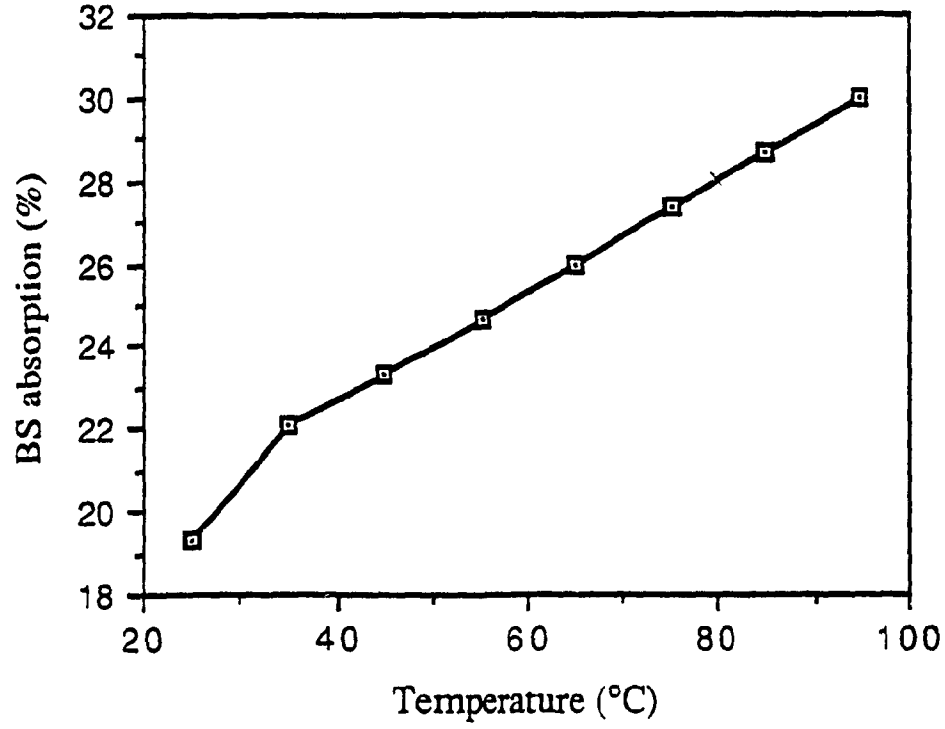


Figure 5.1 Temperature dependency of PCM absorption.

Table 5.2
Percentage weight gain of PCM in GB specimens as a function of immersion time.

No.	Initial Wt.(g)	Final (g)	Temp. (°C)	Time (s)	% Wt gain	Gain in Wt. (g)
1	338.00	384.27	82 ± 0.5	25	13.69	46.27
2	335.50	404.95	"	50	20.70	69.45
3	340.00	423.98		75	24.70	83.98
4	337.70	432.36		100	28.03	94.66
5	336.40	440.78		125	31.03	104.38
6	339.10	453.34		150	33.69	114.24

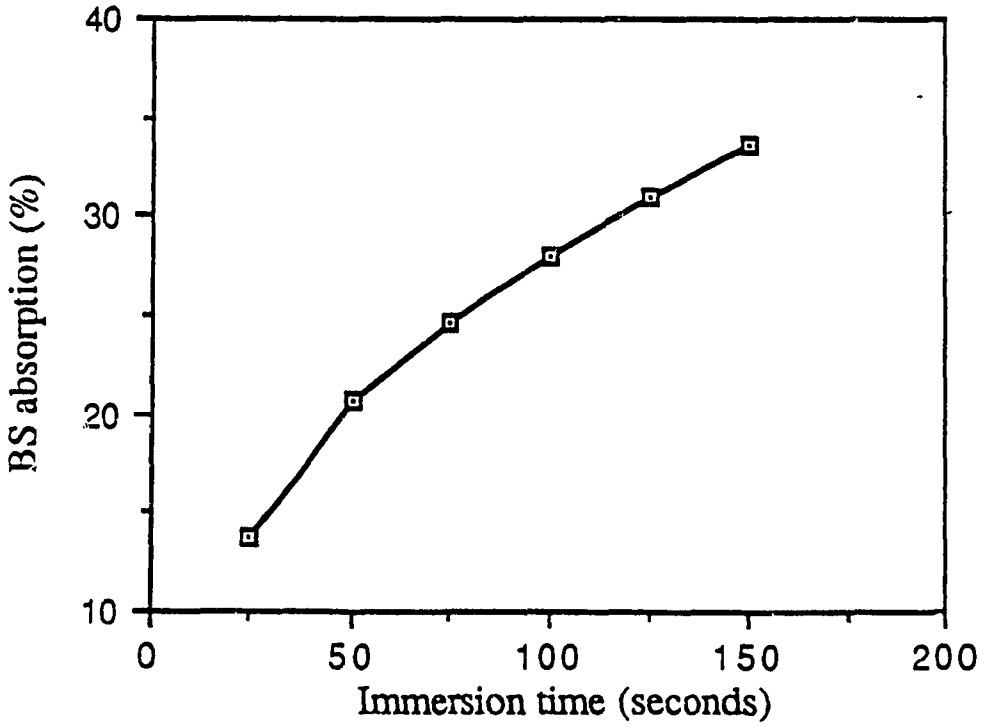


Figure 5.2 Immersion time dependency of PCM absorption.

5.3 DIRECT INCORPORATION METHOD

The direct incorporation technique depends on the oleous PCM to have sufficient surface tension to retain it within the voids in the GB matrix without being expelled when it is incorporated into the gypsum paste at temperature above the melting point.

As mentioned in section 2.5, GB is produced by the thorough mixing of calcined gypsum, water and additives into an aqueous slurry which is then sandwiched between coverpapers in a continuous process. The resulting product is then passed on to a moving conveyor belt where the setting process occurs. The board is then cut to the desired length, after which it is passed through a dryer where the excess water is removed. It is supposed that if the PCM can be introduced directly during the process of slurry preparation, comparatively little change in the existing production facilities will occur. To examine the practical aspects of this proposed incorporation method a number of experimental procedures were developed. These were thoroughly investigated in laboratory scale tests which are described in detailed below.

5.4 DEVELOPMENT OF LABORATORY SCALE PROCEDURES

Since it is evidently not possible to perform full scale development testing using automated and computerised production facilities, such as those found in GB production companies, a serious attempt has been made to produce similar results using facilities developed at the CBS which uses manual operation. Full attention is given to the

condition of the ingredients, the precision of mix quantities as well as the timing and the mixing order of ingredients into the blend.

To start with, based on random mixing of stucco and water, many apparently workable plain stucco pastes were prepared and formed. The Water/Gypsum (w/g) ratios were evaluated and compared with those found from literature . The final procedure has been developed through the following stages described below.

5.4.1 Stage 1: Plain wallboard

Plain GB of 7.62 x 10.16 x 1.27 cm (3x4x1/2 inches) was produced in the following manner:

The gypsum powder (*stucco*), accelerator and starch were weighed, hand mixed and placed in a plastic container. A specified volume of water was added to the solid ingredients and manually mixed for 10 seconds, then mixed for a further 20 seconds at 800 rpm using a Heidolf RZR-50 Laboratory mixer. It was found that the use of a 5 cm helical impeller was best suited for obtaining thoroughly homogeneous blends. Foam (equal to 5% of the weight of the paste) was then introduced into the mix and the entire slurry mixed again for another 20 seconds at 400 rpm. Fig 5.3 shows the block diagram of the mixing process. The foam, produced at the same time as the slurry, was prepared by mixing 120 ml of a 1%(wt) solution of Westroc Foaming Agent (WFA) in a Waring model 33BL73 blender for 2 minutes at high speed (the density of this foam was 0.15 g/cm^3). The required quantity of foam was poured into the paste 65-70 seconds after blending.

Upon completion of mixing the resulting mixture was immediately poured into a 7.62 x 10.16 x1.27 cm mold, the excess paste was removed from the top face and the specimen leveled using a special blade. After15 minutes the specimen was unmolded, weighed, and placed in a forced air oven at a temperature of 105°C for 2 hours (to remove the excess of free water). After drying, it was weighed to determine the amount of evaporated free water

Specimens produced in such a manner (without coverpaper) were found to have an average density of 0.56 g/cm^3 which was in agreement with commercially available GB products.

5.4.2 Stage 2: Introduction of Cover Paper

Coverpaper for both the back and face of the GB was cut to the appropriate size to fit the mold. The backing paper was moistened, to decrease its rigidity and also to activate the cellulose molecules present in the paper. It was then positioned in the mold, the slurry poured onto it and both were vibrated so that the slurry filled every corner of the mold. As before, the excess slurry was removed so that the face of the mold was perfectly level. The face paper was then soaked and carefully positioned on the face, after which it was gently pressed onto the slurry using a rubber roller. After unmolding and drying for two hours, their densities were found to be in the range of 0.59 - 0.60 g/cm^3. These values had to be increased by 0.03 - 0.05 g/cm^3 to meet the requirements of standard specifications for plain GB. Another problem which arose at this stage was the weakness of the bond between the coverpaper and the core. This will be discussed later in section 5.4.4.

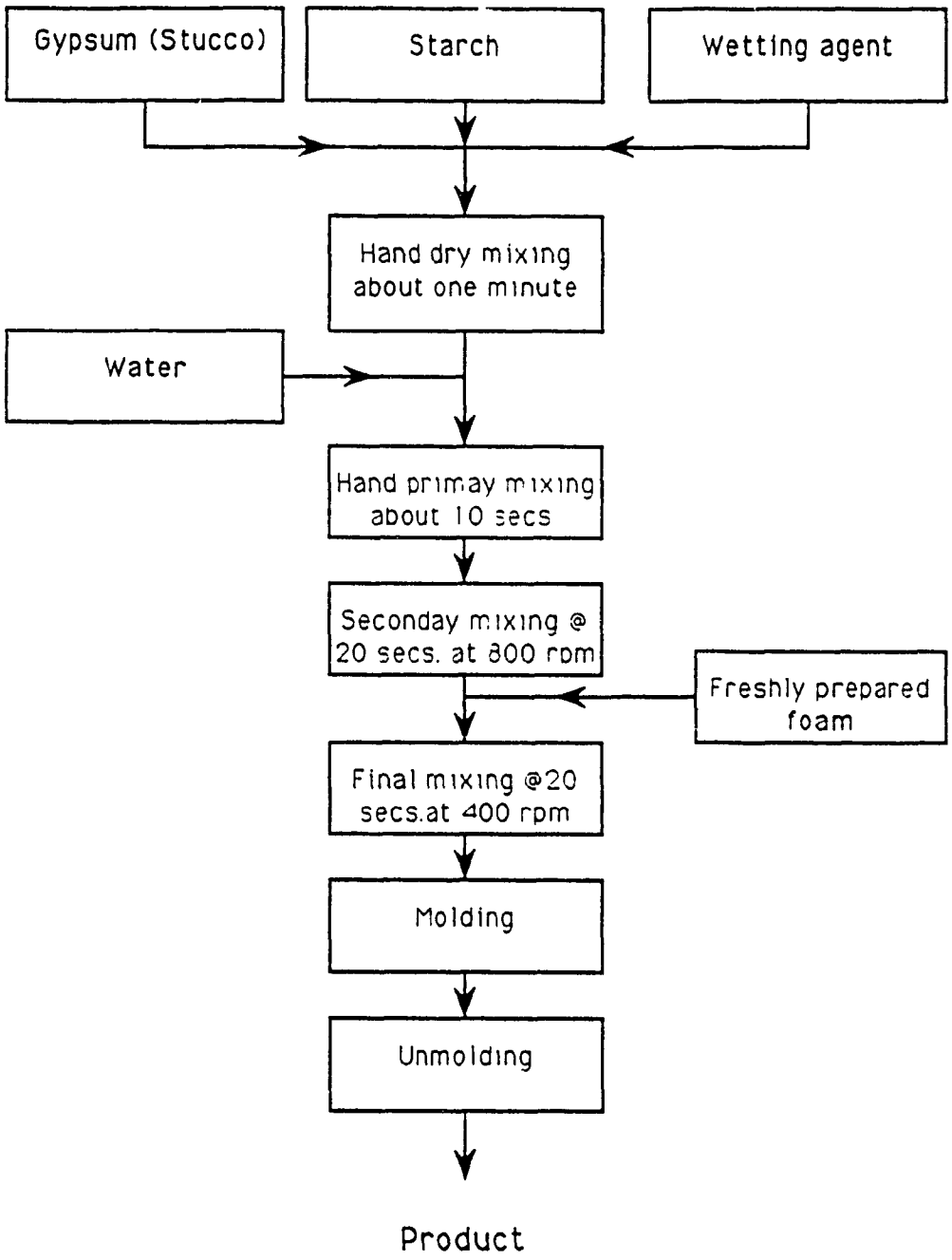


Fig.5.3 Block diagram of the mixing process of plain board production.

5.4.3 Stage 3: Incorporation of PCM

Having established a suitable laboratory procedure for producing standard GB, the next step was to incorporate the fatty ester mixture into the gypsum paste. The results of the tests to illustrate the most practical laboratory production process based on the order of mixing the blend components are given below:

No	Order of mixing	Result
1	Premixed dry components + Simultaneous water and PCM + Foam	Considerable phase separation
2	Premixed dry components + Emulsion of water and PCM + Foam	Satisfactory
3	Premixed dry components + Emulsion of water/PCM/dispersants solution + Foam	Very good
4	Premixed dry components + Water + simultaneous dispersants solution and PCM + Foam	Satisfactory
5	Premixed dry components +Simultaneous dispersant solution, Water, PCM + Foam	Poor separation of phase & heavy product
6	Premixed dry components + (Water/ dispersant solution) mix + PCM + Foam	Little separation
7	Premixed dry components + (Water/ dispersant solution) mix + dropwise stream of PCM + Foam	Very good

Mixing methods No.3 and No.7 were found to be the best procedures for a laboratory scale production process. The manual mixing procedure for both No.3 and No.7 methods is shown in Fig. 5.4 and 5.5 respectively. However, procedure No.7 is more direct and hence more practical than No.3. since it does not require the preparation of the emulsion in separate mixer. This procedure is described in detail below. A quantity of BS (22-23 wt % of stucco) was (dropwise) introduced into a pre-homogenized mixture of dry ingredients, DA, and water and mixed for 25 s at 1300 rpm. A specified amount of preformed foam was then added to the mix and further mixed for another 20 s at low speed (300 rpm). It was then poured immediately into the mold. It was observed that when the foam was added to the mix the fatty esters showed a tendency to migrate from the paste and render the foam unstable (foams are generally unstable in the presence of oleous materials). To deal with this problem 15% of the water required for the mix was replaced by a 1 - 2.5% (wt) solution of suitable DA (optimisation of paste workability with respect to the use of DA is described in the following section). Both mix orders were chosen for the preparation of larger specimens having 20.32 x 20.32 x 1.27 cm (8" x 8" x 1/2") dimensions. Samples were to be compared with factory made plain GBs and immersion type specimens.

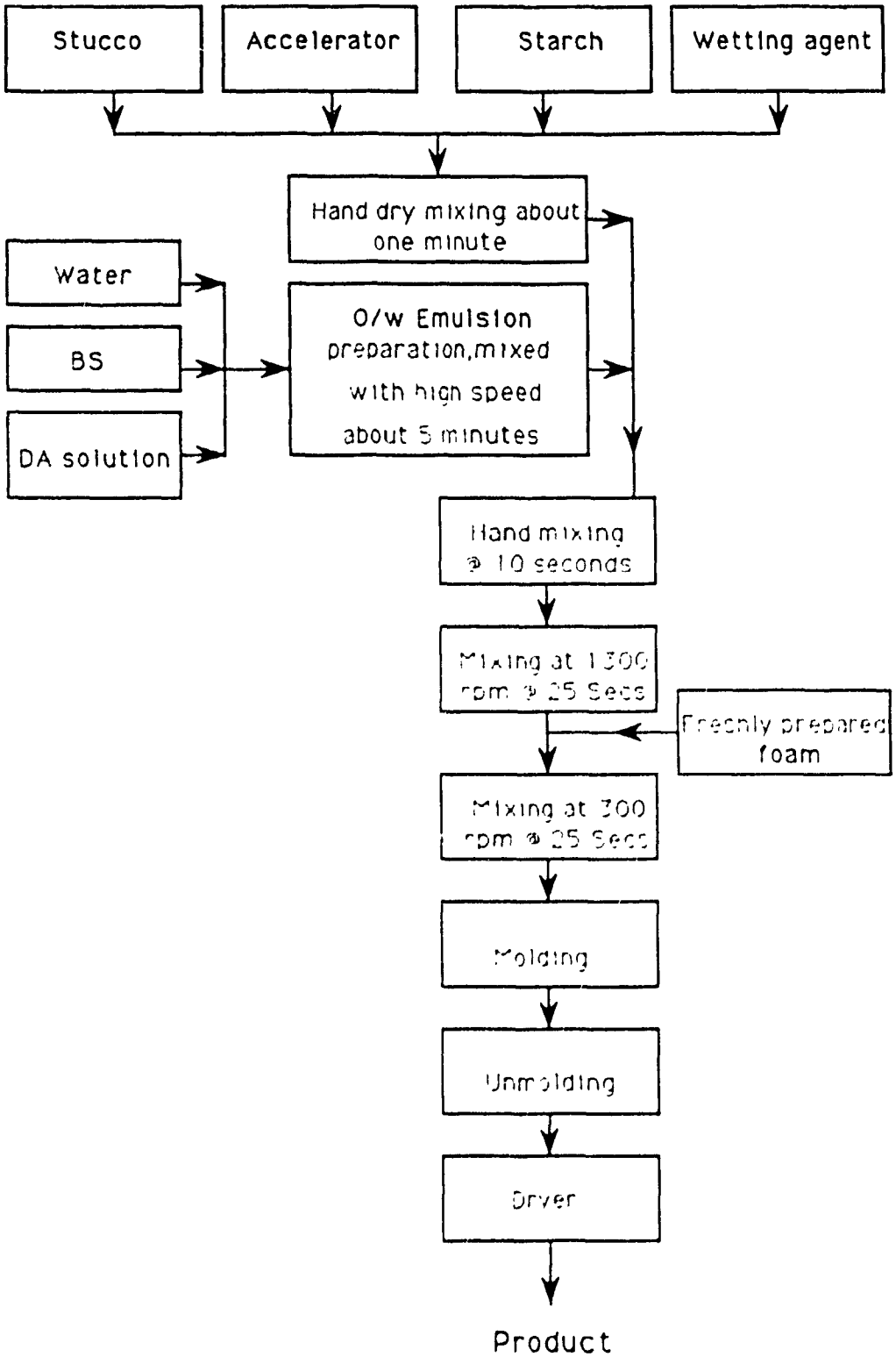


Fig.5.4 Block diagram of the mixing process of thermal storage board production (mix order No.3).

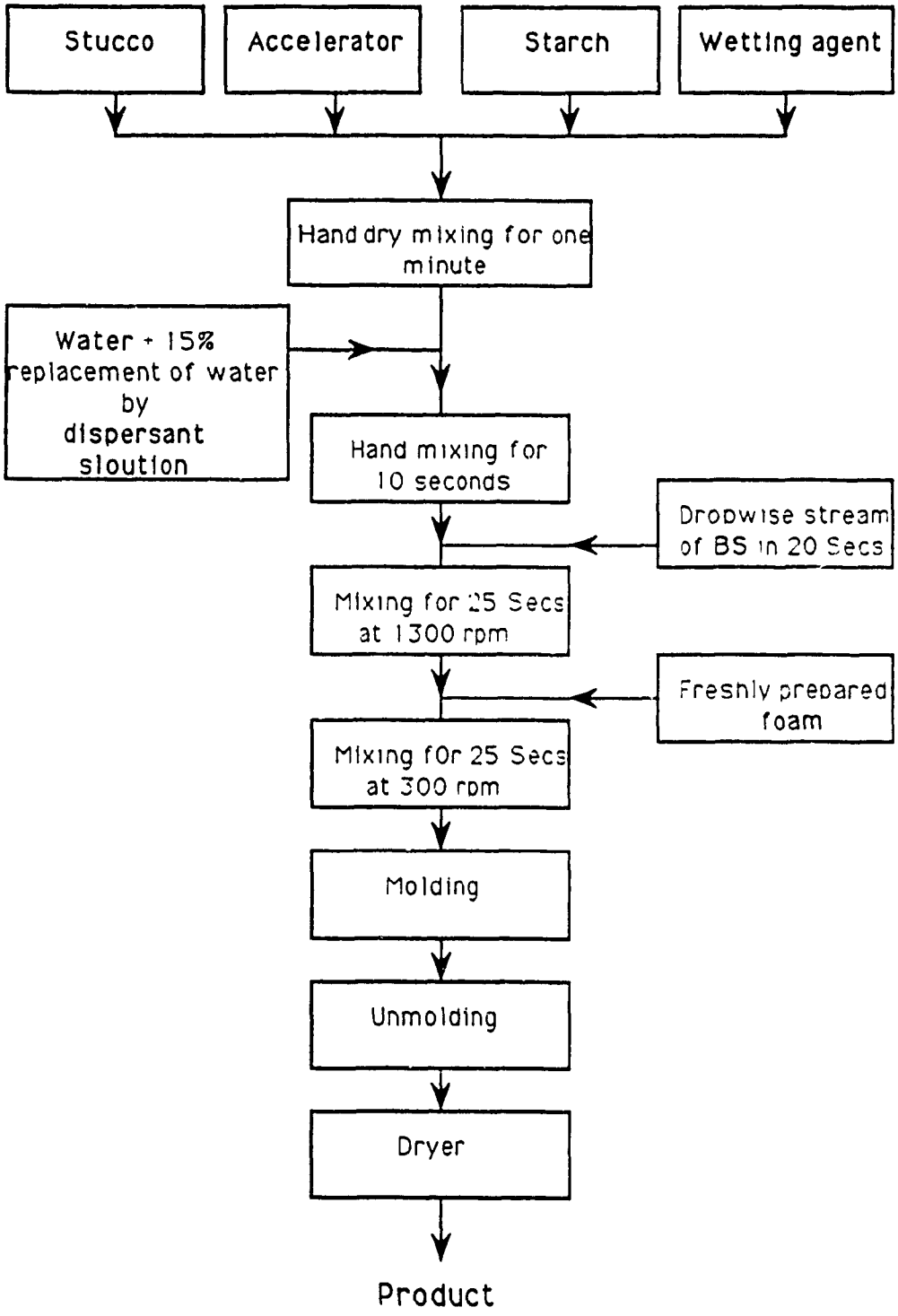


Fig.5.5 Block diagram of the mixing process of thermal storage board production (mix order No.7).

5.4.4 Stage 4: Improvement of paper/Core bond and foam density optimization

Specimens of 20.32 x 20.32 x 1.27 cm were prepared according to the above procedure, with WFA as DA. Due to the increased size of the specimens a number of processing difficulties arose. In the first instance it was no longer possible to level a specimen easily, because the consistency of the paste and its tendency to remain on the leveling blade prevented a smooth and even application of the mix into the mould. Paste consistency is a function of foam density and consequently a method was required whereby the effects of this parameter could be evaluated in order to optimize the mix consistency. This is discussed in section 5.4.4.2 below.

In the second instance, it was observed that the coverpaper became detached from the gypsum core during the drying process. The reasons for this occuring and the method used to improve the bond are dealt with in the following section.

5.4.4.1 Coverpaper bond improvement

The following reasons are given as possible causes of bond weakness between the core and coverpaper:

- Rigidity of the coverpaper in relation to the size of the specimen;
- Moisture content (over absorption) of the coverpaper;
- Influence of the PCM on the starch macro molecule in the paste and coverpaper (i.e. the effectiveness of the starch molecules to produce a bond between the paste and paper is reduced in the presence of the PCMs)
- Quantity of starch in the paste

The coverpaper would not stay flat because it retained the curved shape of the role from which it had been cut. To flatten the coverpaper and to improve the bonding process by activating the cellulose macro molecules of the paper itself, the coverpaper was moistened for one minute in a waterbath and then wiped by using paper towels ,after which it was positioned on the face of the sample. This process initially seemed to be suitable, however, when samples were removed from the air circulating oven, the problem of detachment persisted. To find the cause of this behavior a series of tests was conducted to determine the rate of moisture absorptance of the coverpaper. Table 5.3 shows the results of the tests to determine the increase in coverpaper size as a fuction of soaking time. Since the method by which the coverpaper is manufactured produces an alignment of the fibers, the dimensional changes due to water uptake are different in either direction. Little or no dimension changes occur upon miosture absorption in the direction parallel to the paper's fiber, whereas a much more significant change can be observed in the direction perpendicular to the fiber.

The results show there is an increase of about 2.1 mm for a 1 minute residence in water for the edge perpendicular to the direction of the fiber. When this coverpaper is placed in the oven for 2 hours at 105°C, there is a return to the original size but the coverpaper is in a deformed shape. However, it was found that using a coverpaper which has been pre-soaked was useful for positioning the coverpaper easily on the samples. Nontheless, these samples continued to have the coverpaper peel off in the drying stage. In order to take

Table 5.3
Result of coverpaper expansion, perpendicular & parallel to the direction of paper's fiber due to the water absorption.

Exp No.	Soakening Time (min)	Length (cm)		Increase in Length (mm)	
		Before	After	perpendicular	parallel
1	15	19.65	24.2	4.55	0.00
2	10	19.8	24.0	4.2	0.00
3	5	19.78	23.8	4.02	0.00
4	2	19.8	22.5	3.30	0.00
5	1	19.85	22.0	2.15	0.00
6	0.5	19.75	21.5	1.75	0.00
7	0.0	19.8	19.8	0.00	0.00

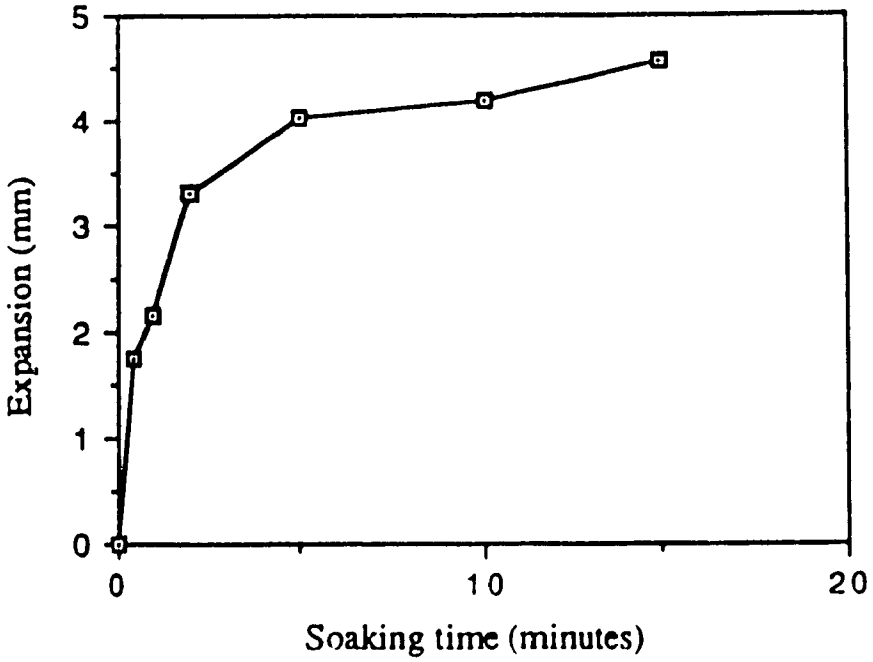


Fig 5.6 Relation between the expansion and soaking time of the coverpaper.

advantage of the pre-soaking process and to reduce the incidence of coverpaper peel-off, a 5% solution of starch was prepared and used for soaking purposes. Using this process it was found that the

pattern of expansion in the starch solution was the same as that in water and the degree of peeling off was not significantly reduced. Consequently, this process was abandoned.

The next alternative was to increase the quantity of starch in the mix (Table 5.4) and use a dry coverpaper. The dry paper was placed on the sample and kept in position by placing a perfectly flat sheet of plexiglass on it over which was positioned a 2 kg. weight. The specimen was left to stand for 15 minutes.

Another reason for the formation of a weak bond between the core and paper seemed to be due to the existence of the organic PCM (BS). It is supposed that BS, being a water repellent material, could position itself between starch molecules in the slurry and the coverpaper and, in so doing, block the bonding mechanism between core and paper, (i.e. BS reduces the number of attachment "spots" which in turn reduces the overall adhesion properties of the starch).

It is also felt that the paper/core bond problem is partly a laboratory scale problem because of the tendency of the coverpaper to deform in relatively small specimens. Furthermore, the lack of proper pouring facilities, metering and production timing all contribute, to a lesser extent, to the bond problem.

5.4.4.2 Foam density optimization

The foam density was optimized in order to:

- Obtain a better leveling action;
- Control the density of the product;
- Obtain a more persistent foam;

- Obtain the same size bubbles;
- Have a better distribution of voids in the matrix
- Determine the relationship between various variables including time, density, concentration of FA solution, speed of mixing and foam stability.

Table 5.4
Quantity of extra starch to be added to the mix, to strenghten the bond between coverpaper and the core.

Test No.	Proposed amount of starch %	Amount for each sample (g)	Condition of bond between coverpaper & core after drying
1	0.1	0.43	No bond (peeloff)
4	0.2	0.85	Partly no bond
7	0.3	1.36	1/3 no bond
8	0.4	1.70	There are small regions with no bond
9	0.5	2.13	Weak bond
11	0.6	2.55	Good
13	0.7	2.98	Very good (economical)

In order to obtain a foam having an optimum density the following series of tests was conducted.

Solutions of 0.85, 1, 1.25 and 1.5% (wt) concentration of the FA (*Westroc*) were prepared and used for foam preparation. The foams with the abovementioned concentrations were used for foam density determination, foam stability and for the optimization of the FA concentration.

The densities were measured at 15 ± 5 seconds and 65 ± 5 seconds after preparation to evaluate the reduction in foam density

after 60-70 s of elapsed time. Although there was no apparent significant difference between values obtained for both times, but physically, it was found that the foam life after 60-70 s was dramatically lower and hence was not suitable for mechanical stirring. Use of this foam resulted in higher board densities because the bubbles break much more easily in comparision to those present in fresh foam. It should be noted that the slight increase in the board density is due to the fact that results obtained when performing these tests manually are slightly different from those obtained from an automatic metering system and instantaneous measurement facilities commonly used in large manufacturing companies.

The foams were prepared in a Waring Commercial Blender (No. 7011 Model 33BL73) using different mixing speeds (high and low) as well as, different stirring times(1.5 and 2 minutes). The results of foam density measurements taken at both 15 and 65 seconds after foam preparation are shown in tables 5.5 through 5.12. In all calculations the weight of the container is 15.5 g and its volume 190 cm^3.

Figures 5.7a & b show the relationship between foam density as a fuction of the concentration of FA for 1.5 minutes of high speed stirring at 15 ± 5 and 65 ± 5 seconds after foam preparation. Results indicate that an optimum FA concentration is 1% (wt). Further tests were used to determine the relationship between agitation time, density and stirring speed at the optimum FA concentration. Results of these tests are shown in table 5.13 and figure 5.8. Hence, using an optimum FA concentration, with 1.5 minutes stirring at high speed, results in a stable foam having densities of 0.116-0.128 g/cm^3.

Table 5.5

Foam Density Measurement Results

(15 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	38.8	38.9	38.5	38.7	23.2	0.122
1.00	38.6	38.0	38.1	38.2	22.7	0.120
1.25	36.1	36.6	36.1	36.3	20.8	0.109
1.50	35.5	35.7	36.0	35.7	20.2	0.106

Stirring speed scale; Low
Stirring time ; 1.5 Minutes

Table 5.6

Foam Density Measurement Results

(65 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	36.9	37.0	36.7	36.8	21.3	0.112
1.00	36.5	36.8	36.5	36.6	21.1	0.111
1.25	35.0	34.7	35.3	35.0	19.5	0.103
1.50	34.4	35.0	34.6	34.7	19.2	0.100

Stirring speed scale; Low
Stirring time ; 1.5 Minutes

Table 5.7

Foam Density Measurement Results
(15 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	39.6	39.2	39.7	39.5	24.0	0.126
1.00	39.5	39.0	39.1	39.2	23.7	0.124
1.25	38.4	37.8	38.0	38.1	22.6	0.119
1.50	37.7	37.9	37.3	37.6	22.1	0.116

Stirring speed scale; High
Stirring time ; 1.5 Minutes

Table 5.8

Foam Density Measurement Results

(65 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	37.8	38.0	37.5	37.7	22.3	0.117
1.00	37.8	37.4	37.2	37.5	22.0	0.116
1.25	36.9	37.1	36.6	36.7	21.2	0.112
1.50	36.1	36.0	35.8	36.0	20.5	0.107

Stirring speed scale; High
Stirring time ; 1.5 Minutes

Table 5.9

Foam Density Measurement Results

(15 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	38.3	38.5	37.8	38.2	22.7	0.119
1.00	36.4	36.7	36.2	36.4	20.9	0.110
1.25	35.0	35.3	34.7	35.0	19.5	0.103
1.50	35.6	35.1	35.5	35.4	19.9	0.104

Stirring speed scale; Low
Stirring time ; 2 Minutes

Table 5.10

Foam Density Measurement Results

(65 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	36.7	37.0	36.5	36.7	21.2	0.111
1.00	35.0	35.4	35.1	35.2	19.7	0.104
1.25	34.3	34.1	34.6	34.3	18.8	0.099
1.50	34.1	34.0	34.3	34.1	18.6	0.098

Stirring speed scale ; Low
Stirring time (Minutes); 2 Minutes

Table 5.11

Foam Density Measurement Results

(15 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	39.1	39.3	38.9	39.1	23.6	0.124
1.00	39.5	39.0	38.9	39.1	23.6	0.124
1.25	36.5	36.6	36.0	36.4	20.1	0.110
1.50	37.0	37.3	37.1	37.1	21.6	0.113

Stirring speed scale; High
Stirring time ; 2 Minutes

Table 5.12

Foam Density Measurement Results

(65 ± 5 seconds after preparation)

Wt. % Conc.	Wt. with Container (g) Test 1	Test 2	Test 3	Average Wt. (g)	Wt. of Foam (g)	Density (g/cm^3)
0.85	37.7	38.0	37.5	37.7	22.2	0.117
1.00	38.0	37.6	38.3	38.0	22.5	0.118
1.25	35.7	35.3	35.5	35.5	20.0	0.105
1.50	35.2	35.6	35.3	35.4	19.9	0.104

Stirring speed scale; High
Stirring time ; 2 Minutes

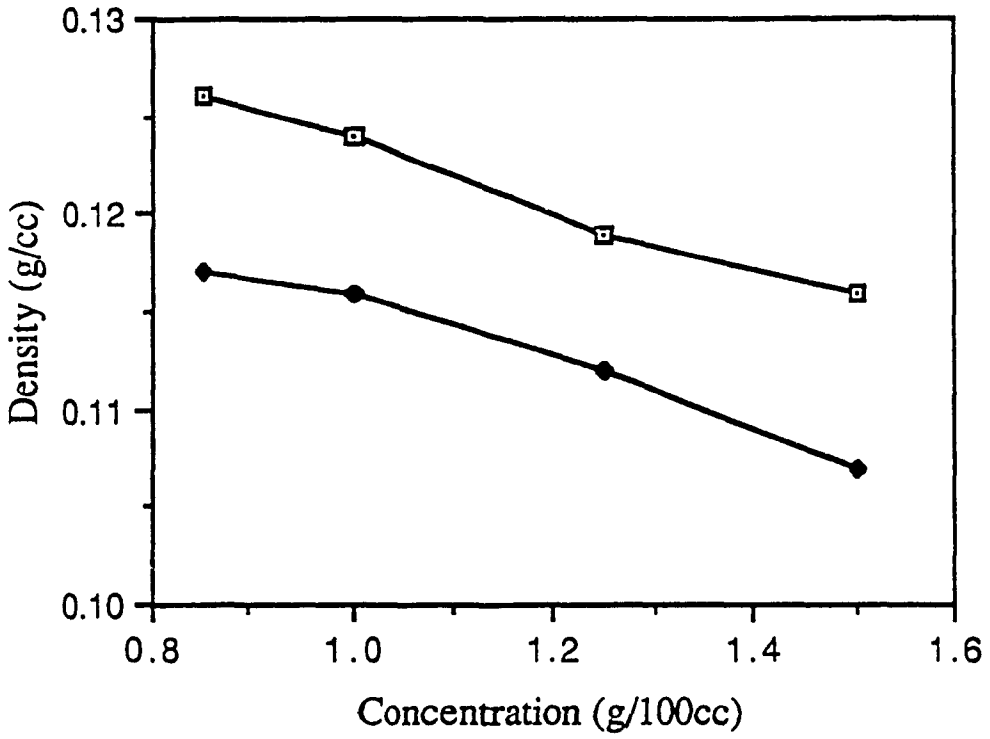


Fig 5.7a Foam density vs FA concentration
Stirring speed; High
Stirring time ; 1.5 Minutes
Foam density measured 15 ± 5 seconds after preparation;
Foam density measured 65 ± 5 seconds after preparation.

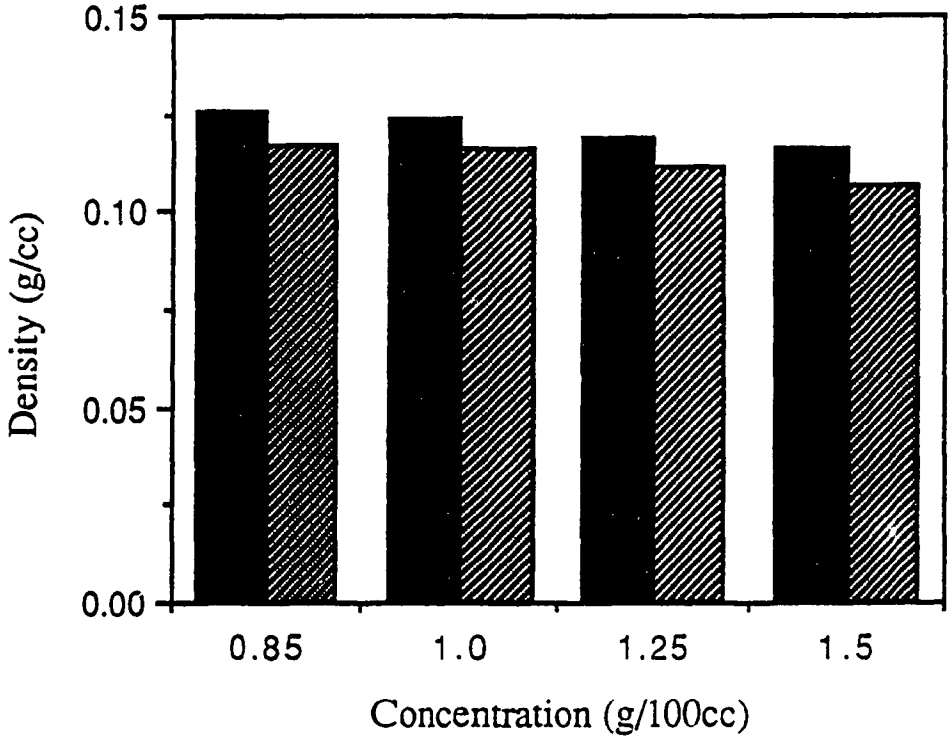


Figure 5.7b Foam Density as a function of FA concentration

Stirring speed; High
Stirring time ; 1.5 Minutes
■ Foam density measured 15 ± 5 seconds after preparation;
▨ Foam density measured 65 ± 5 seconds after preparation.

Table 5.13
Agitation (stirring) time/density measurements results

Test No.	Speed		Agitation time (min)	Density (g/cm^3)
	Low	High		
1	Low	--	1	0.134
2	Low	--	1.5	0.111
3	Low	--	2	0.105
4	---	High	1	0.139
5	---	High	1.5	0.128
6	---	High	2	0.120

Concentration of solution is 1 %

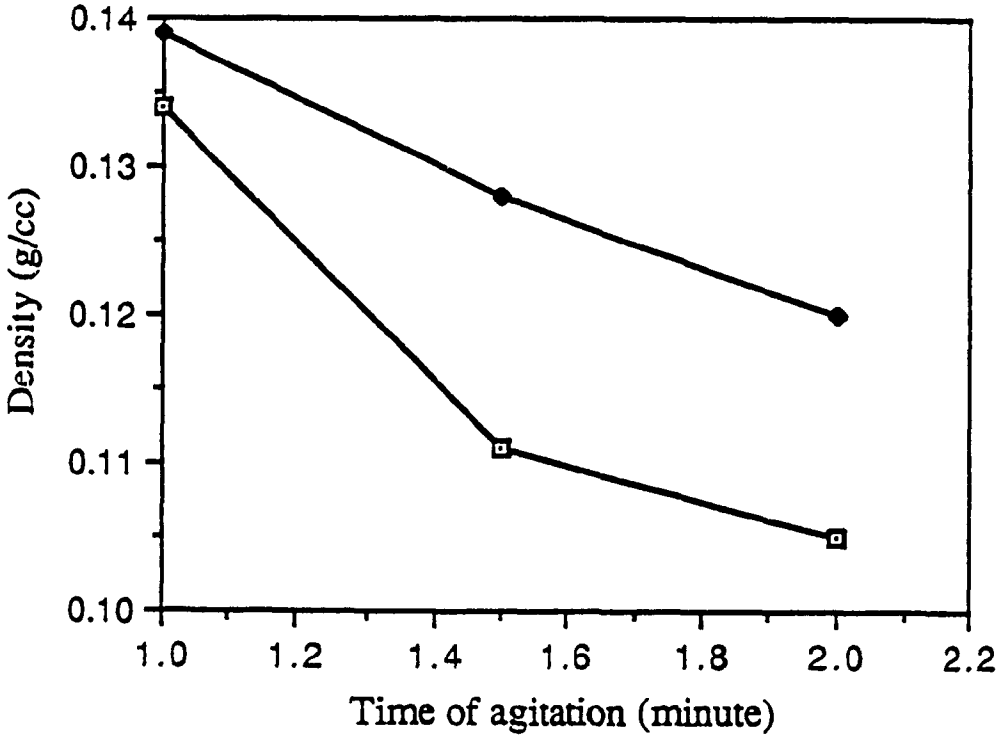


Fig 5.8 Foam density as a function of agitation speed.
-□-; Low speed.
-◆-; High speed

The most important factor among foam characteristics which is vital to the successful production of GB is foam stability (persistence). Foam persistence was evaluating by the amount of foam converted from the foamy state to the liquid state as a function of time and FA concentration. The results of which are shown in Table 5.14 and Figure 5.9.

Table 5.14
Test Results on the Conversion of Foamy State to Liquid State.

Test No.	% Conc. of FA solution (g/100 cc)	Conversion, foamy state to liquid state (cc/5 min)	Persistence (cc/min)
1	0.5	17.0	0.059
2	1.0	7.0	0.143
3	1.5	9.0	0.111
4	2.0	14.0	0.071

Persistance (stability) = 1/conversion of foamy state to liquid state.

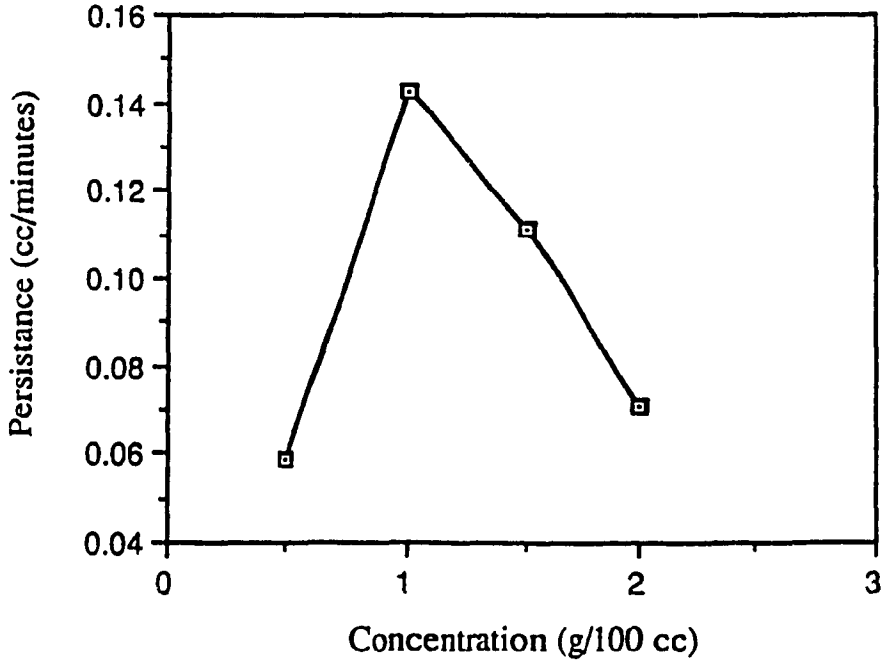


Fig. 5.9 Foam stability as a function of solution concentration.

The foam with the greatest persistence i.e.0.143 cc/min was made using a FA of 1 %(wt) concentration . This is in accordance with previous results obtained for optimum foam density.

5.4.5 Stage 5: Optimization of water quantity

Samples were prepared using w/g ratio of 0.7. However, pastes with different fluidity (workability) were still being obtained. This resulted in specimens having different strengths and different residence times in the drying stage. Consequently, in order to provide specimens having predetermined workability and strength characteristics, the quantity of water required was evaluated. To obtain the desired paste properties, the quantity of water used in the mix was optimized from the point of view of paste workability as well as final board strength. Table 5.15 shows the results of these tests which indicate that a w/g ratio of 0.628 was optimum from the point of view of workability.

The abovementioned set of tests on paste workability were performed on the plain board to determine the optimum amount of water which is required for the hydration reaction and workability. However, the workability of the gypsum paste, when taking into consideration the addition of PCM to the mix, is related not only to the quantity of water but also to the kind and amount of EAs used to prepare the oil in water (o/w) emulsion. The EAs are used to evenly disperse PCM particles in the slurry . Three kinds of EAs were evaluated, the results of which are tabulated in Table 5.16. Optimum values (shown with an asterisks *) of 55, 45, and 40 cc of EA were obtained for WFA, PVA and SSKL respectively.

Table 5.15

Results of water quantity optimization

Test No.	Water quantity (cc)	w/g ratio	Paste Workability
1	240	0.450	Not workable
4	260	0.487	Too sticky, many part could not be filled properly.
7	280	0.525	Sticky, some parts could not be filled properly.
8	300	0.563	Sticky, corners could not be filled properly.
9	320	0.600	Needs considerable vibration
10	330	0.619	Good workability
13	335	0.628*	Excellent workability
14	340	0.638	Slightly fluid
15	350	0.656	Fluid
17	370	0.694	Excess fluididty

* ; Indicates the optimum result

Table 5.16

Results of tests to determine the quantity of EA

Test No.	Type of EA	Water Used (ml)	Water Replaced by EA Solution (ml)	Paste Mixing behaviour
1		325	10	Thorough mixing
	Westroc			impossible
2	Foaming	315	25	Partial mixing
3	Agent	300	35	Slight phase
	(WFA)			separation
4		290	45	Good
5		280	55 *	Very good
1		325	10	Partial mixing
2	Poly	315	25	Slight phase
	Vinyl			separation.
3	Alcohol	300	35	Good
4	(PVA)	290	45 *	Very good
5		280	55	Slightly fluid
1		325	10	Partly mixing
2	Sodium	315	25	Slightly phase
	Salt			Separation.
3	of Kraft	300	35	Good
4	Lignin	290	40 *	Very good
5		280	50	Little fluid

EA: Emulsifying agent

* ; Indicates the optimum result in each category

5.4.6 Stage 6: Introduction of pulp

Results from preliminary mechanical tests performed on selected samples shows that board specimens were quite brittle and that, furthermore, there was a decrease in the mechanical properties in comparision to plain GB. In order to reduce their brittle behavior, which could be detrimental when the product is being shipped or otherwise handled and also to increase the strength of the product, a pulp was prepared from waste newspaper, starch, potasium sulfate and water in specified proportions.

In order to obtain a homogeneous pulp paste and to prevent the evaporation of water in the paste, due to heat produced by the mixing operation, the continuous mixing time was fixed at 25 minutes. This operation was performed in two separate steps of 12 minutes and 30 s each with a 15 minute gap in between each step. The samples prepared by using pulp were shown to be much less brittle and possess greater strength than the previous samples. Consequently, our final mixing procedure for the production of storage board is as shown in Fig. 5.10.

This procedure may, no doubt, be further improved by using automated machines and instruments. These would permit more accurate metering of the various amounts of components and provide control over the stirring time, and residence time in the drying zone during the manufacturing phase. Here, the important point was evaluating the feasibility of mixing of oleous materials in an aqueous gypsum slurry. Results indicate that the laboratory procedure could easily be adopted to the manufacturing phase. Outlined in the

following chapters are the results obtained from performance testing of the products.

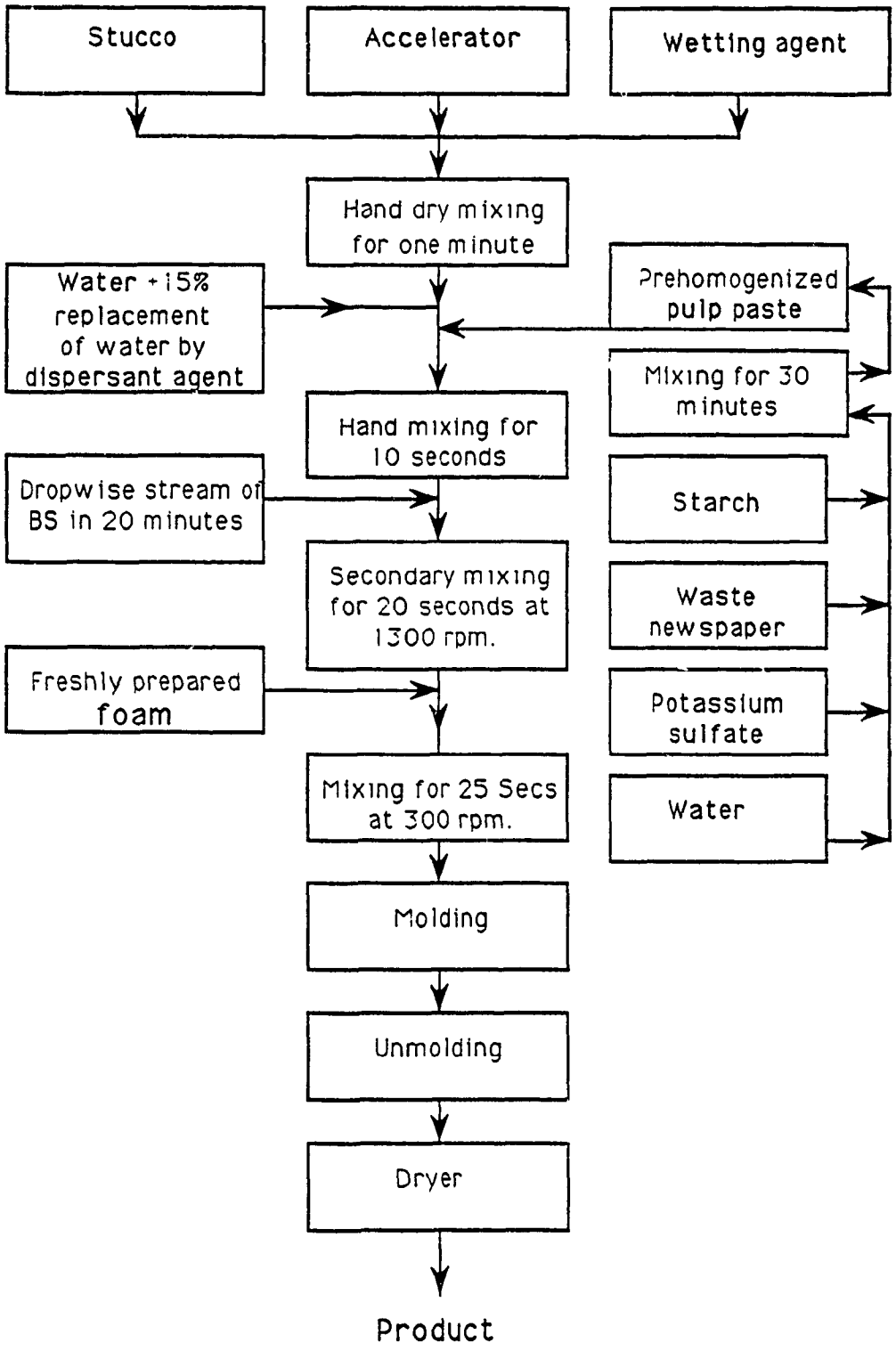


Fig.5.10 Block diagram of the mixing process of storage board production (final procedure).

CHAPTER 6

TESTING OF PRODUCT

6.1 INTRODUCTION

Among the most important industrial uses of materials are their applications in building equipment and structures to perform specified functions under given conditions. To obtain the best performance, the behavior of the material under service conditions must be known. Hence various performance tests were conducted on the energy storage wallboard to evaluate its behavior under specific conditions.

Destructive testing is mostly done to determine the mechanical properties of the products under applied loads and non-destructive testing is commonly used to locate defects in products. Most testing, however, was performed under static loads and applied in such a way that, the impact force was negligible. Visual examination was used, primarily to identify surface defects. Although some tests were performed at either sub-zero or high temperature (@105°C), most were done at room temperature. Finally, when testing, attention was given to such factors as:

- Characteristics of the specimens to be tested.
- Characteristics of the tools and equipment used.
- Accuracy required.
- Testing procedures.

6.2 DESTRUCTIVE TESTS

6.2.1 Compression Test

Compression tests require the specimen to be subjected to a uniaxial compressive loading until it fractures. Specimens for compression tests and density evaluation were prepared (by the same proportion of ingredients as was used in the case of gypsum boards.) according to the ASTM C-472-79.

To find out the effect of the addition of foam, butyl stearate and various emulsifying agents on the strengths and density of the gypsum paste, 4 sets of cubes with dimensions of 5.0 x 5.0 x 5.0 cm were prepared. The samples were conditioned at a relative humidity of 52± 2% for 24 hours and dried at 38 - 42°C in an air circulating oven for a week, until a constant weight was attained. The specimens were then tested in compression using an INSTRON Universal testing machine (Fig 6.1) model 1125. The cross head speed (XHD) was 0.5 mm/min, chart speed of 20 mm/min and full scale load (FSL) from 0 - 50 KN. The results of these tests for various samples are shown in Tables 6.1 to 6.4.

It can easily be seen from the tables that the compressive strength of the samples containing PCM diminishes from 19.44

Kgf/cm^2 to 10.53, 13.16 and 16.07 Kgf/cm^2 for WF, AF, and LF series respectively.

These results indicate that blends incorporating SSKL have enhanced mechanical properties. The use of these DAs also have been shown to be very effective in the mixing stage during slurry formation. These characteristics coupled with the extremly low cost of the lignin derivatives, make them one of the best candidates for use in gypsum products.

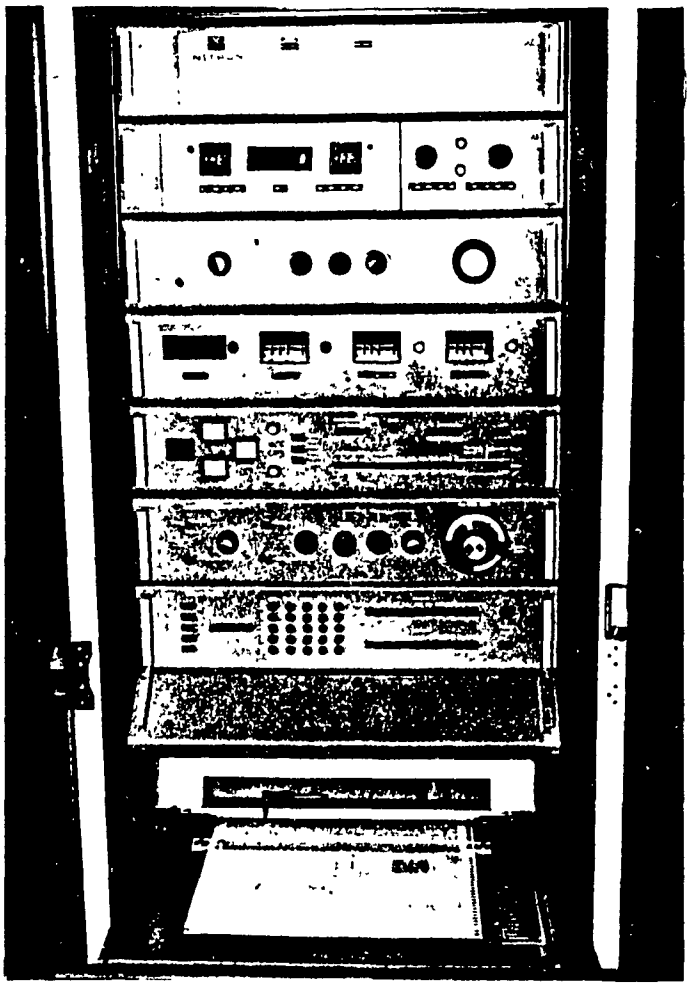


Fig. 6.1a Controlling and recording portion of the INSTRON Testing Machine

The amount of care and effort required to handle and ship products is related to the weight and strength of the products. Lighter products are easier to handle and less costly to ship; resilient boards remain intact even when carelessly handled. The weight and strength of boards is governed by its density, hence this factor must be controlled to within an acceptable range in order to achieve minimum performance standards.

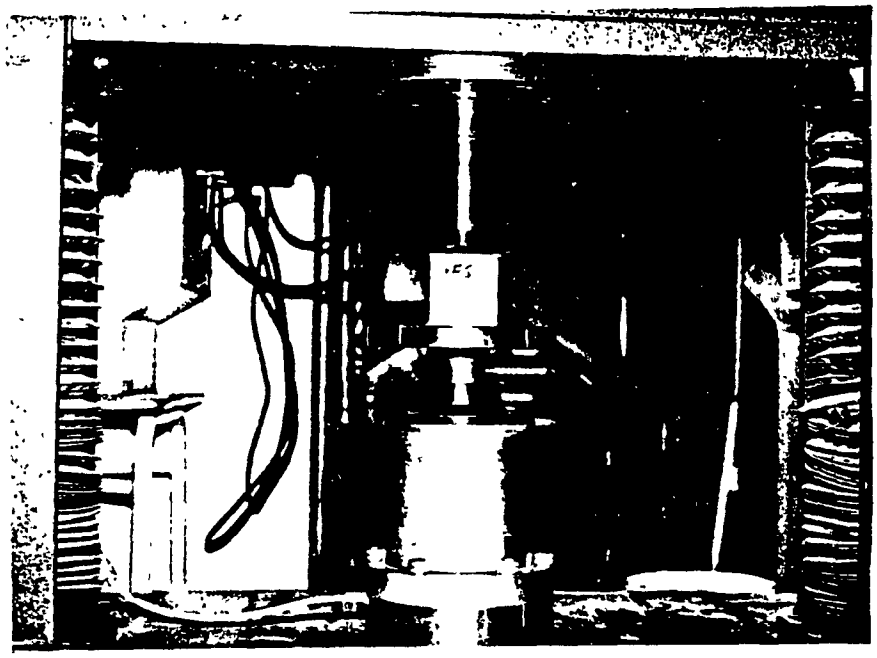


Fig.6.1b Compression test on INSTRON Testing Machine.

Table 6.1
Compressive strength of specimens, PF series

Test Ser.	Test No.	Dimensions of specimen (cm)	Area (cm^2)	Applied Load (N)	Compressive Strength (N/cm^2)	
	1	5.08x5.08x5.08	25.8	4940	191.5	
	2	"	"	4980	193.0	Ave.
PF	3	"	"	4950	191.9	190.7
	4	"	"	4730	183.3	
	5	"	"	4750	184.1	
	6	"	"	5170	200.4	

Paste: Here "paste" means gypsum paste with all ingredients in it for production of the plain wallboard with the exception of foam

PF Series: Cube specimens made of gypsum paste + Foam + Pulp.

Table 6.2
Compressive strength of specimens WF series

Test Ser.	Test No.	Dimensions of specimen (cm)	Area (cm^2)	Applied Load (N)	Compressive Strength (N/cm^2)	
	1	5.08x5.08x5.08	25.8	2550	98.80	
	2			2540	98.40	Ave.
WF	3	"	"	2600	100.8	103.3
	4	"	"	2800	108.5	
	5	"	"	2700	104.6	
	6	"	"	2800	108.5	

WF Series: Cube specimens made of gypsum paste + Foam, pulp, BS and WFA as EA.

Table 6.3
Compressive strength of specimens, AF series

Test Ser.	Test No.	Dimensions of Specimen (cm)	Area (cm^2)	Applied Load (N)	Compressive Strength (N/cm^2)	
	1	5.08x5.08x5.08	25.8	3540	137.2	
	2	"	"	3620	140.3	Ave.
AF	3	"	"	3580	138.6	129.0
	4	"	"	3040	117.8	
	5	"	"	3100	120.2	
	6	"	"	3100	120.2	

AF Series: Cube specimens made of the gypsum paste + Foam, Pulp, BS, and PVA as EA.

Table 6.4
Compressive strength of specimens LF series

Test Ser.	Test No.	Dimensions of Specimen (cm)	Area (cm^2)	Applied Load (N)	Compressive Strength (N/cm^2)	
	1	5.08x5.08x5.08	25.8	3950	153.1	
	2	"	"	4400	170.5	Ave.
LF	3	"	"	4300	166.7	157.7
	4	"	"	3850	149.2	
	5	"	"	3930	152.3	
	6	"	"	3980	154.3	

LF Series: The cube specimens made of the paste + Foam, pulp, BS and SSKL as EA.

6.2.2 Paste density evaluation

Prior to testing the cubes for compression, they were both measured and conditioned. Their densities were determined according to the ASTM 473-7606. Results are shown in tables 6.5 through 6.8.

As can be seen from the Tables 6.5-6.8, the differences in densities are in the range of 0.06 to 0.1 g/cm^3. These differences would add a weight of 2 - 3 kg to a regular panel having dimensions of 244 x 122 x 1.27 cm. This weight difference is what would normally be obtained from manufactured board due to the differences between gypsum board densities and the percentage error regarding the dimension of the wallboard (the foam density used in manufacture of the wallboard normally varies from 0.05 to 0.15 g/cm^3). In comparison to the products manufactured from different companies it can be shown that the excess weight is in the acceptable range (Board manufacturing companies often obtained varying product densities depending on the grade of calcium sulfate used). Decreases in weight can be achieved by using a greater particle size stucco and a higher density foam. Densities for the laboratory prepared gypsumboards also gave similar results; These are shown in Table 6.9.

Table 6.5
Results of density determination PF series

Test Ser.	Test No.	Dimension of Specimen (cm)	Volume (cm^3)	Weight (g)	Density (g/cm^3)	Average Density
	1	5.08x5.08x5.08	131.0965	85.0	0.6521	
	2			86.5	0.6598	
PF	3	"	"	97.0	0.7399	0.6768
	4	"	"	84.8	0.6468	
	5	"	"	86.2	0.6575	
	6	"	"	92.4	0.7048	

PF Series; Cube specimens made of the gypsum paste + foam and pulp.

Table 6.6
Results of density determination WF series

Test Ser.	Test No.	Dimension of Specimen (cm)	Volume (cm^3)	Weight (g)	Density (g/cm^3)	Average Density
	1	5.08x5.08x5.08	131.0965	95.2	0.7262	
	2			96.5	0.7361	
WF	3	"	"	96.5	0.7361	0.7395
	4	"	"	98.0	0.7475	
	5	"	"	98.0	0.7475	
	6	"	"	97.5	0.7437	

WF Series; Cube specimens made of the paste + foam, pulp BS,and WFA as EA.

Table 6.7
Results of density determination AF series

Test Ser.	Test No.	Dimension of Specimen (cm)	Volume (cm^3)	Weight (g)	Density (g/cm^3)	Average Density
	1	5.08x5.08x5.08	131.0965	104.5	0.7971	
	2			103.9	0.7925	
AF	3	"	"	104.6	0.7979	0.7793
	4	"	"	100.0	0.7628	
	5	"	"	100.0	0.7628	
	6	"	"	100.0	0.7628	

AF Series; Cube specimens made of the paste + foam, pulp, BS and PVA as EA.

Table 6.8
Results of density determination LF series

Test Ser.	Test No.	Dimension of Specimen (cm)	Volume (cm^3)	Weight (g)	Density (g/cm^3)	Average Density
	1	5.08x5.08x5.08	131.0965	103.3	0.7864	
	2			103.5	0.7879	
LF	3	"	"	103.0	0.7841	0.7783
	4	"	"	101.0	0.7689	
	5	"	"	101.2	0.7704	
	6	"	"	101.4	0.7719	

LF Series; Cube specimens made of paste + foam, pulp, BS and SSKL as EA.

Table 6.9
Results of density determination of different series of gypsum boards.

T. S	No.	PCM %	Dimensions of Specimen (cm)	Vol. (cm^3)	Wt. (g)	Den. g/cm^3	Ave.
	1	-	20.1x20x1.28	514.56	335.8	0.6526	
W	2	-	20.1x20.1x1.25	505.01	334.2	0.6616	0.660
	3	-	20.2x20.1x1.26	511.58	341.0	0.6666	
		-					
	1	-	20.35x20.35x1.3	538.36	369.6	0.6865	
PP	2	-	20.4x20.4x1.36	565.97	390.0	0.6890	0.688
	3	-	20.4x20.4x1.31	545.17	375.9	0.6895	
	1	21.42	20.35x20.4x1.3	539.68	426.2	0.7897	
BL	2	21.13	20.35x20.35x1.3	546.64	425.6	0.7786	0.783
	3	21.70	20.4x20.4x1.38	570.14	445.1	0.7807	
	1	21.53	20.36x20.36x1.39	576.20	445.9	0.7738	
DP	2	21.36	20.35x20.35x1.34	554.92	442.3	0.7970	0.786
	3	21.60	20.4x20.38x1.37	569.58	449.2	0.7886	

W series; Factory made plain GB samples.

PP series; Laboratory made plain GB samples.

BL series; Laboratory made PCM incorporated GB samples with SSKL as EA.

DP series; Laboratory made PCM incorporated GB samples with PVA as EA.

6.2.3 Bending (Flexural) Tests

Bending tests are conducted in order to evaluate such properties as the modulus of fracture, modulus of elasticity, the ultimate deflection and ductility of the material. Storage wallboard specimens were prepared for flexural tests to a size of 20.32 x 15.24 x 1.27 cm (8" x 6"x 1/2") weighed and conditioned at temperature of 23 ± 2 °C and relative humidity of 50 ± 2 percent until all specimens reached a constant weight.

After reaching a constant weight, the various specimens were tested on the Instron machine (Fig 6.2) for flexural strength under a cross head speed of 2 mm/min, chart speed of 50 mm/min, and full load scale of 0-100 kg. Tables 6.10 - 6.14 show results from flexural tests of the plain, fibrous and the BS incorporated GB.

Tests were conducted according to the CSA A82.20.3-M, in which GBs are tested both perpendicular and parallel to the bearing edges of the test apparatus. Tests show very good results with respect to parallel and perpendicular directions to the bearing edges of the specimens and these results are in full agreement with ASTM standards.

However, in comparison with products made by Westroc and Canadian gypsum Co., breaking strengths of specimens tested perpendicular to the bearing edges showed a slight decrease but specimens tested parallel to the edges are almost in the same range. This decrease in strength is thought to be due to the following factors:

i) Weak bond between core and coverpaper due to the lack of proper facilities with respect to paper application and rigidity of paper in small sizes.

ii) Absorption of BS by facing and backing coverpaper, thereby, decreasing the strength of coverpaper.

iii) Slight saponification of free fatty acid and esters with impurities such as CaO, NaCl, MgO etc. in the slurry.

iv) High percentage of impurities in stucco and consequently less hydration reaction and crystal formation.

v) Lack of simultaneous metering of components.

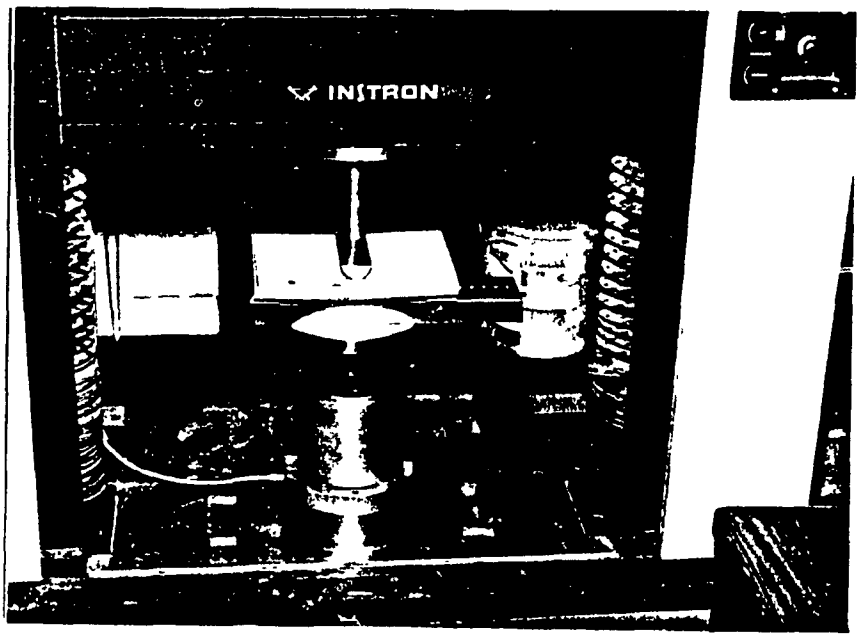


Fig. 6.2 Bending Test on INSTRON Testing Machine.

The factors (i) and (v) could be removed at the manufacturing stage. However more work would be required to investigate the nature of the remaining factors and methods which might be used to counteract their effects.

The specimen size used in the ASTM C-473-76a flexural strength test is 305 x 405 x 12.7 mm. Since it was not practical to produce specimens of standard size in the laboratory, an alternative size of 203 x 152 x 12.7 mm was used. Although the thickness to length ratio of specimens used is not the same as the standard size, the purpose of the test was only to show comparative values between the strength of the regular plain GB specimens and those which have been incorporated with the PCM.

Table 6.10
Results of flexural tests on gypsum wallboard PP and P Series.

Test ser.	Test No.	Direction of fiber, to the edge	Wt. of specimen (g)	% PCM sample	Breaking load /deflection (kg) / (mm)	Ave. load (kg)
	1	Parallel	284.50	-	23.0/0.8	
	2	"	280.04	-	24.5/0.7	21.5
PP	3	"	278.40	-	17.0/0.8	
	4	Perpendicular	284.20	-	50.0/2.1	
	5	"	286.90	-	62.0/2.1	57.0
	6	"	297.80	-	59.1/1.9	
	1	plain	277.50	-	9.5	
P	2	no }	269.54	-	7.3	8.1
	3	cover	260.04	-	7.5	

PP Series: Plain laboratory prepared GB; test span : 150 mm for all samples.
P Series : Laboratory prepared plain GB without back and face coverpaper.

Table 6.11
Results of flexural tests on gypsum wallboard, PW Series

Test ser.	Test No.	Direction of fiber to the edge	Wt. of specimen (g)	% PCM sample	Breaking load/ deflection (kg) / (mm)	Ave. load (kg)
	1	Parallel	242.00	--	27.0/4.3	
	2	"	242.10	--	28.3/5.5 }	28.0
PW	3	"	242.00	--	28.7/5.5	
	4	Perpendicular	242.20	--	60.2/2.5	
	5	"	241.50	--	58.0/2.3 }	59.8
	6	"	242.50	--	61.2/2.6	

PW Series; Plain GB from the Westroc industries.

Table 6.12
Results of flexural tests on gypsum wallboard, PF Series

Test ser.	Test No.	Direction of fiber to the edge	Wt. of specimen (g)	% PCM sample	Breaking load /deflection (kg) / (mm)	Ave. load (kg)
	1	Parallel	257.30	--	20.5/1.1	
	2	"	256.50	--	19.5/0.8 }	19.8
PF	3	"	262.00	--	19.5/0.7	
	4	Perpendicular	259.00	--	65.5/2.7	
	5	"	260.02	--	61.5/1.6 }	64.03
	6	"	257.50	--	65.0/2.8	

PF Series; Fibrious (Fiber glass) GB from the Canadian Gypsum Company.

Table 6.13
Results of flexural tests on PCM incorporated storage GB, DP Series

Test ser.	Test No.	Direction of fiber, to the edge	Wt. of specimen (g)	% PCM sample	Breaking load /deflection (kg) / (mm)	Ave. load (kg)
	1	Parallel	325.4	22.9	17.3/0.8	
	2	"	319.3	22.4	18.5/0.8 }	18.03
DP	3	"	328.5	21.9	18.3/0.8	
	4	Perpendicular	339.7	22.1	44.7/1.7	
	5	"	335.0	22.2	47.8/1.9 }	46.0
	6	"	334.5	21.5	45.5/1.7	

DP Series; Laboratory prepared storage GB composed of BS and 2.5% (weight percentage) solution of PVA as EA.

Table 6.14
Results of flexural tests on PCM incorporated storage wallboard, BL Series

Test ser.	Test No.	Direction of fiber, to the edge	Wt. of specimen (g)	% PCM sample	Breaking load /deflection (kg) / (mm)	Ave. load (kg)
	1	Parallel	337.30	21.4	18.3/0.7	
	2	"	334.00	21.7	18.6/0.7 }	18.16
BL	3	"	340.01	21.3	17.5/0.6	
	4	Perpendicular	327.80	21.5	46.5/1.7	
	5	"	341.70	21.3	52.5/2.0 }	50.33
	6	"	344.20	21.9	52.0/1.8	

DP Series; Laboratory prepared storage GB made of BS and 1.5% (weight percentage) solution of SSKL as EA.

6.2.4 Fire Tests

The main concern when constructing a building, from the point of view of fire safety, is to use components and designs that have been fire tested and approved. A building is composed of three elements which determine its fire safety [50]:

i- The contents of the buildings;

ii- The structural elements; and

iii- The non-structural elements.

Surface contents include furniture, drapes, carpets and anything else contained in a building. These items usually ignite and start a fire.

The structural elements of a building include floors, walls, columns, and beams that, when weakened, can collapse.

Non-structural components such as acoustical tiles, air conditioning ducts and interior finishes are found in all buildings. Many of these components require fire ratings as specified in the appropriate building codes. Since they can contribute to fire load or transfer of fire, their regulation is essential to minimize movement of the fire and its potential lethal effects [50]. Therefore, prime considerations with respect to any building product are its combustibility and the effects of its combustion products. The fire tests conducted in present work, with respect to the energy storing GB were:

- Fire resistance test;
- Flame spread test.

These tests were both preliminary in nature and do not constitute a fire rating. However, they do provide some indication of

the current status of energy storage GB with respect to these properties.

6.2.4.1 Flame spread tests

Flame spread may be defined as the rate of travel of a flame front under given conditions of burning. This characteristic provides a measure of fire hazard in that flame spread can transmit fire to more flammable materials in the vicinity and thus enlarge a conflagration even though the transmitting material itself contributes little fuel to the fire [50]. One measures of flame spread is burning extent or distance of flame travel.

Tests for flame spread have been classified and numerically described by the angle formed by the exposed surface with the horizontal (as in ASTM D 1692, D3014, E84 and E162.).

The surface angle determines the extent to which hot combustion gases will preheat the surface ahead of the advancing flame front. However, a flame spread test requires that successive sections of surface be brought to the ignition temperature as a result of heat flux from the advancing flame. The ignitability characteristics of the material, therefore, have a direct bearing on flame spread.

The amount of heat released from a burning material and the rate at which that heat is released, influence the temperature of the fire environment and the rate of fire spread. In our case, tests were carried out on the flame spread measurement apparatus illustrated in Fig. 6.3. This test has been conducted to obtain some indication of the flame spread characteristics of the BS incorporated GB.

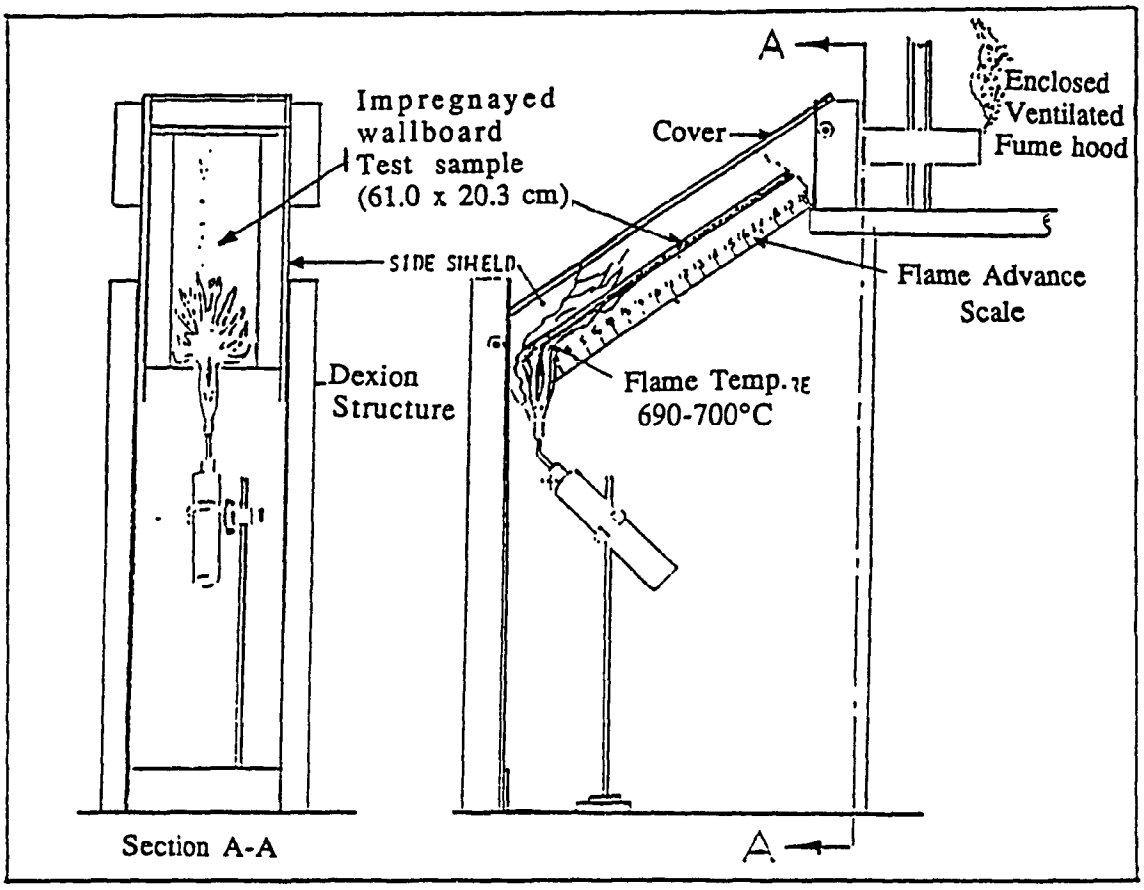


Fig.6.3 Flame Spread measurement apparatus.

Three samples of 20.3 x 20.3 x 1.27 cm were butted together tightly in the above mentioned apparatus at an angle of approximately 30°C from the horizontal . A flame of approximately 700°C was directed vertically upward at a distance of 5 cm from the lower end of the test specimen to strike the sample. Flame travel was observed for 10 minutes against the scale and its extent was recorded. The results of these tests, which are in full agreement with standard ratings, are given in Table 6.15. The physical appearance of the samples after testing is shown in Fig. 6.4.

Table 6.15
Results of fire spread test

Test No.	Type of specimen	Flame front distance (cm)	Observations
01	Plain GB (Lab. product)	15.3	No evolution of smoke; no glowing spot; no continued burning observed after immediate removal of the flame.
02	Plain GB (Westroc product)	15.0	Same as # 01
06	Immersion type BS impregnated GB (Westroc product)	22.0	Traces of smoke; slightly smell of fatty esters; persistance of glowing spot for 3-5 seconds after immediate removal of flame.
03	Immersion type BS impregnated + 10 % fire-retardant *	28.7	Same as # 06
04	Direct incorporated type with 2.5 % PVA as EA	22.7	Same as # 06
05	Direct incorporated type with 1.5% SSKL as EA	23.0	Same as # 06

* ; Fire retardant used was Cereclor-Kronitex (10% of weight of BS.)

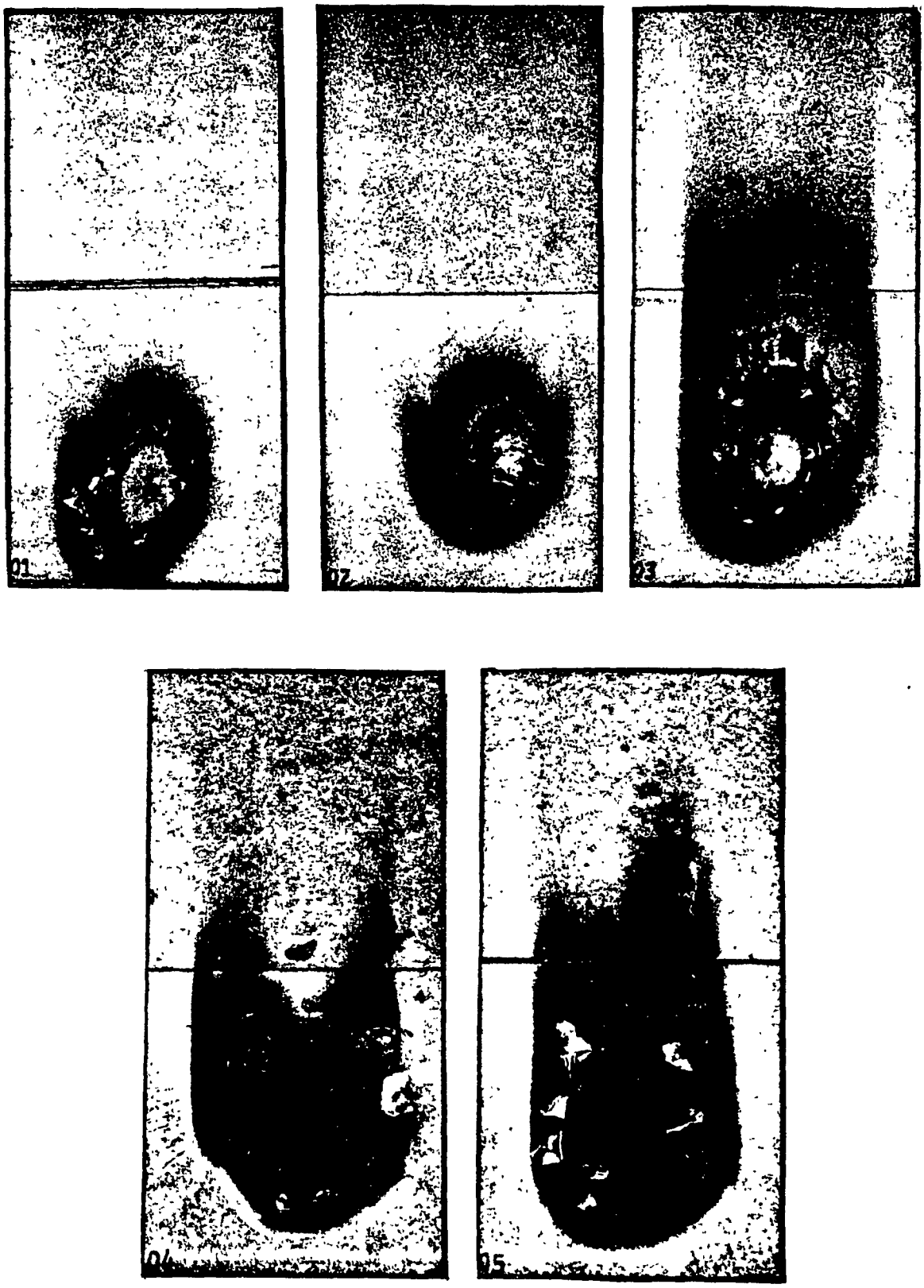


Fig. 6.4 Results from flame spread tests

6.2.4.2 Fire resistance tests

One of the most fire-resistant products is gypsum board. The properties that give GB its fire-resistance are derived, in part, from the crystalline structure of the gypsum core. These crystals contain nearly 50% chemically combined water by volume which results in an effective resistance to fire. When GB is exposed to fire, the temperature of the board slowly increases and stabilizes near the boiling point of water, until most of the chemically combined water is driven off as steam. During this transition the crystalline structure of the gypsum core changes to a denser structure with lower core strength but still enabling it to remain in place as a barrier to fire for extended periods of time [50].

There are, however, some flammability problems due to its paper surface and burn through, due to over calcination. With the incorporation of PCM into the board, this property may change slightly (decrease), due to the organic nature of the PCM. However tests to compare the different products would verify the nature and the amount of the expected changes. In these tests, specimens were subjected to a direct perpendicular flame strike at distance of 5 cm and a temperature of 700°C for 10 minutes. In the case of PCM impregnated GB, both the immersion type and the direct incorporation samples were used. In these tests, no evidence of burn-through on the opposite side of the samples was observed. However, in the case where the plain GB was tested, a slight discoloration of the opposite facing coverpaper was noticed. For the BS incorporated samples, exposed for 20 minutes, smoke evolution

was observed. However,the results of these tests are, preliminary in nature and full scale fire rating and smoke analysis tests must be carried out in the next phase of the work.

6.3 NON-DESTRUCTIVE TESTS

Any test which does not destroy the specimen can be classified as non-destructive. Non-destructive tests have been used primarily for inspection purposes to locate defects in materials and products. However non-destructive tests are increasingly employed to determine properties and characteristics of materials.

Visual examination tests are the oldest and most widely used inspection methods. They are primarily used to identify surface defects. These tests are inexpensive, simple, fast to perform and very flexible whenever possible optical aids such as microscopes or other magnifying lenses might be used. In this work we are interested identifying defects induced by such processes as molding, heat treating, freeze/thaw cycling and painting of our product. A description of results obtained from the non-destructive testing of storage GB is given below.

6.3.1 Cycling Test

Durability of building materials may be defined in terms of resistance to changes in state or property with time or in terms of behaviour in relation to the physical and chemical environments in which it operates [51].

These properties are established for a material in one of the following ways:

- Outdoor exposure tests;
- Accelerated aging tests.

Accelerated aging tests are experiments in which one or more of the stress variables typically encountered in-service, are elevated to the levels higher than normal, thus increasing the rate of degradation of the materials under investigation [52].

In the laboratory testing takes place in controlled conditions and it is often possible to follow the conduct of the test effectively. In this case, the purpose of the freeze/thaw cycling test was to investigate and evaluate the physical or chemical changes, if any, in PCM incorporated storage GB under specified conditions. To determine the effect of aging, the properties were measured before and after the aging test under exactly the same environmental conditions [53].

Testing:

Specimens with the dimensions of 20.3 x 20.3 x 1.27 cm with three different DAs (all other ingredients remained unchanged) were prepared for the laboratory scale cycling test. Three different DAs were prepared with the following %(wt) concentrations.

- 1.5%(wt) solution of WFA, (Commercial grade provided by Westroc industry).
- 1.5%(wt) solution of SSKL (a very cheap byproduct of pulp and paper industry)
- 2.5%(wt) solution of PVA (technical grade).

In addition to the above mentioned samples, specimens were painted with two different types of paints (general application grade of oil and water base paint) as well as normal decorating wallpaper. The application of the paint and the wallpaper to the surface of the GB was to verify their compatibility with the plain and PCM incorporated GB, under freeze/thaw cycling test.

All samples were weighed at ambient temperature of 23°C and 52% RH. They were then placed in a controlled freeze/thaw cycling chamber which permitted a cycling operation of 2-4 cycles per day. Temperatures in the chamber were fixed in a 60-62°C range, between a maximum temperature of +35°C and a minimum of -26°C. The samples were maintained in the chamber for a period of 100 cycles. Figures 6.5a & b show the record sheets of the cycling test. Periodical inspection of the samples was performed during the test. After 100 cycles no apparent physical changes, such as deterioration of the matrix, cracking, blistering or spotting of the paint, peeling off of the coverpaper was noticed. It should be noted that the plain samples were destroyed due to the high level of water condensation inside the chamber. Twelve hours after removal from the chamber the samples were placed in an air circulating oven at 30°C for period of 72 hours, and then were allowed to cool to ambient temperature (23°C and 52% RH) for at least 48 hours, so that they reached the same condition they were in before the cycling test. They were then weighed to observe any change in weight. The details and results of the tests are shown in Table 6.16.

As the results show, the plain gypsumboard samples were softened under the aforementioned conditions. This, on one hand,

shows that the plain GB can not be used in a high humidity area and on the other hand, demonstrates the degree of impermeability of the thermal storage GB in moist areas. This is a very positive attribute of the thermal storage GB.

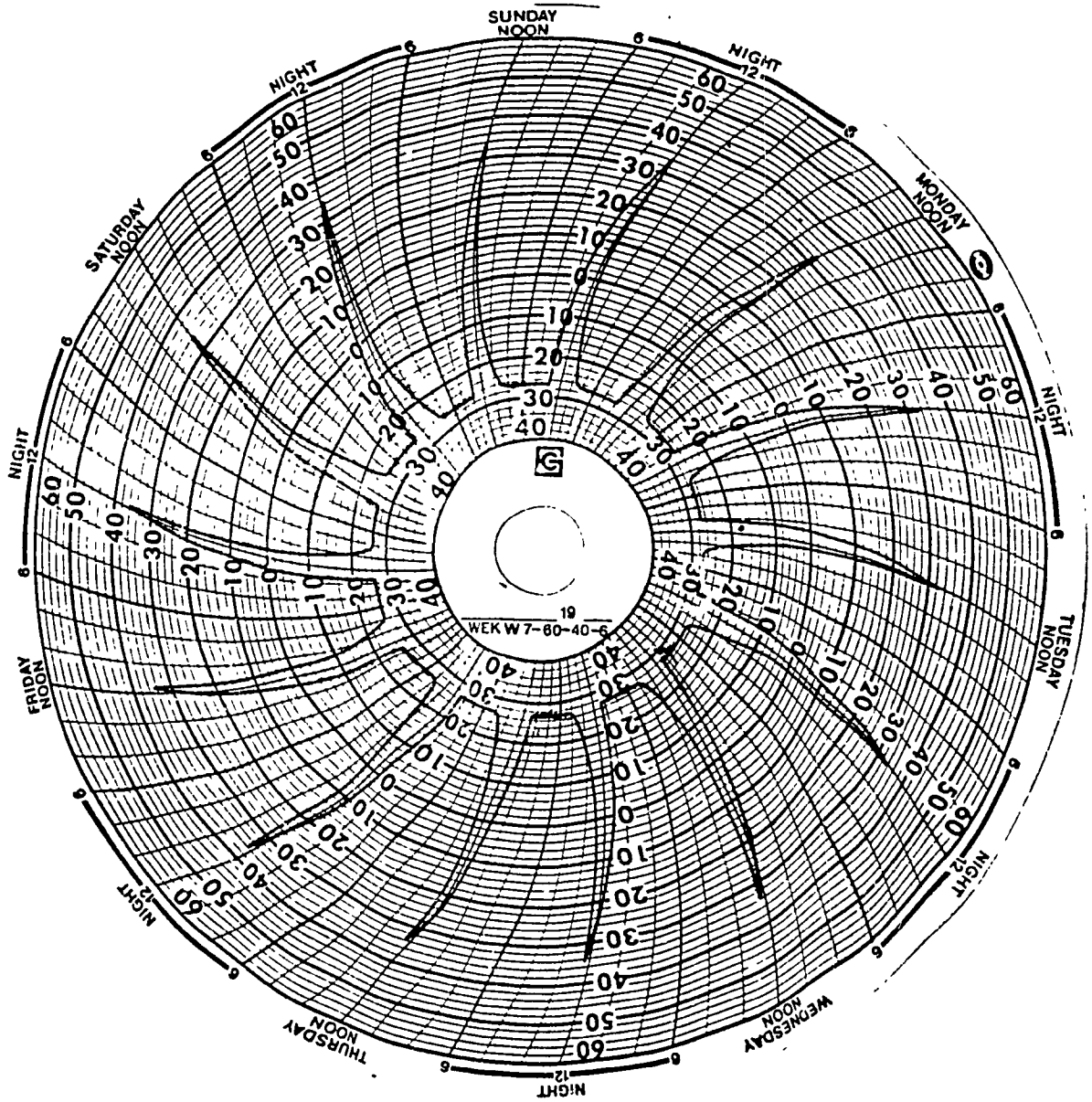


Fig.6.5a Record sheet of freezing/thawing cycling test on storage board (2 cycles per day).

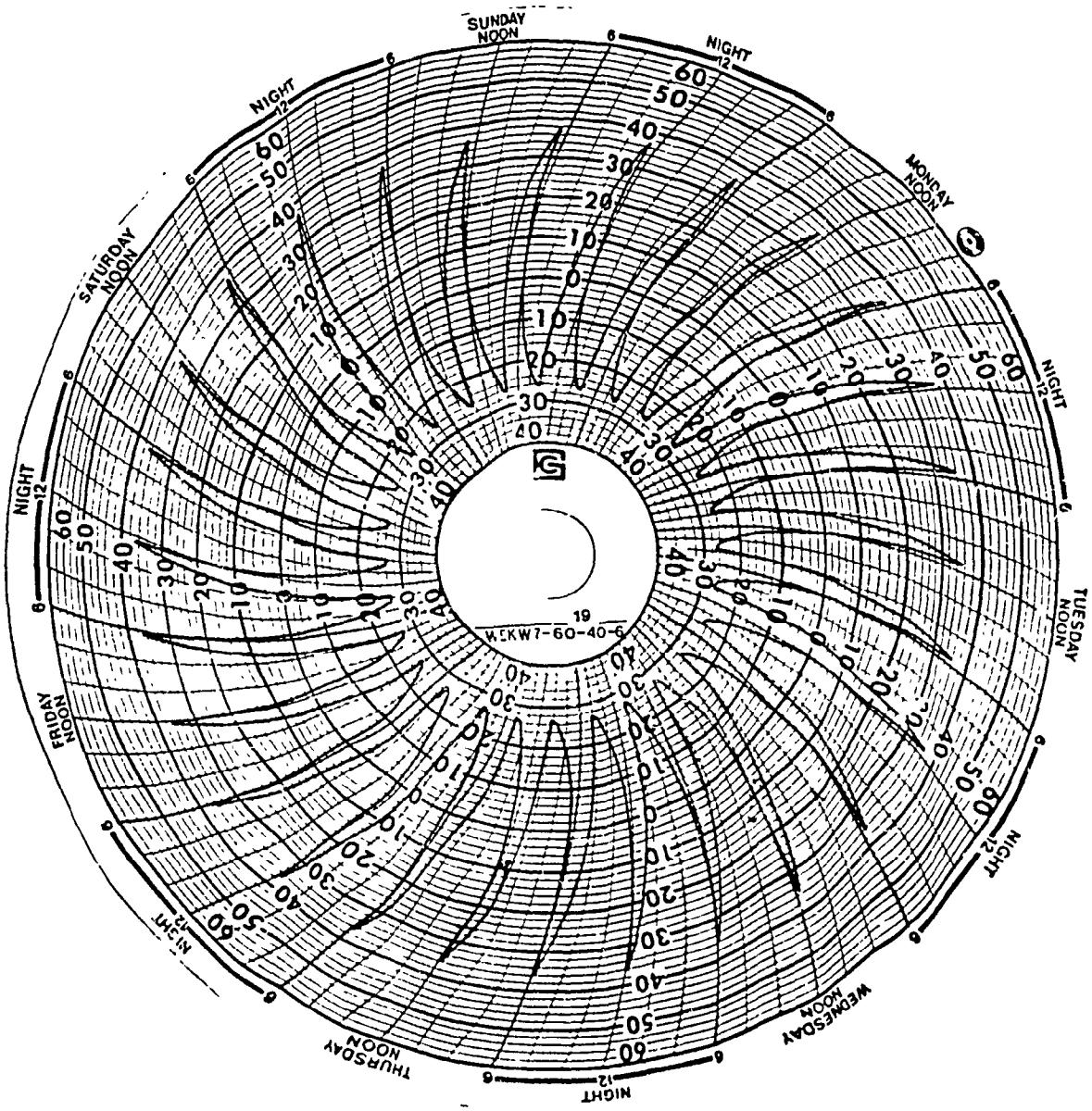


Fig6.5b Record sheet of freezing/thawing cycling test on storage board (4 cycles per day).

Table 6.16

Results of freeze-thaw cycling test on thermal storage wallboard

Test Series	Board Type	PCM %	Weight (g) Before	After	Δ	Weight Increase %	Oil exudation after 100 cycles
P1	Plain (Westroc)	-	342.3	351.5	9.2	2.68	softened
P2	"	-	333.9	342.7	8.8	2.63	softened
P3	Plain lab. made	-	330.7	340.1	9.4	2.84	softened
P4	"	-	329.1	336.6	7.5	2.27	softened
IP1	Immersion Type	24.2	422.2	426.1	3.1	0.92	None Visible
IP2	"	25.0	416.6	420.7	4.1	0.97	"
IP3	+5% FR/836	24.6	414.2	418.1	3.9	0.94	"
IP4	+15%FR/836	24.0	412.0	416.3	4.3	1.04	"
DP9*	+Waterbase Paint	24.0	411.4	415.6	4.2	1.02	Stained slightly at edges
DP12	No Treatment	23.0	430.0	433.7	3.6	0.84	"
BL2*	+Oilbase Paint	23.7	456.5	460.0	3.5	0.77	"
BL6+	+Wallpaper facing	23.2	465.9	469.8	3.9	0.83	"
BL10	No Treatment	22.8	457.0	460.7	3.7	0.81	"

P Series ; Westroc and lab. made plain GBs.
IP Series ; Westroc made, immersion type specimens.
DP Series; Laboratory prepared specimens with 2.5 weight % of PVA as EA.
BL Series; Laboratory prepared specimens with 1.5 weight% of sodium salt of KL as EA.
* ; Shows samples with two coats of paint (DP9 covered with waterbase paint, and BL2 with oilbase paint.
** ; Samples with one coat waterbase paint and wallpaper covering.

6.3.2 Paint and Wallpaper Compatibility

6.3.2.1 Paint tests

Since some PCM materials can react with paint or the adhesives found on wallpaper, it is important to establish the compatibility between these materials. These tests are only intended to demonstrate the feasibility of such combinations and can not be considered a comprehensive compatibility test. Oil and water based paints were applied to the laboratory prepared samples of the direct BS incorporated wallboard to assess their compatibility. Representative paints were applied as follows:

- One undercoat of oil based paint, (premium interior alkyd, white paint, No.35650) followed by a finishing coat of oil based paint (premium interior alkyd pearl No. 35610.)
- Two coats of water based paint (white semi glass latex No. 35560.)

The following results were obtained for compatibility test with respect to paint covering.

i) Examination of the painted specimens in ambient conditions for three months demonstrated the apparent compatibility between paint and coverpaper.

ii) Compatibility test performed in the cycling chamber for one month. These tests indicate that there was no apparent change in paint condition, (eg. cracking, peeling, blistering and or flaking).

iii) Tests in 50°C temperature for 168 hours in an air circulating oven, did not show any apparent visual change.

iv) Face down exudation tests were performed. These did not give rise to any exudation of the BS nor was any pelling and blislering of the paint observed.

v) Test were conducted with samples containing different percentage of BS to determine their compatibility with respect to oil and water base paints.

Incorporated samples of 20.3 x 20.3 x 1.27 cm nominal size were painted with the afore-mentioned types of paints, and were examined after progressive periods of time at ambient temperature. The result of tests are shown in Table 6.17. Results indicate that the quantity of BS affects the early drying time of oil based paint but does not affect that of the water based paints. These results demonstrate the compatibility of BS storage GB with different paints.

6.3.2.2 Wallpaper compatibility test

Two types of normal prepasted wallpaper which was used for this compatibility:

- Light, pulp based wallpaper;
- Heavy, fiber based, knitted cross fibrous wallpaper.

In both cases, as in the case with paint compatibility test, test points (i) to (v) were performed for wallpaper compatibility test as well. The results indicate that there were no signs of apparent physical change in the condition of the wallpaper. A more comprehensive test including many different types of wallpaper would be beneficial.

Table 6.17
Compatibility test results for BS incorporated wallboard with common types of paints.

Test No.	BS %	Type of paint							
		Oil Base				Water Base			
		48 hrs	72 hrs	7 days	30 days	48 hrs	72 hrs	7 days	30 days
1	21	dry	dry	dry	dry	dry	dry	dry	dry
2	23	"	"	"	"	"	"	"	"
3	25	Sticky	Sticky	"	"	"	"	"	"
4	27	Sticky	Sticky	Sticky	"	"	"	"	"

BS; Butyl Stearate

6.3.3 Water absorption test

In consideration of the fact that wallboard is often used in places where the degree of moisture is high (e.g. bathroom areas) it would be considered essential to verify the degree of water absorbancy of the PCM board. To test this property, samples of 20.3 x 20.3 x 1.27 cm of both plain and BS incorporated boards were weighed at 23°C and 52% RH. They were then placed in a bath of clean tap water (20°C) for a period of 2 hours. At the end of the immersion time, the samples were drained for 1 minute and wiped dry using a paper towel, and immediately weighed to determine the percentage of water absorption. The results of this test are given in Table 6.18.

Table 6.22
Water absorbancy of plain and PCM incorporated board

No.	Sample Type	PCM %	weight (g)			Weight Increase %
			Before	After	Δ	
PP4	Plain board (Lab.)		376.0	573.8	197.8	52.0
PW3	Plain board (WR)		341.6	511.3	169.7	49.7
DP1	PCM wallboard with PVA as EA	22.2	425.5	496.7	71.2	16.7
BL1	PCM wallboard with SSKL as EA	21.4	455.7	511.7	56.0	12.3

Clearly, water absorptance depends on the porosity or volume of voids present in the material. Gypsum wallboard is a highly porous material with a random distribution of voids due to the intentional use of foam in its production. Being a porous material implies having a high capacity of water absorption.

In BS incorporated GB the fatty esters fill up a certain percentage of voids in the board. Furthermore, fatty acids and their ester derivatives are highly water repellent organic compounds which would not allow easy passage of water molecules to the voids. These two factors clearly account for the less hygroscopic nature of the BS incorporated GB. As has been seen from the test results, the moisture absorption property of the board is decreased by almost 50% which indicates that BS incorporated GB may be used in those areas which have higher moisture contents.

6.3.4 Olefactory Test

Twelve samples (20.3 x 20.3 x 1.27 cm) of BS incorporated wallboard using PCM samples, Emery 2325 which were (six years old and six months old) prepared to detect and differentiate the smell of the PCM incorporated specimen.

i) Two sets of clean plastic containers were used which could easily be sealed.

ii) Each group of six samples were placed in a separate container and sealed for a week to acertain the possible existance of any objectionable smells (gases) which could be collected in the container. The presence of any odour could subsequently be detected by the olefactory senses.

Five persons out of ten, could not detect any strong smell; three noticed a slight, unidentifyable (but not objectionable) smell, and two noticed a slight (almost negligible) smell of oil and or fatty substances.

These tests clearly show that there is no significant gas evolution from the samples (i.e. no chemical reaction occurs), and the smell is believed to be due to the light impurities (PCM is 98% pure) present in the PCM. Further purification of the PCM distillation under vacuum should reduce the smell to non-sensible levels, or it can be nullified by addition of a trace of a pleasent and chemically neutral aromatic ester (normally used in cosmetics).

CHAPTER 7

ANALYTICAL TESTS

7.1 TIME OF SETTING

Tests were performed on plain as well as BS incorporated gypsum paste using three different types of EAs to compare and determine the effect of BS and surface active agents on the time of setting.

A multiple channel recording potentiometer (DORIC) with indicator and multi-point selector having a sensitivity of ± 0.5°C was used to record the temperature change of the specimens being tested. A heat sink compound was used to transmit heat between the thermocouples and surface at which the temperature was measured.

The paste was prepared according to the ASTM C-472 in which 200 g of dry components were mixed with wet components excluding accelerator or retarder, in the same proportion as had been used for the other tests. The paste was then introduced into a waxed paper cup (268 ml) which was placed inside another cup, and the entire assembly held in a rigid polystyrene foam insulation block (Fig 7.1).

Five temperature sensers were positioned one third the distance up from the bottom of the cup. The first one was in the core of the inner cup, the second one on the inner side wall of the first cup, the third, fourth, and the fifth ones were placed between the inner and outer walls of adjacent cups, between the outer cup and the insulation block and on the outer wall of the insulation box respectively. After preparing the apparatus, tests were conducted on four groups of specimens.

The results were recorded from the time the samples were first introduced into the water to the time of maximum temperature rise. The latter corresponded to the time of setting. Table 7.1 shows the results of tests of the core and interface between the cups (complete results of these tests are given in appendix A) and Fig 7.2 to 7.6 shows the time-temperature relationship among the four groups.

As can be seen from Table 7.1, the setting time of the BS incorporated pastes was increased by 3.7, 5 and 2 minutes corresponding to 4, 4 and 3°C rise in peak temperatures, for WFA, PVA and SSKL additives respectively.

On the one hand, these changes may affect the speed of production in the manufacturing process (this can be solved by adding a small amount of suitable accelerators) and, on the other hand, they would decrease the mechanical strength of the product due to the smaller hydration reaction which, in turn, implies less crystal formation.

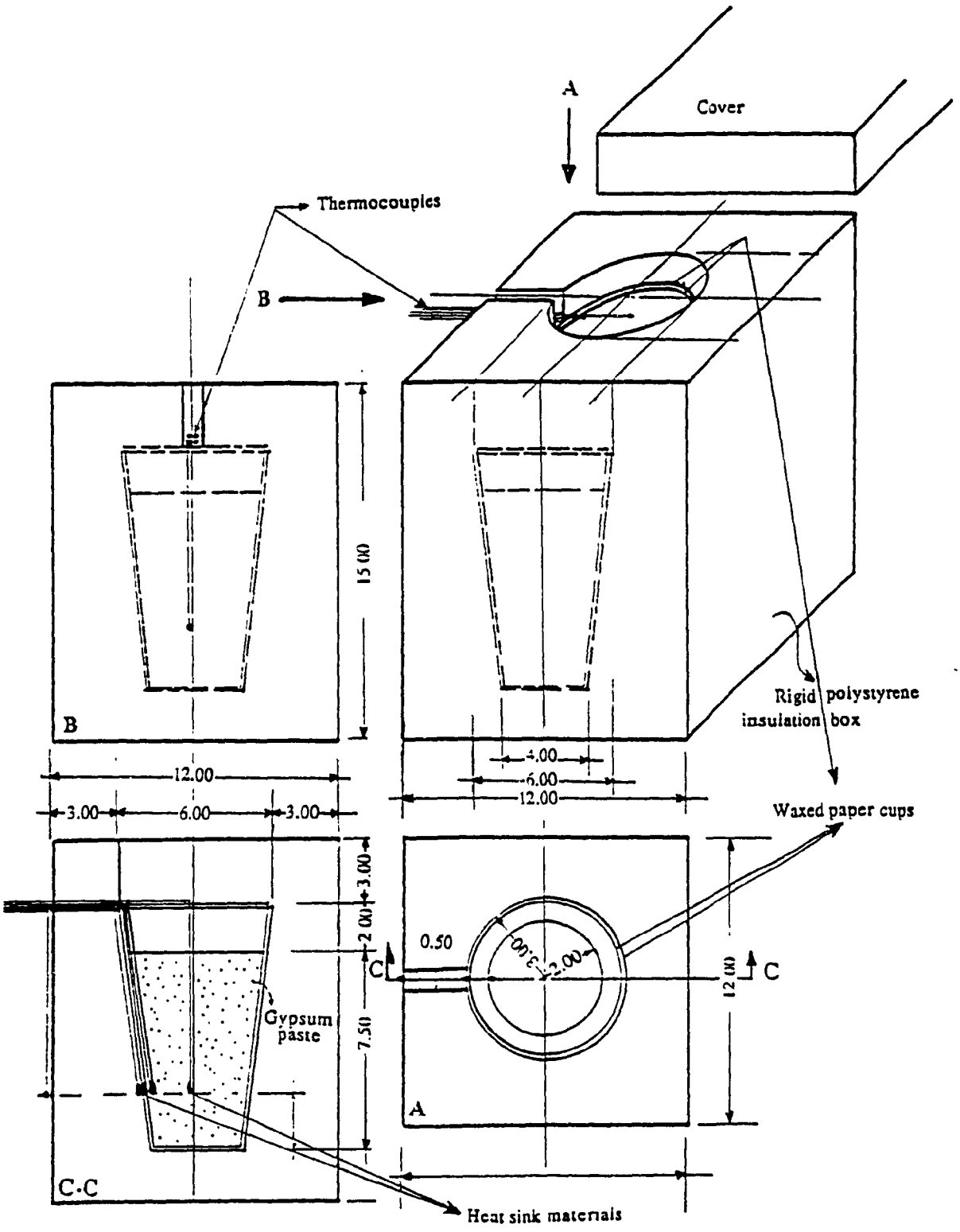


Fig. 7.1 Insulation box apparatus for time of setting measurement.

Table 7.1
Thermal setting time of plain, and BS incorporated gypsum paste.

Type of Paste	Test No.	Time elapsed (min)	Peak Temp (°C)		Ave. setting time (min)
			Core	Interface betw. cups	
Plain Paste	1	17	53	50	
	2	18	53	50	17.6
	3	18	53	50	
PCM incorp. paste with WFA as EA	1	21	49	48	
	2	21	49	47	21.3
	3	22	49	47	
PCM incorp. paste with PVA as EA	1	23	49	47	
	2	23	49	47	22.6
	3	22	49	47	
PCM incorp. paste with SSKL as EA	1	19	50	48	
	2	20	50	48	19.6
	3	20	50	48	

No accelerator was used in these tests.
Materials and mixing water were maintained at 20-22°C and tests performed at the same temperature.

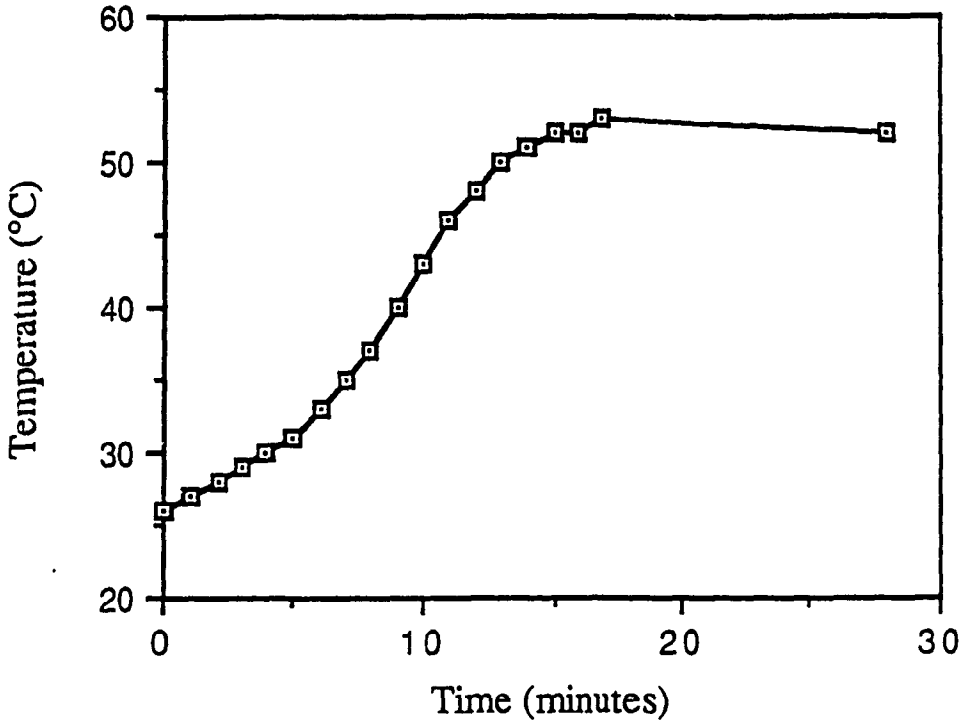


Fig.7.2 Time/Temperature relationship of hydration reaction in plain gypsum paste.

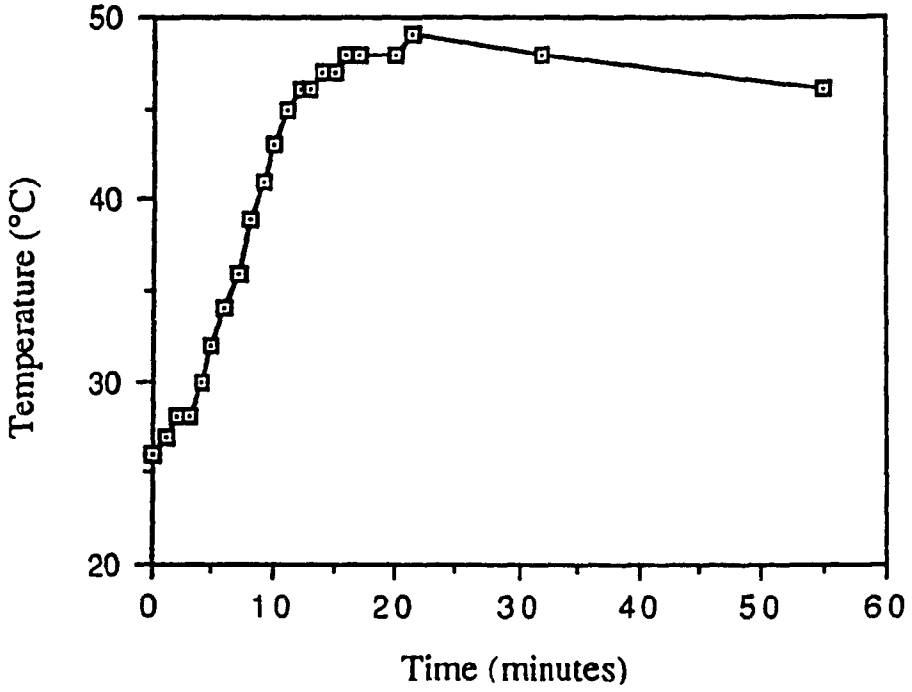


Fig.7.3 Time/temperature relationship of hydration reaction in PCM incorporated gypsum paste with WFA as EA.

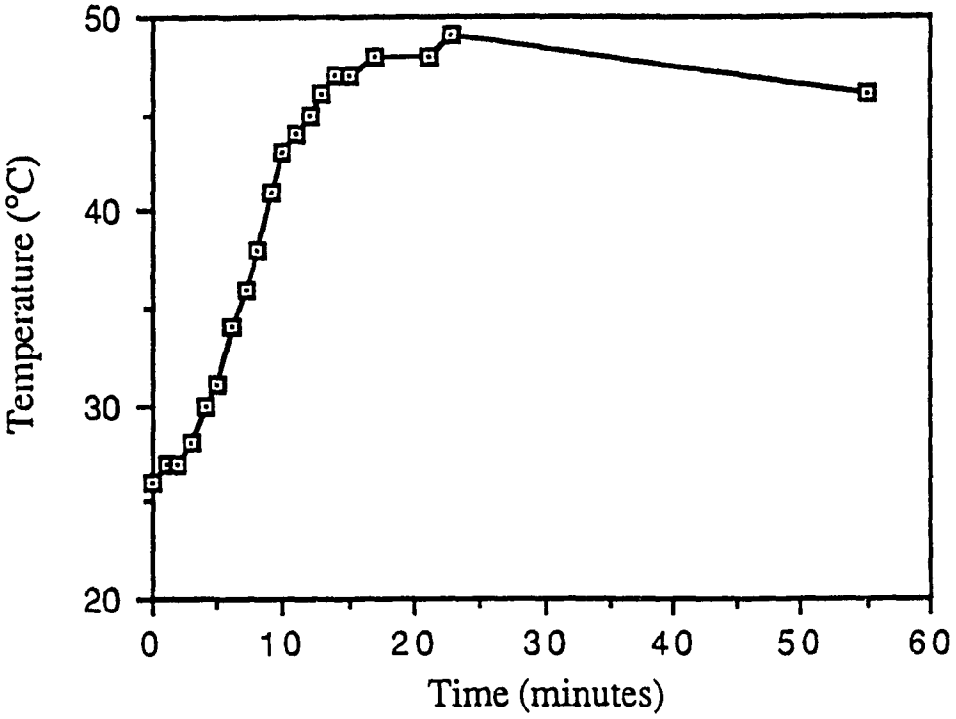


Fig.7.4 Time/temperature relationship of hydration reaction in PCM incorporated gypsum paste with PVA as EA.

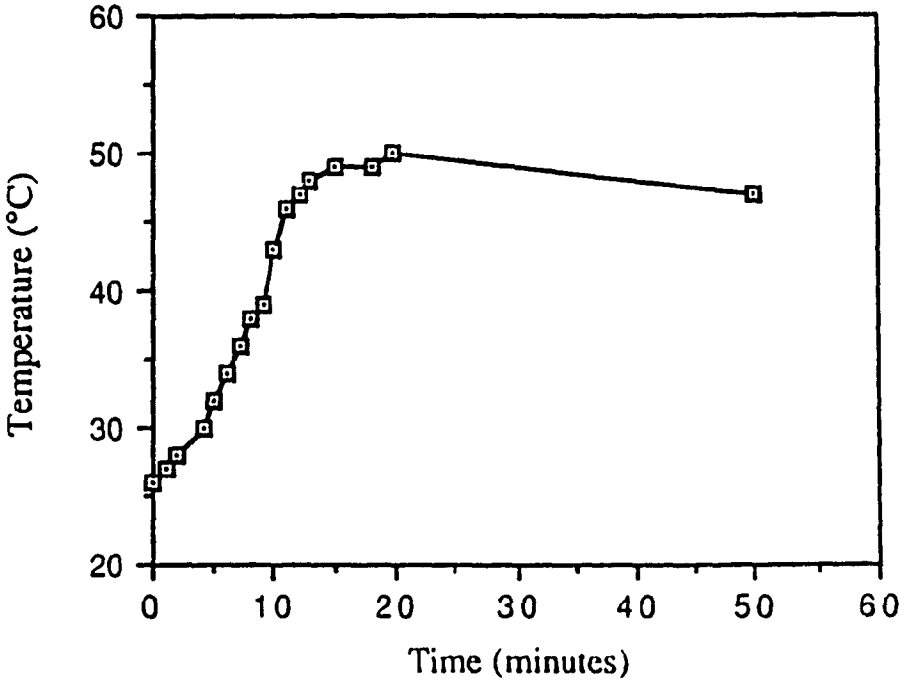


Fig.7.5 Time/Temperature relationship of hydration reaction in PCM incorporated gypsum paste with SSKL as EA.

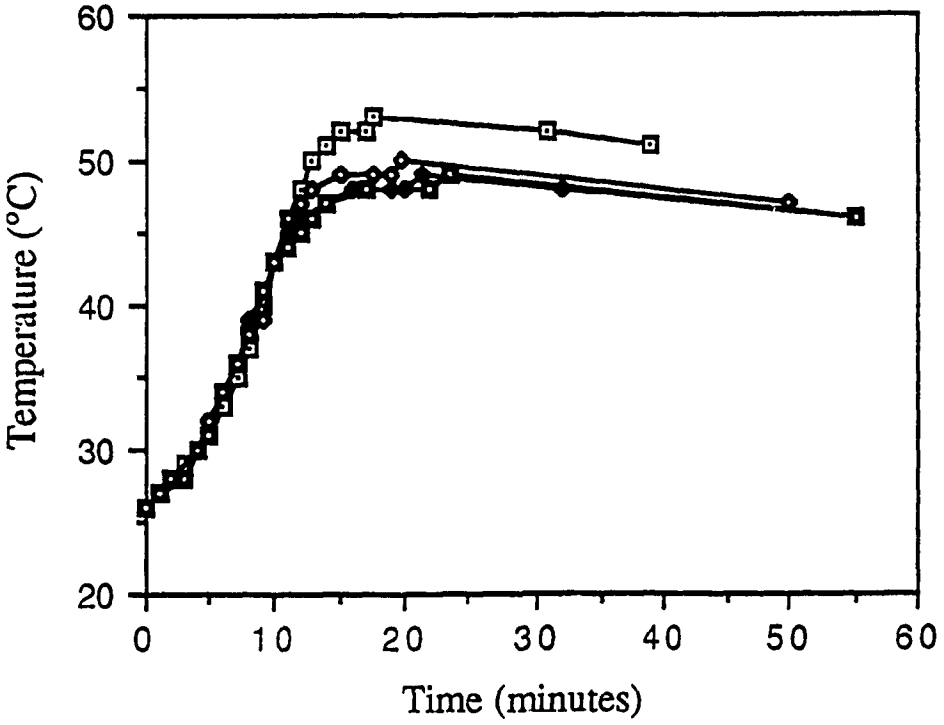


Fig.7.6 Comparative Time/Temperature relationship.

-□- : Plain paste

-◆- : PCM incorporated paste with WFA as EA

-■- : PCM incorporated paste with PVA as EA

-◇- : PCM incorporated paste with SSKL as EA

7.2 DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS

The primary purpose of this method is to detect enthalpic changes and to approximate the initiation temperatures of these events. The method of differential scanning calorimetry offers the advantage of using very small samples, in the order of few milligrams. DSC techniques may be performed on solids, liquids or slurries. A quantitative measurement is made of the energy (ΔE) associated with the observed change of enthalpy, [ASTM-E537].

Thermal analysis, i.e. the determination of transition temperature, heat of fusion and heat of crystallization of directly incorporated BS gypusmboard, were conducted by differential scanning calorimetry (DSC) method by using a Dupont model 910 differential scanning calorimeter (Fig.7.7). The instrument consist of a cell base module which has been directly connected to a programmer-recorder thermal analyser (Dupont 1090) as shown in Fig.7.8. The device has the following operational range.

- Temperature range from - 160 to 600°C.
- Heating rate from 0.5 to 100°C/min
- Temperature repeatability of ± 1°C
- Latent heat variation of 1%

The DSC cell (Fig.7.9) has a thermoelectric constantan (an alloy of copper containing 10-55% nickel as its electrical resistance which does not vary with temperature) disk as a primary heat transfer element which conducts heat to the sample as well as an empty reference pan which is positioned on the raised portion of the disk. The temperatures of the pan with the sample and empty reference pan are measured by constantan-chromel thermocouples.

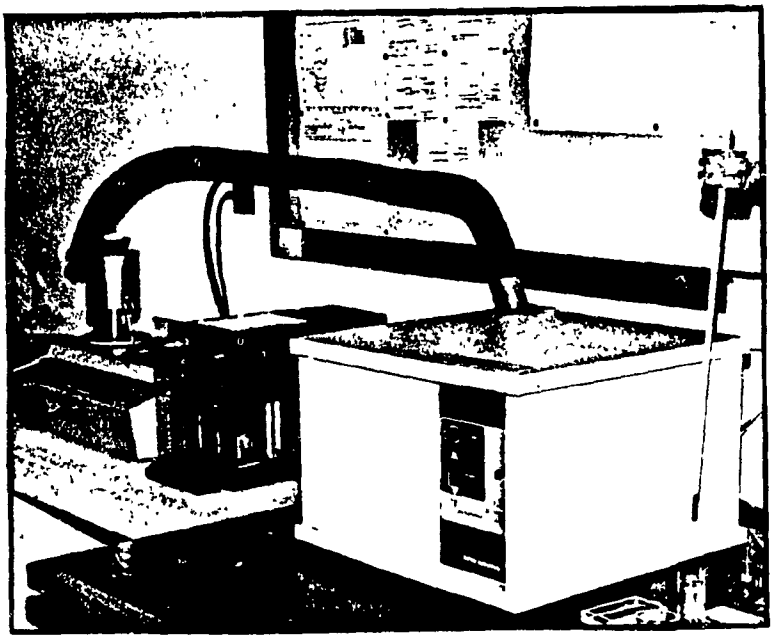


Fig.7.7 Dupont Model 910 Differential Scanning Calorimeter.

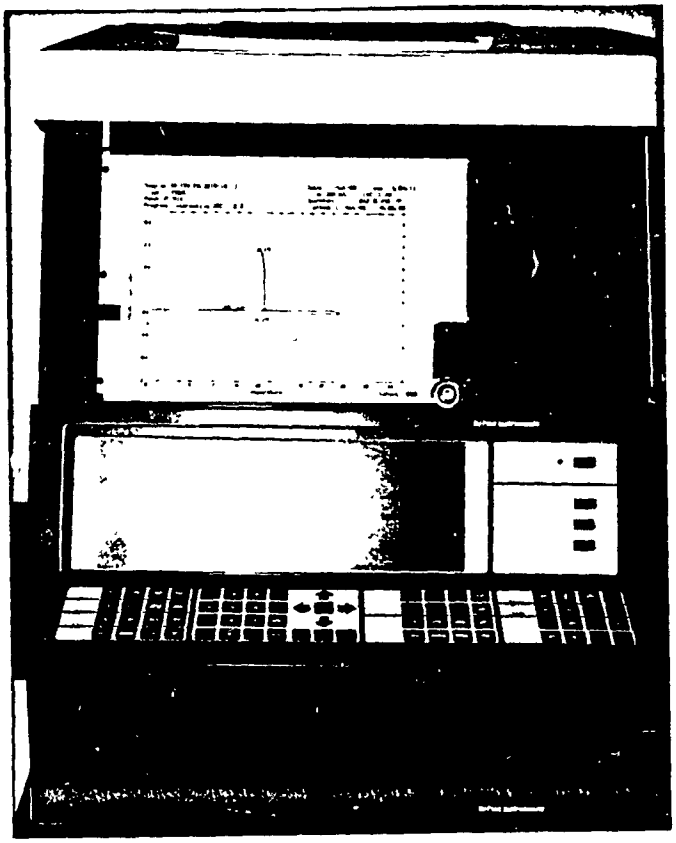


Fig.7.8 Dupont Model 1090 Thermal Analyser programmer/recorder.

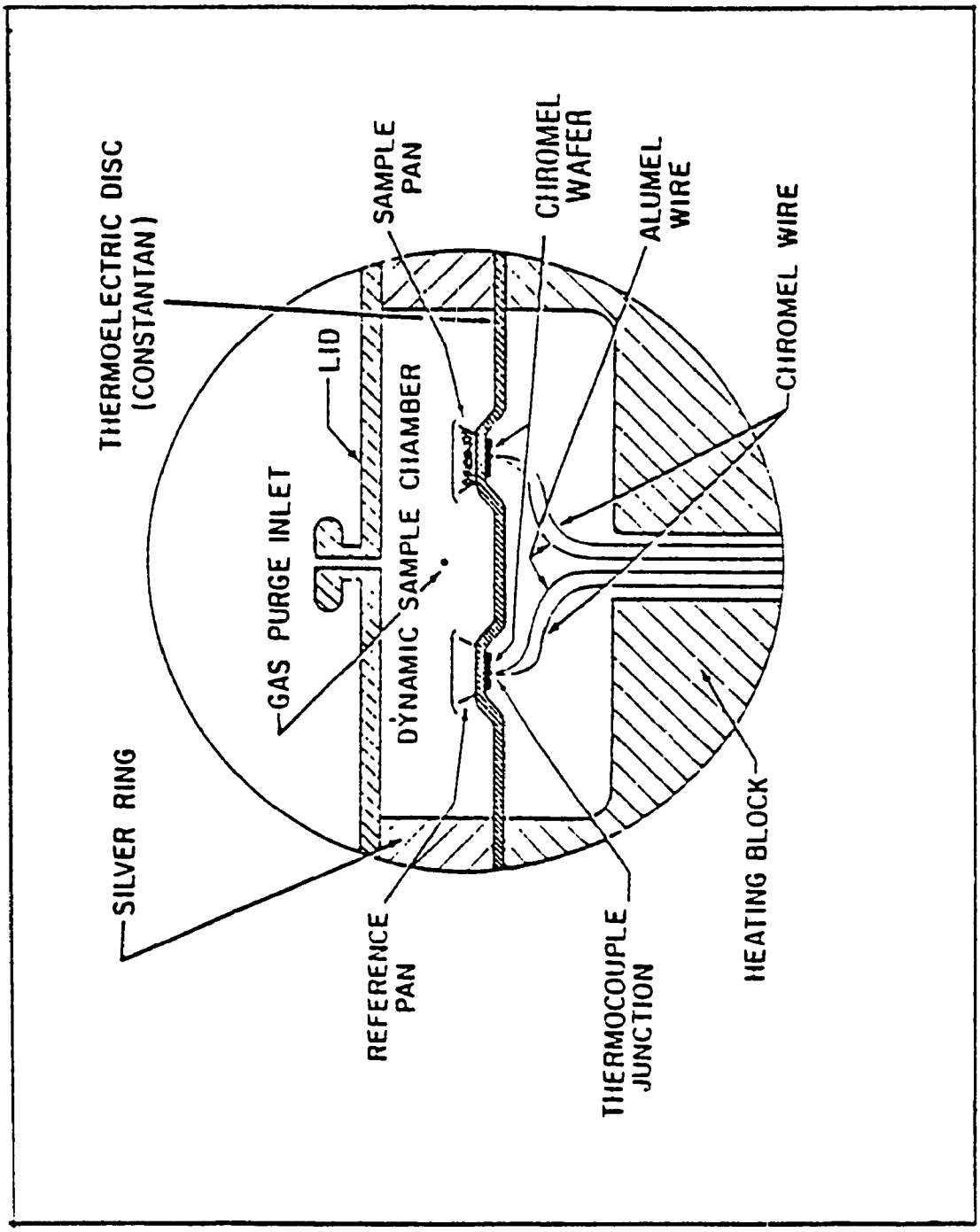


Fig.7.9. DSC Cell cross section [38].

Since the two pans are identical, heat flow differences due to the presence of the sample, can be measured and calculated by using the known weight of the sample.

This measurement can be stored or plotted as a curve with heat flow (ΔE) on the ordinate and the temperature (T) on the abscissa. The data, in turn, is analyzed using an interactive data analysis program. This program, which has been provided by the manufacturer, calculates the temperatures and heats of transition associated with the sample materials.

Having precisely controlled environmental conditions for both sample and reference pans, the method provides an accurate measurement of the transition temperature. This value is calculated by integration of the related heat flux vs time curve. Typical thermograms of the heating/cooling cycle during the testing of specimens is shown in Figs.7.10-7.13.

Different parameters are used to evaluate the various thermograms. These are briefly explained below:

i) Melting point

The initial melting point (m.p.) is the temperature at which the solid and liquid phase of a substance are in equilibrium at standard atmospheric pressure. The m.p. temperature may be calculated directly from the thermogram by plotting the intersection of the base line on the heating curve with the tangent to the inflection point on the same curve.

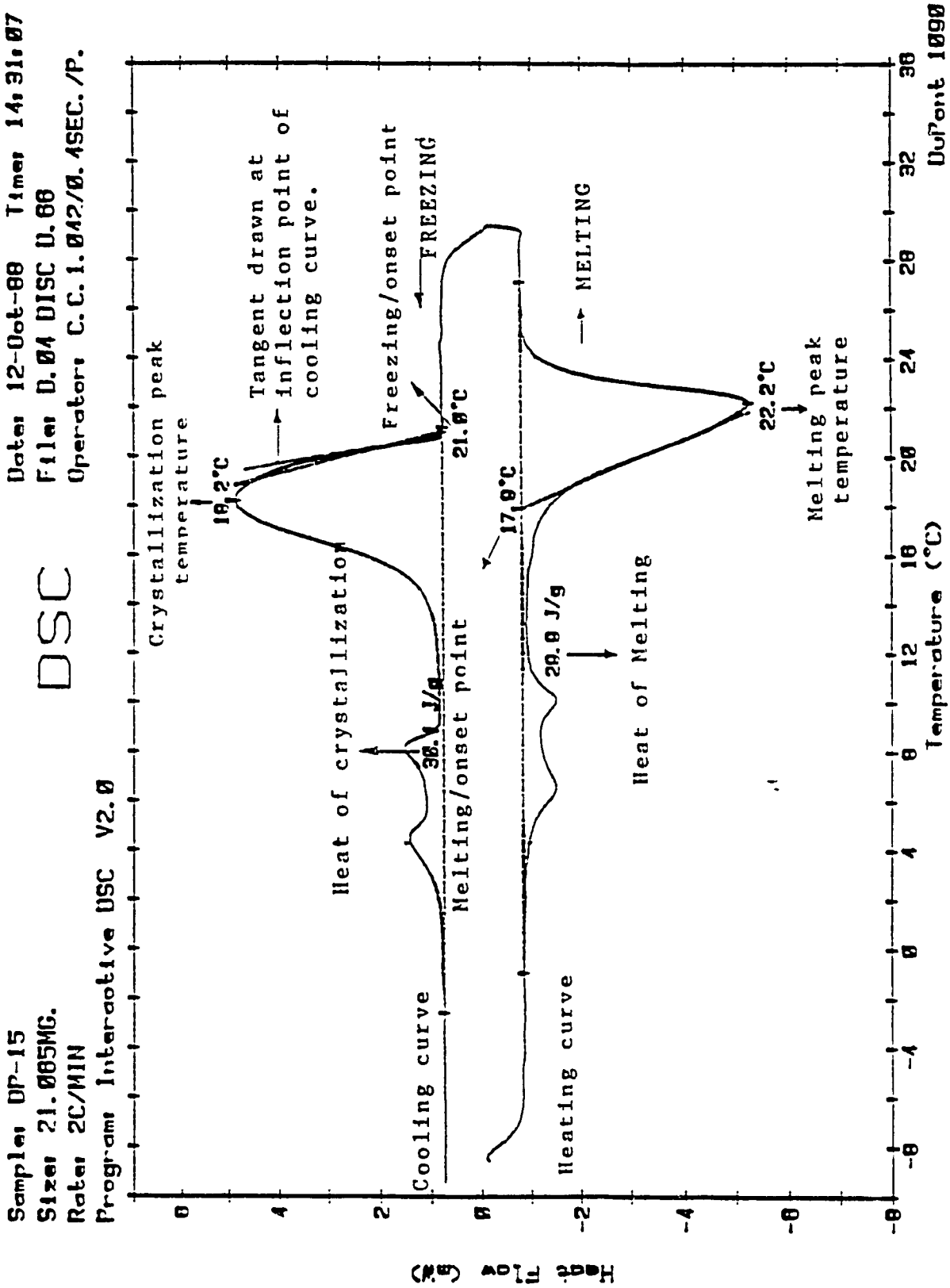


Fig.7.10 Thermogram of heating/cooling cycle of specimen (DP-15).

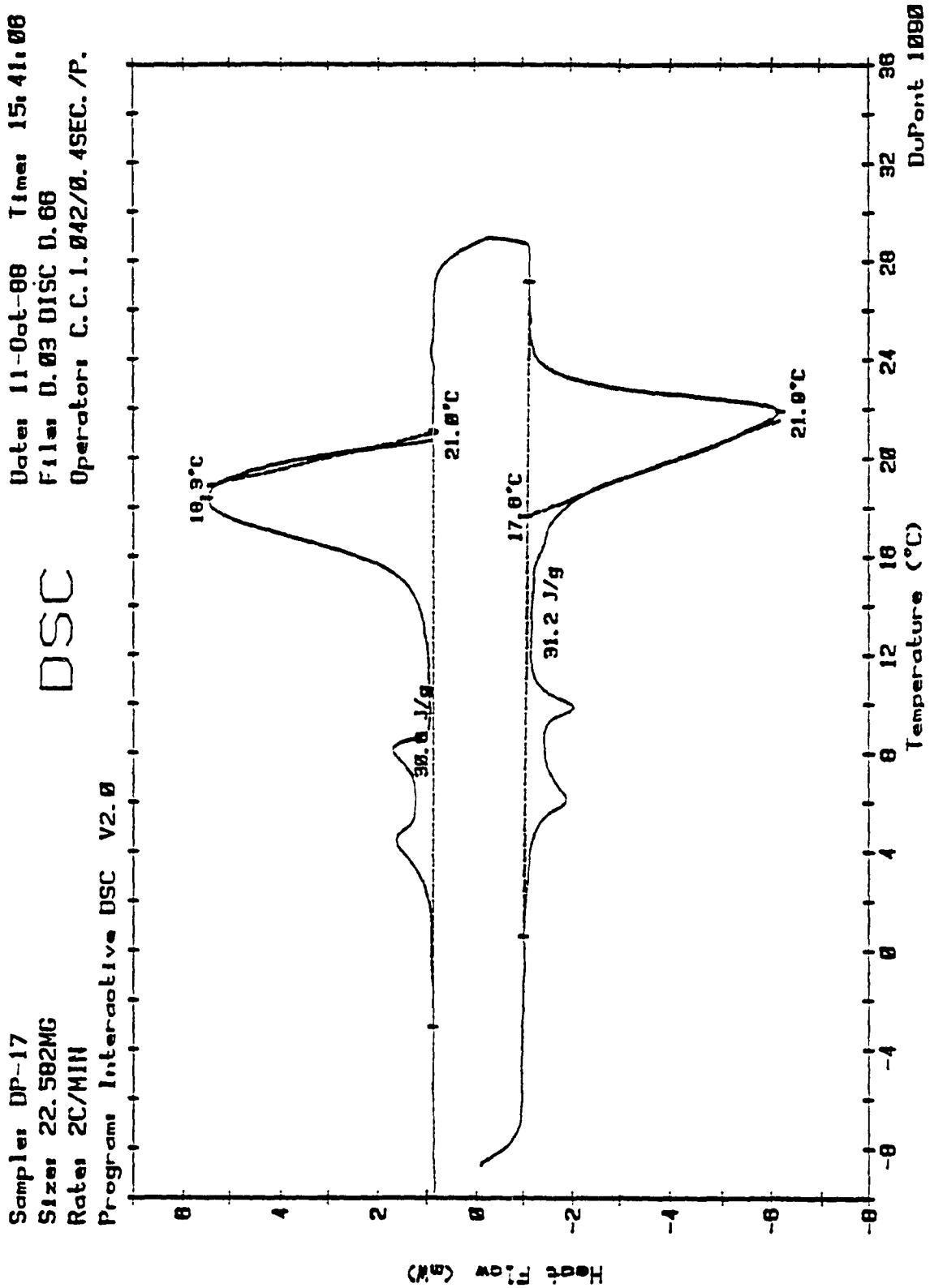


Fig.7.11 Thermogram of heating/cooling cycle of specimen (DP-17).

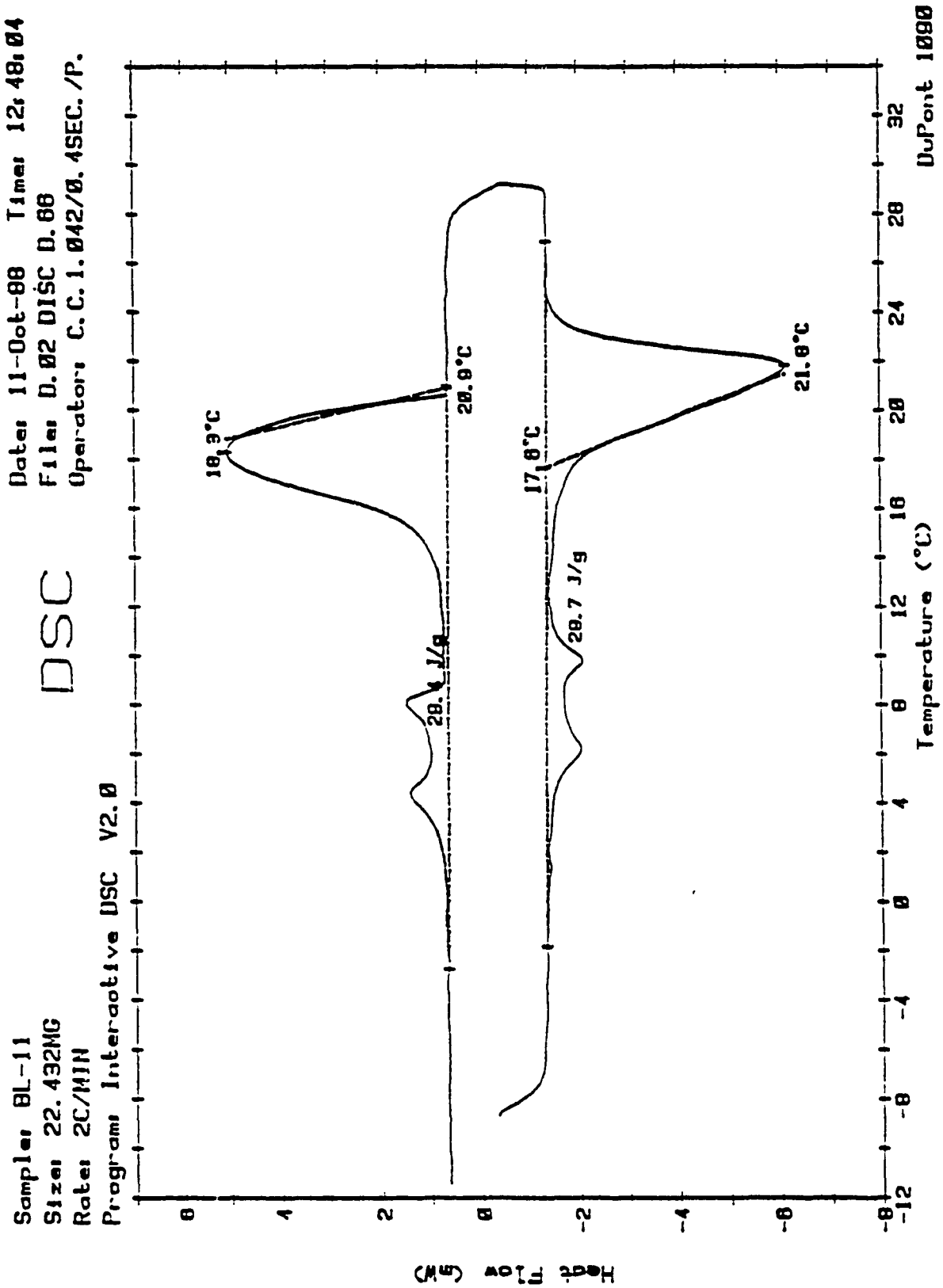


Fig.7.12 Thermogram of heating/cooling cycle of specimen (BL-11).

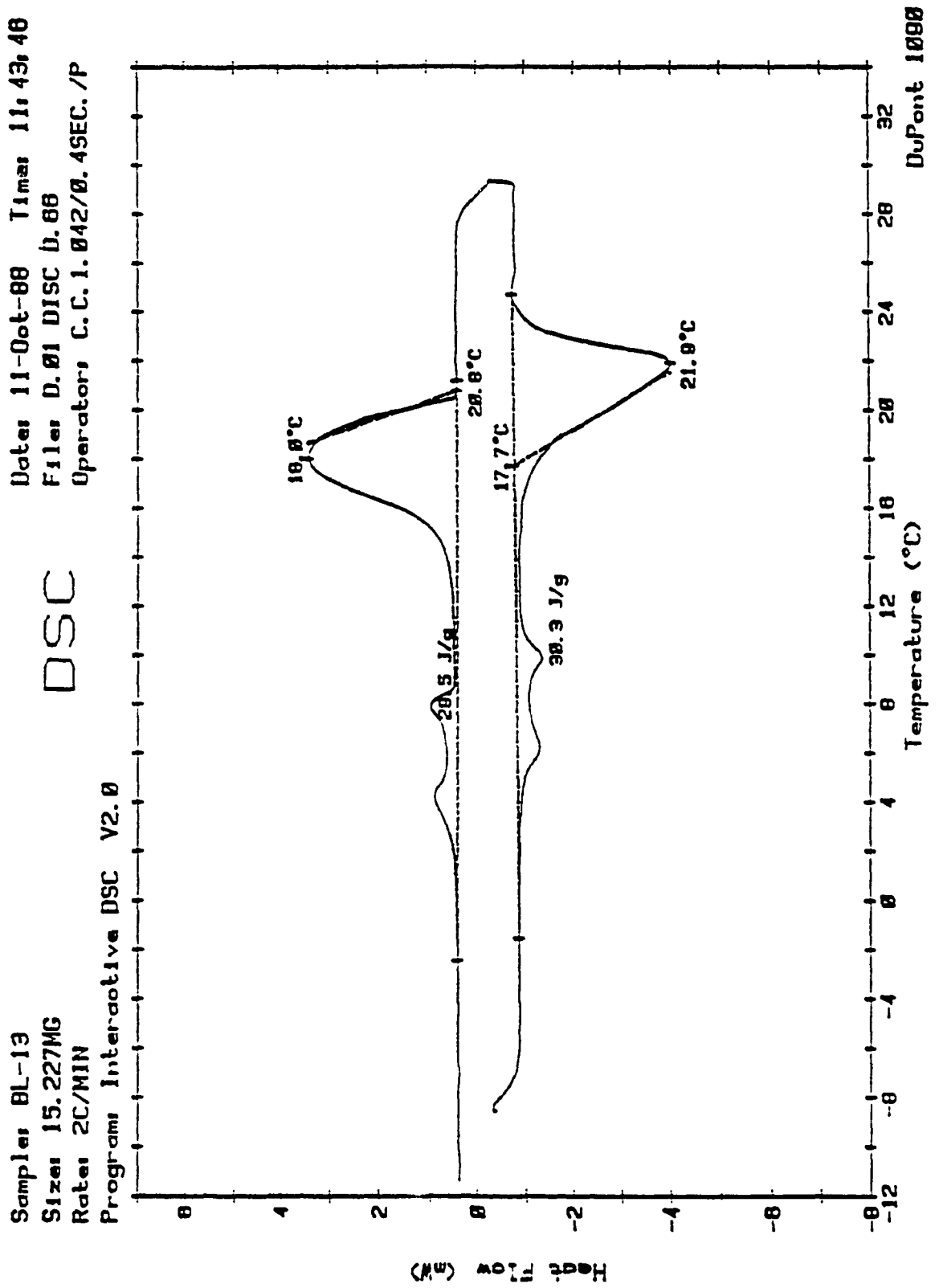


Fig.7.13 Thermogram of heating/cooling cycle of specimen (BL-13).

ii) Freezing point

The initial freezing point f.p. is the temperature of equilibrium between solid and liquid substance at a pressure of one standard atmosphere. The f.p. temperature is generally affected by the sample behaviour since it is not an equilibrium measurement. The freezing point on the cooling curve may shift if initial crystal formation is slow. However, in absence of supercooling, the initial f.p temperature on the cooling curve may be calculated from the thermogram again by plotting the intersection of the base line on the cooling curve with the tangent to the inflection point on the same curve.

In the case where the sample supercools and the temperature rises more than 0.25°C upon crystallization, the initial freezing point is taken as the highest temperature observed. In both melting point and freezing point calculations, a least squares fitting method is used to draw the tangent.

The tangent is drawn by moving along the curve and examining regions of interest until the inflection point is found. The line is then drawn at this inflection point and projected to intersect the base line.

iii) Melting Peak Temperature

This parameter is defined as the temperature which corresponds to the maximum value on the heating curve. Its location depends on the heating and consequently, in order to afford easy comparison between trials, a standard heating rate of 2°C/min was used in all tests.

iv) Crystalization peak temperature

This parameter is defined as the temperature which coressponds to the point located at the peak of the cooling curve, and coressponds to the maximum distance from the base line of the curve.

v) Heat of fusion and crystallization

The heat of fusion and heat of crystallization are calculated from the above mentioned calorimetric measurements and represents the integration of the area below the heating/cooling curve for the heat flow associated with endothermic or exothermic processes based on the following formula:

$$\Delta H = k/m \int_{t_1}^{t_2} \Delta Q/f(T)\ dt \qquad [38]$$

where:

ΔH = heat of transition

m = mass of specimen

ΔQ = heat flux

t = time

f(T) = temperature dependent heat transfer function, &

k = DSC constant (obtained by calibration).

The constant k is non-linerarly dependent on temperature. To ease the problem, k is electronically linearized to a temperature dependent straight line by the thermal analyser system. The heat of transition is calculated and reported in J/g, as can be seen from thermograms.

7.3 IR ANALYSIS

7.3.1 Introduction

A beam of electromagnetic radiation, when passed through a chemical substance, is only partially transmitted. The remainder is reflected or absorbed in varying degrees, depending on the substance, and the frequency of radiation.

The absorbed energy is transferred to the atoms or molecules in the sample substance and, as a result, the particles are promoted from lower to higher energy state. [54]

In absorption or emission spectroscopy several energy components, namely rotational, vibrational, and electronic energy are important. A significant factor in molecular energies is the existence of discrete levels for the rotational, vibrational and electronic components. Under appropriate conditions, the molecules can absorb or lose energy and change from one level to another (but cannot assume an energy level other than those aforementioned). The energy differences between electronic levels are greater than those for vibrational levels which, in turn, are greater than those for rotational levels. Thus, electronic transitions involve high energy radiation of the visible, UV and X-Ray regions, while vibrational transitions involve IR radiation and rotational transitions the relatively low energy at far-infrared radiation [54].

Thus the energies associated with vibrational and rotational changes are comparable to those for I.R. radiation and may be studied in this spectral region. While it is true that each molecular vibration involves the whole molecule, most of the energy of this is involved within a single group. As long as the rest of the molecule is

neglected, a given group can be expected to absorb approximately at the same frequency in all molecules. In effect then, we may consider this group as vibrating independently of the other atoms, a situation which gives rise to characteristic group absorptions. In addition to these characteristic group frequencies, vibrations occur in which the energy is distributed nearly uniformly through the molecule. Therefore, the resulting absorption bands reflect the entire assemblage and provide a unique property of the individual molecules [55]. These are the most useful aspects of IR spectroscopy.

Studies have shown that light atom groups absorb at high frequencies, while heavy atom systems are characterized by low frequencies. Likewise, tightly-bound groups will vibrate at higher frequencies than loosely-bound groups. For example, the C-H stretching (a molecule is constantly vibrating, its bonds stretch, contract and also bend) vibration is observed near 3000 cm^{-1} while the more ponderous C-Cl group is found near 750 cm^{-1}. The C-O stretching vibration absorbs at about 1000 cm^{-1}, while the tighter C=O group absorbs at near 700 cm^{-1} [56].

Atoms and molecules of a substance under examination are in constant motion at all temperatures above absolute zero. When the frequencies of these motions coincide with the frequencies of the applied I.R. radiation, then interaction takes place. As a result, energy is imparted to the molecule with a resulting increase in the amplitude of the vibration. This absorption involves transition from lower to higher energy states.

Here the study of the frequencies of the absorbed molecules provide a means for identifying and analysing chemical substances.

A plot of transmittance vs frequencies yields a minimum at the absorption frequency and therefore, the curve represents the absorption spectrum. Such spectra is dependent upon the physical state and the environment of absorbing species as well as other related factors [54].

Thus the widespread use of IR spectroscopy in analytical chemistry can be attributed to the fact that useful absorption bands are found for all but a very few substances. Both organic and inorganic compounds can be studied whether they exist as volatile gases, vapors, liquids or as solids.

7.3.2 IR spectrometer

The essential features of an IR spectrometer include a source of IR light, a monochromator and a detector. Light from the source is passed through an organic sample, split into individual frequencies in the monochromator and the relative intensities of the individual frequencies are measured in the detector.

i) Source:

The source is always some form of filament which is maintained at red or white heat by an electric current. The common sources are;

- Nernst glower filament; i.e. sintered mixtures of oxides of Zirconium (Zr), Yttrium (Y), Erbium (Er)
- Globar filament, a rod of silicon carbide (SiC)
- Various ceramic materials, which provide IR radiation [56].

ii) Optical path and monochromator:

The beam is guided and focused by mirrors to the point where the sample is to be placed. In order to reduce absorption and

scattering of the radiation, essential windows are made of mineral salt transparent to IR radiation. Most used transparent minerals are Sodium Chloride (NaCl) and Potasium Bromide (KBr). Both prisms and gratings are used for dispersion, but rotatable gratings give much better resolving power.

iii) Detector:

Most modern instruments use thermopile detectors. Outcoming current from the detector is proportional to the intensity of radiation falling on the thermopile.

iv) Sample:

Liquid samples are held between plates of polished mineral salt and are studied in thicknesses of about 0.1-10 mm. Gas samples, at pressures of up to 1 atmosphere or greater, are usually contained in a glass cell 5-10 cm long, which is closed at its ends with salt crystals.

Solid samples are more difficult to examine since the particles reflect and scatter the incident radiation and transmittance is always low. One technique of handling solids is to grind them very finely with KBr, under very high pressure. This material will flow slightly, and the mixture can usually be pressed into a transparent disk. This may then be placed directly in the IR beam in a suitable holder [55].

7.3.3 Operation of IR Spectrometer

Figure 7.14 shows the simplified description of the path of light and operation in an IR spectrometer. Light from the source A is split into two equal beams, one of which (B) passes through the sample, the other (C) behaves as a reference beam. The function of such a

double-beam operation is to measure the difference in intensities between the two beams at each wavelength. The two beams are then reflected to a chopper (D), which consists of a rotating segmented mirror. As the chopper rotates, it causes the sample beam and the reference beam to be reflected alternately to the monochromater grating (E). As the grating slowly rotates, it sends individual frequencies to the detector thermopile (F), which converts the IR thermal energy to electrical energy. The amplifier (G), which receives this signal, is coupled to small servo-motor and from there to a pen recorder (H) [55].

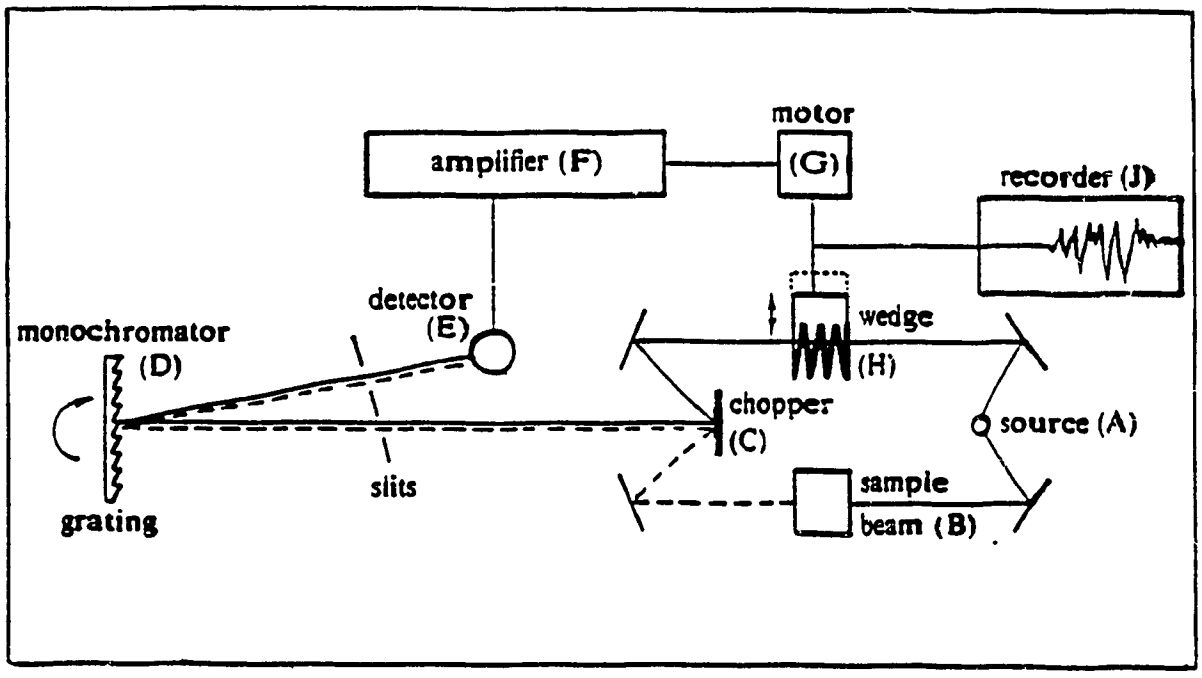


Fig.7.14 Path of light in the IR Spectrometer [57].

7.3.4 Sample preparation and test procedure

Test samples were prepared according to the ANS/ASTM E168-77 standard as follows:

- Solid samples were dried in an oven at 40°C for 24 hours.
- Approximately 0.6 mg of the dried sample and 1 mg of the Potasium Bromide (KBr) were used to prepare a disk.
- The dried sample and KBr were then thoroughly mixed and finely ground in a crucible.
- A transparent disk of this mixture was formed by carefully placing a small amount of the mixture in a special dye. The dye was compressed under a weight of eight tons using a Carver hydrulic press.
- The disks were placed in the sample compartment of the IR spectrometer (Beckman model 4240; Fig.7.15) and tests were performed at 3000 cm^{-1} and a speed of 300 cm^{-1}/min.
- The sample for the pure BS run, was prepared by making two pure disks from KBr and placing a drop of BS in between the disks. The run was performed as before.

7.3.5 Analysis and results

The IR structural analysis was performed to verify if there was any chemical interaction between the other components and impurities in the composite board with incorporated BS, (i.e. mixture of Butyl Stearate with chemical formula of CH_3-$(CH_2)_{16}$-$COOC_4H_9$ and Butyl Palmitate with chemical formula of CH_3-$(CH_2)_{14}$-$COOC_4H_9$

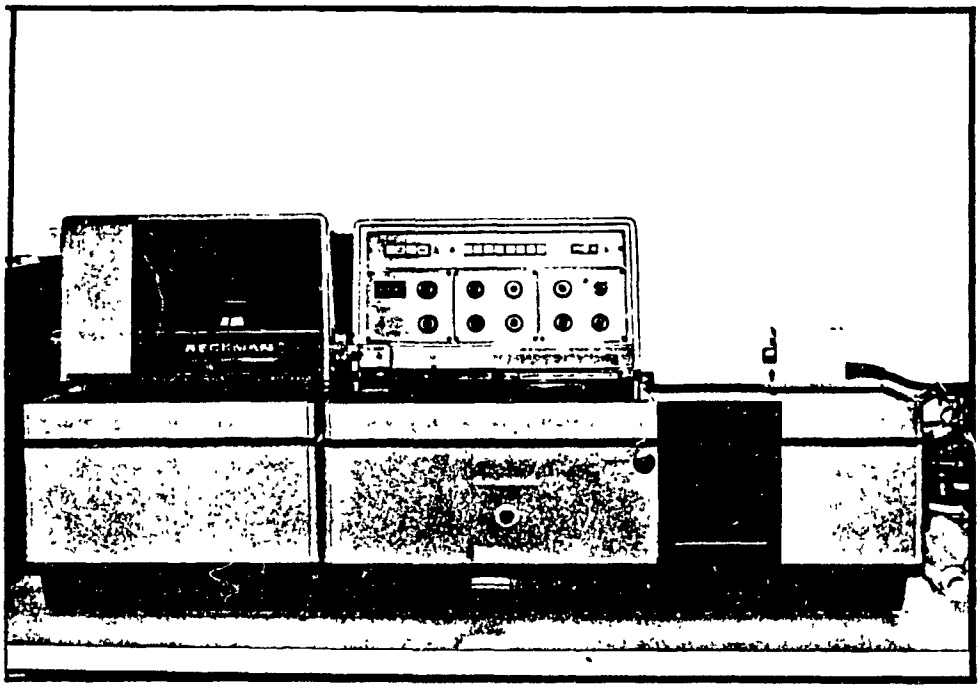


Fig.7.15 IR Spectrometer Beckman Model 4240.

If any chemical reaction takes place, impurities can readily be detected by comparing the sample spectrum with that for a known pure sample and locating the presence of spurious bands.

Hence, if any chemical reaction occurs in the composite, the characteristic IR absorption peaks should at least be partially shifted from the saturated aliphatic esters ($-COOC_4H_9$) having a characteristic peak at 1740 cm^{-1}, to some other peaks.

The results of the IR absorption spectra of samples made of 21-22% (wt) of BS are shown in Figs.7.16 to 7.19. No characteristic signs

indicative of ester hydrolysis, any salts of FAs and or free acids were found. Furthermore, there were no signs of any chemical reactions with other components of the boards or impurities observed.

Specimens were tested after eight months at ambient temperature to determine if any slow reactions took place. Again the result was negative and, therefore, it was concluded that since there were no signs of interaction between the BS and the other components of the storage board, the amount of BS in the board would not likely decrease during the life of the board.

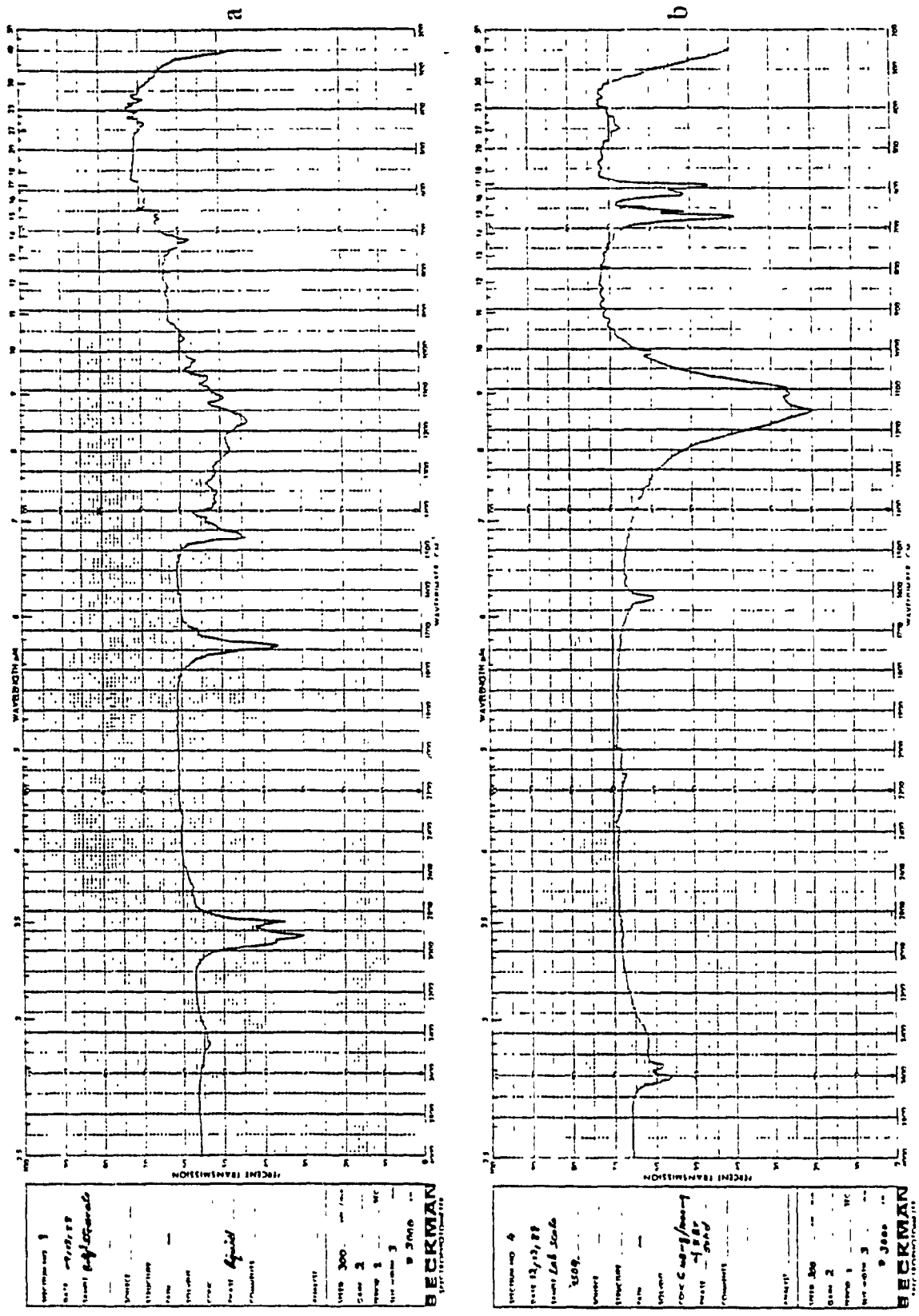


Fig.7.16 IR spectrum of (a); BS, (b); $CaSO_4$ laboratory grade.

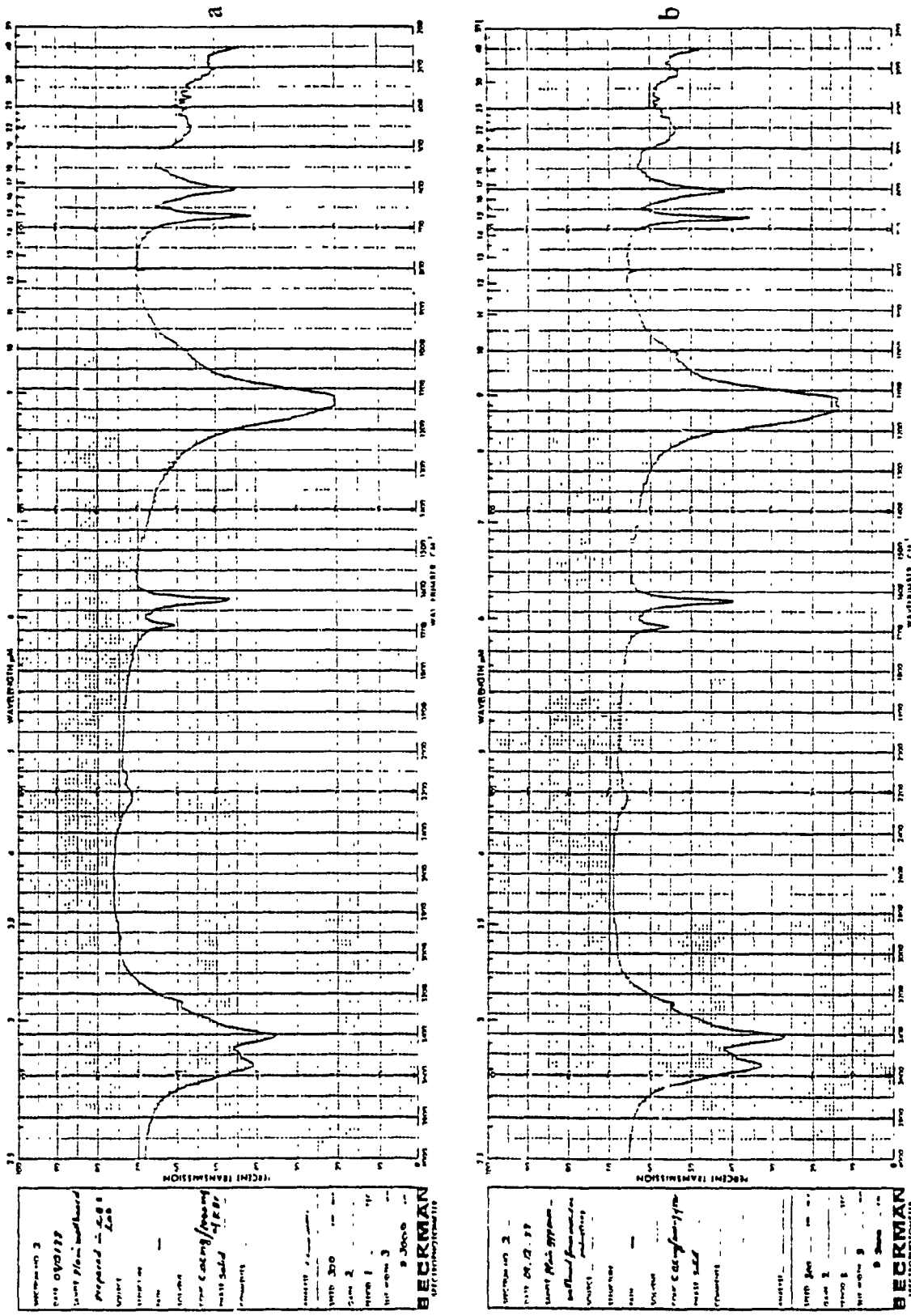


Fig.7.17 IR spectrum of (a); Laboratory prepared plain board, (b);WR made plain board.

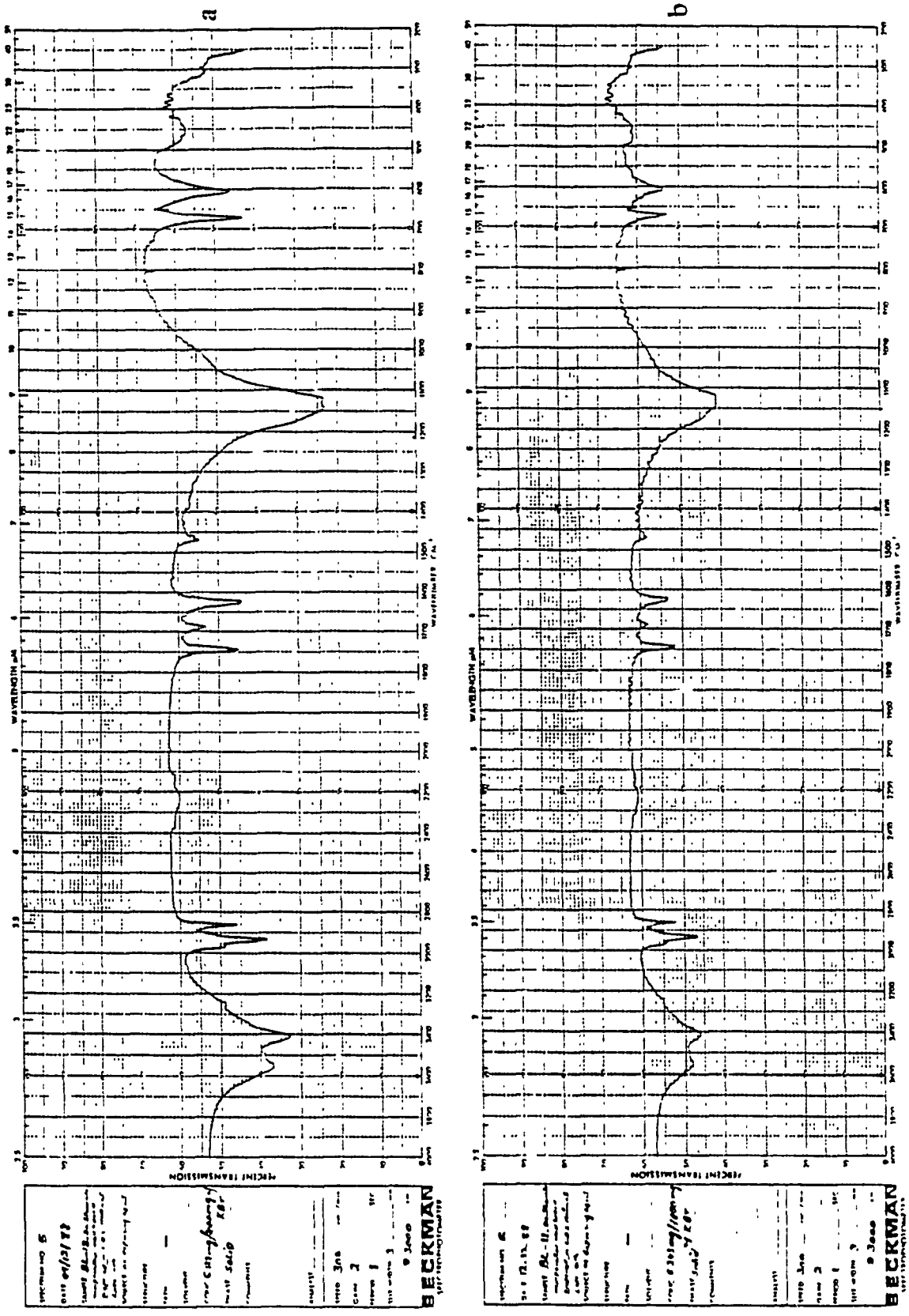


Fig.7.18 IR spectrum of BS incorporated boards. (a); BL-13, (b); BL-11

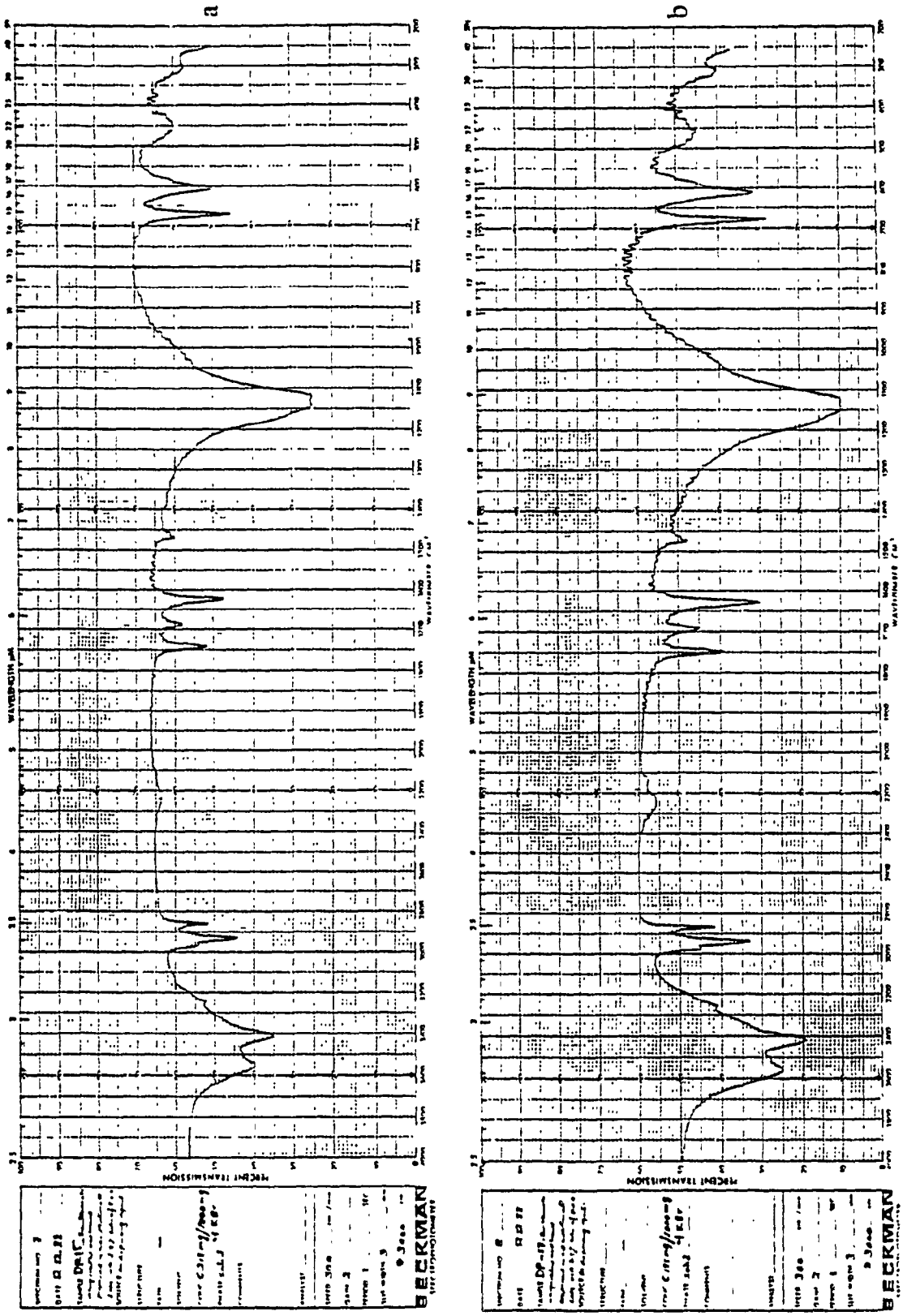


Fig.7.19 IR spectrum of BS incorporated boards, (a); DP-15, (b); DP-17

7.4 THERMAL CONDUCTIVITY ANALYSIS

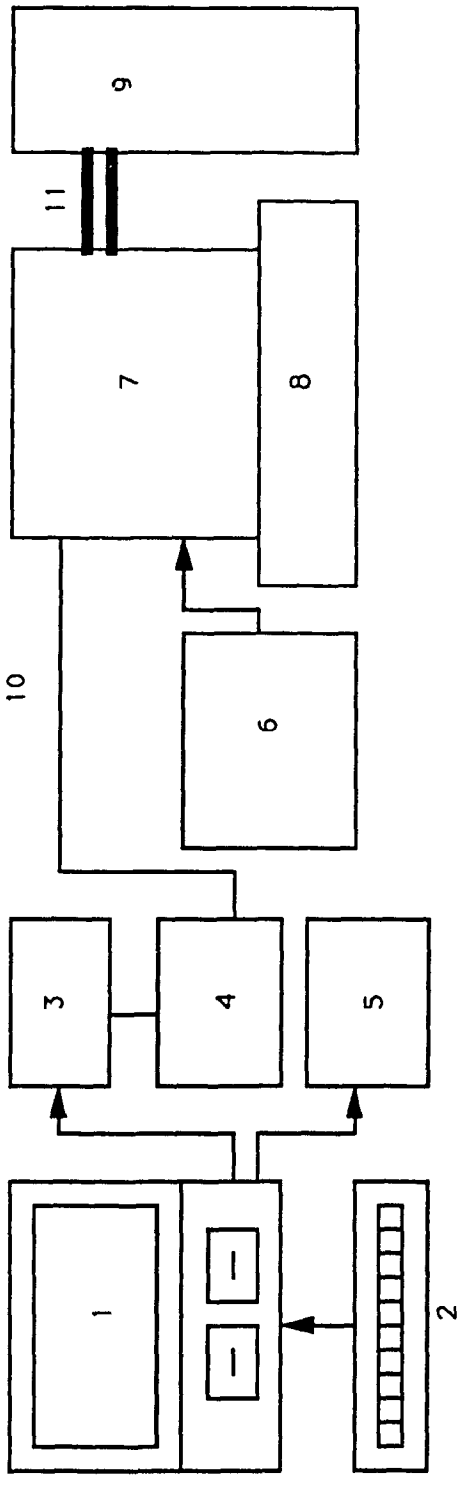
Since the GB will be used as a barrier to heat flow, it is essential to determine the thermal characteristics of the composite board. The thermal conductance and transmittance of built-up sections is evaluated by means of a guarded Hot Box according to ASTM test method C177. It is evident that the thermal transmission properties of a material may

- Vary due to change in components of the material. They may also
- Be affected by moisture or other conditions, such as:
- Testing time
- Specimen thickness.
- Temperature differences across the specimen or
- Fluctuations in testing (sample) temperature.

The facility used to assess the thermal conductance and transmissions of the various samples is shown in Fig.7.20. This facility is comprised of the following equipment:

- Thermal conductivity instrument: Dynatech TCFGM-N4;
- Regulated power supply: Lambda RS;
- Switch controller: Fluke 2205A;
- Multimeter: Fluke 8840A;
- IBM PC with monitor, and GPIB printer, Epson spectrum model LX80.

The tests on various specimens, performed according to ASTM standards C177, were used to compare the effects of incorporated BS



1 IBM PC AND SCREEN
2 IBM KEY BOARD
3 FLUKE MULTIMETER MODEL 8840 A
4 FLUKE SWITCH CONTROLER MODEL 2205 A
5 EPSON SPECTRUM PRINTER MODEL LX80
6 LAMBDA REGULATED POWER SUPPLY MODEL 342 A
7 THERMAL CONDUCTIVITY TEST CABINET
8. DYNATECH GUARDED HEAT PLATE THERMAL CONDUCTIVITY INSTRUMENT MODEL RCS
9 LAUDA COOLANT CIRCULATOR MODEL RCS
10 THERMOCOUPLE SENSOR LEADS
11 COOLANT PIPES

Fig. 7.20 Thermal conductivity measurement apparatus.

on the conductivity of energy storage GB with that of plain gypsum board. Tests were also performed on painted tiles (polymer based paint) and pre-pasted resinous wallpaper specimens to evaluate their effect on the conductivity rate. The specimens which were tested were:

i) Laboratory produced plain tiles comprised of gypsum hemihydrate, pulp (waste newpaper based) and foam.

ii) Tiles with same ingredients as for (i), incorporated with 21-22% (wt) BS, in the presence of a 2.5% (wt) solution of PVA as EA.

iii) Tiles with the same ingredients as (ii) plus two coats of alkyd white No. 35650 and alkyd pearl No. 35610 paint.

iv) Tiles with the same ingredients as (ii) but in presence of 1.5% solution of SSKL as EA.

v) Tiles with same ingredient as (iv) plus a sheet of resinous backed wallpaper.

Results of the tests are presented in Table 7.2 and a comparitive analysis is shown in the bar graph in Figure 7.21. These results demonstrate that the thermal conductivity (K-factor) of the BS incorporated tile is reduced from 0.190 W/m°C to 0.158 W/m°C for the BS/PVA combination in comparison with the plain board and, in turn, there is further decrease from 0.158 W/m°C to 0.149 W/m°C when paint is applied on the DP series samples.

Comparison of the results of the plain board with that of the BS/SSKL combination narrows the gap between their respective conductivities. The BS incorporated product, gives better results (0.175 W/m°C) in this combination. After application of paint and wallpaper on the BS/SSKL combination its K-factor decreases by

0.006W/m°C. The complete computer results of these tests are provided in appendix B. The test results show that storage wallboard is comparable to the standard plain wallboard by virtue of the conduction provided by the gypsum matrix. Since the conductivity of standard wallboard has been found suitable for use with PCMs and since the conductivity of SSKL incorporated wallboard has been found comparable to that of standard wallboard, it is then considered suitable from the point of view of thermal conductivity when impregnated with PCM. If it is desired to increase the rate of heat transmission of the board the addition of aluminum flakes in the mixing stage would be useful for this purpose.

Table 7.2
Thermal conductivity results of plain and
BS incorporated sample tiles

Sample set	PCM (wt) %	Thick-ness (cm)	Weight (g)	Density (g/cm^3)	Mean Temp. (°C)	K factor W/m°C	R value m^2°C/W
PP7	-	1.30	395.6	0.741	22.40	0.190	0.068
PP9	-	1.30	391.2	0.731			
DP10	21.29	1.29	443.2	0.828	22.18	0.158	0.0814
DP11	21.60	1.29	428.4	0.804			
BL5	21.31	1.30	456.0	0.850	22.15	0.175	0.0741
BL18	21.88	1.29	451.5	0.844			
DP3*	21.53	1.32	415.0	0.763	22.28	0.149	0.0896
DP5*	22.13	1.35	427.5	0.755			
BL6**	21.93	1.38	453.5	0.796	22.07	0.169	0.0815
BL9**	21.56	1.38	467.3	0.807			

PP = Plain tile with no PCM

DP = Tiles with PCM and PVA as emulsifying agent

BL = Tiles with PCM and SSKL as emulsifying agent.

* = Tiles with PCM and PVA covered with two coats of alkyd oil base paint.

** = Tiles with PCM and SSKL covered by one coat of water base paint then cross fiber resinous backed wallpaper.

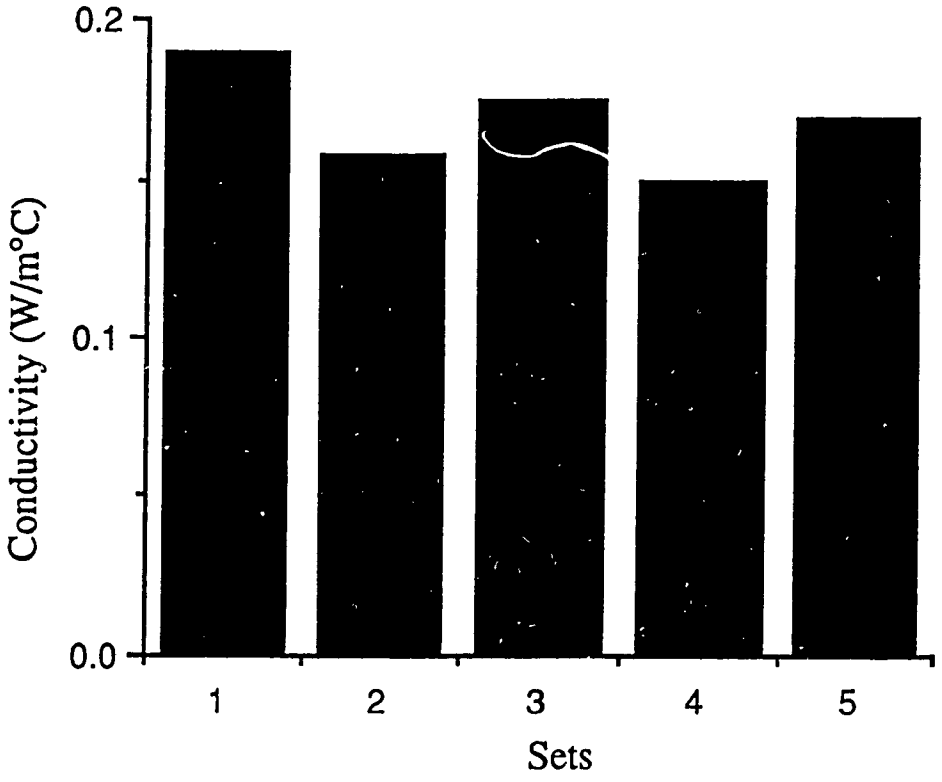


Fig 7.21 Comparison of thermal conductivities of the tested samples.

Legend:

Set 1: Plain tiles with no PCM (PP set).

Set 2: Tiles with PCM and polyvinyl alcohol as EA (DP set)

Set 3: Tiles with PCM and SSKL as EA (BL set).

Set 4: Tiles with PCM and PVA covered with two coats of alkyd oil base paint (* set)

Set 5: Tiles with PCM and SSKL painted with one coat of water base paint, covered with cross fiber resinous backed wallpaper (** set).

CHAPTER 8

DISCUSSION ,CONCLUSIONS AND RECOMMENDATIONS

8.1 DISCUSSION

In addition to development of the incorporation process, various physical and analytical tests have been performed on the sample products to verify, compare, maintain and control the required properties necessary for the production of energy storage gypsumboard. The characteristics of these composite boards, composed of more than ten different chemical components, can be influenced by many factors. Some factors, which are believed to act in a negative manner, are discussed and recomendations are made for the necessary modifications to the product. While these factors are not of great concern in the manufacturing stage, they must be considered from a scientific point of view.

Mechanical properties as residence time in the drying stage are dependent on the production process, size and type of the gypsum particle as well as the moisture content.

The presence of impurities in gypsum reduces the rate of hydration and this, in turn, affects the nature of crystal formation.

Fewer interlocking bonds in the crystal structure are formed as a result and this implies a weaker board and hence inferior mechanical properties.

BPG also contains deleterious contaminants, which, if used, would further decrease the mechanical properties. What is believed to be the cause of loss in mechanical strength are undetectable reactions which may arise from the following situations.

On the one hand, chemical analysis of calcium sulfate shows that oxides such as CaO, MgO, and the salt NaCl are present in natural gypsum. The mineral impurities in water, and the extent of solubility of $CaSO_4 \cdot 1/2H_2O$ in water (8 g/liter), also increases the possibility of free alkali and alkali earth metal cations in the solution. On the other hand, in the binary ester system, there is a very small percentage of free acids as impurites. This situation, coupled with the mechanical stirring of the paste, could produce a saponification (cold process) reaction (even if this reaction is too slow and negligible) which may cause a decrease in mechanical properties.

Absorption of the BS by the cellulosic and other type of fibers present in coverpaper could reduce the friction between these fibers in the paper structure, thereby resulting in a decrease in flexural strength of the board.

The physical presence of the BS during the hydration process could act as an inhibitor to the hydration reaction. This lengthens the setting time and also reduces the crystal formation during the hardening process which, in turn, decreases the mechanical properties of the board.

Compared with the plain wallboard, the incorporation of BS causes a slight increase in the fire spread rate. Traces of smoke were observed during a 30 minute exposure to a 700°C flame. This may be a source of toxic gases which is a potential health problem.

Finally, a slight but unobjectionable smell has been noticed. This is believed to be due to the presence of impurities in the commercial grade BS.

The thermal conductivity test showed a difference of 0.03 W/m°C between thermal storage and plain wallboards. This is because, fatty acids are naturally low in heat conductance. In addition, the use of various types of paints and resinous backed wallpapers also reduced the conductivity since paints and resins are polymers which are not normally good conductors of heat. Although paints and resins are applied in a thin film and as such do not have a significant effect on the overall thermal conductivity of the board, the addition of numerous layers over the years may have a negative effect on the conductivity of the thermal storage wallboard.

8.2 CONCLUSIONS

The present work has demonstrated the feasibility of incorporating BS as PCM into gypsum paste for energy storage wallboard production. This work has been performed successfully through the following stages.

A very straightforward procedure for mixing the oleous compounds with a hydrous medium has been successfully established with help of surface active agents acting as DA, FA and EA at various stages of the production.

The level of BS incorporation in this study was determined to be no greater than 23% (wt) of the board, otherwise exudation of BS may occur, and this was found to be undesirable in respect to the durability and appearance of paint and wallpaper.

The foam density and foam stability were optimized with respect to time and the quantity of foaming agent required to produce optimum foam density and persistance was determined.

The quantity of water and surface active agents was also optimized. These agents, on the one hand, reduce the amount of free water used in process, which in turn reduces the heating load in drying zone and, on the other hand, stabilize emulsion formation.

The optimum concentration of FA solution was found to be 1% (wt). High speed stirring for 1.5 minutes resulted in the most stable foam which had density of 0.116-0.128 g/cc.

For EAs, the use of 2.5% (wt) solution of PVA or 1.5% (wt) solution of SSKL gave the best results. However in this regard the

use of SSKL as EA is more economical in comparison to PVA. Furthermore, the use of SSKL in comparison to the use of other EAs, produced boards with superior mechanical and thermal conductance properties.

The problem of paper/core bonding is believed to be partially due to:

(a)-Laboratory scale problems, because of the rigidity of the coverpaper in relation to its small size.

(b)-The lack of proper pouring facilities.

(c)-Difficulty with metering the components of the composite board on time.

(d)-The presence of BS and an excess of water absorption by coverpaper (low quality paper).

(e)-The long residence time in drying zone.

Mechanical tests to determine the flexural strength of the laboratory produced thermal storage wallboard compare well with values obtained from tests on the standard plain wallboard and fully satisfy strength levels established by testing standards. Further increase in strength could be obtained by the addition of fibers to the paste or by the use of a stronger coverpaper.

Durability of the samples and components of thermal storage board was evaluated by the use of accelerated freeze/thaw tests. The results obtained are completely satisfactory.

Compatibility tests with representative paints (water and oil based) show no sign of any peeling off or blistering of the paint nor evidence of exudation of the BS, even after a year from production.

The water absorption test demonstrate that this product is 50% more water resistant than the plain wallboard and, therefore, is a potential candidate for use in highly humid areas in buildings.

Results of tests to evaluate the setting time of the gypsum paste show a slight increase (by 2-4 minutes) in setting time (i.e. length of hydration reaction) which is considered to be negligible, especially in the case of thermal storage board using SSKL as EA (SSKL gave better results). The setting time can be adjusted by using a small quantity of accelerator during the blending process.

DSC analysis was used to determine the key thermal chracteristics of the BS in thermal storage board, namely the latent heat, melting point, freezing point and transition temperature (the range of temperatures at which heat is released or absorbed). DSC analysis was also used to evaluate the stability of BS subjected to freeze/thaw cycling, no evidence of any change in its thermal characteristics could be detected.

An IR analysis was performed to determine the presence and extent of interaction between BS and the gypsum matrix. A comparison was made between the IR spectum of calcium sulfate (99% pure laboratory grade), butyl stearate (98%), plain gypsum board (Lab. and Industry made) and BS incorporated board using PVA and SSKL as EA. An investigation of spectra shows that no band shifts occured, such shifts would indicate a possible interaction between components after 8 months from the casting date. This indicate that threre is no change of chemical functional groups which, in turn, implies no chemical reactions taking place. These results further confirm that the quantity of BS present in the storage

wallboard will not diminish due to chemical interaction with the matrix during its expected service life.

Tests undertaken to evaluate the thermal conductivity of the storage wallboard revealed that less heat is transmitted through a sample of thermal storage wallboard than that of a plain wallboard and that the wallboard produced with SSKL as EA had the highest thermal conductivity (0.175W/m°C). These tests further demonstrated that the thermal transmission characteristics of thermal storage wallboard are comparable to that of plain wallboard and, by the virtue of the conduction provided by the gypsum matrix it was found to be suitable for the required rate of thermal exchange [38].

The board densities of energy storage products showed a slight increase in weight (2-3 kg) for 244 x 122 x 1.27 cm panel in comparison to plain products. However, densities from various companies show a considerable difference due to the different grades of calcium sulfates used to produce the boards. Furthermore, board weights are found to vary by at least this amount due to dimensional tolerances. In any case, density can be adjusted to produce a lighter board by using a stucco with a greater particle size using a higher density foam.

Impregnation of storage GB using the immersion technique is a straightforward process and production facilities could easily be implemented after the drying stage or prior to the distribution stage at the end of an existing manufacturing plant. Other than the cost of purchasing and installing the process equipment, in our opinion the implementation of this technique would also imply an increase in:

- Energy requiremet; for the equipment
- Production time; brought about by the extra time used to absorb the required amount of BS into the board.
- Labour cost; due to handling and maintenance requirements.

The direct incorporation method, in comparison to the immersion technique, has more of these requirements and can be considered to be a more efficient and economical method of producing energy storage gypsum wallboard.

8.3 RECOMMENDATIONS

The use of the direct incorporation method for the production of energy storage wallboard is more efficient and more economical than the other methods. Furthermore, based on the series of tests performed in this study, it compares well with standard wallboard in terms of strength and durability and it has high potential for energy conservation. In order that this product may meet the necessary requirements and gain market acceptance for use in the building industry, it is recommended that the following studies be carried out to refine the final product.

i)-Fire tests: Further tests should be conducted to evaluate the fire resistance of the storage board and to determine the quantity of fume and smoke emitted from a standard sample as well as to evaluate the probable hazards that may arise from fumes. Further work must be conducted to:

- Select and evaluate potential fire retardants.
- Conduct full scale fire tests.

- analyze gases and fumes which are emitted.

ii)-IR analysis of the product samples clearly indicates the absence of any chemical reactions with BS in the short term. However, tests are required to evaluate the long term durability of the product, and IR analyses should be repeated on 2-3 year old products.

iii)-Microscopical analysis should be used to illustrate the nature of the interaction between BS and the gypsum matrix. Inferences may be made as to the effects of BS on the mechanical properties, durability and composite behavoir of the storage board.

iv)-Regarding painting and wallcovering: more comprehensive tests with respect to different types of paint and wallcovering (which potentially may be used on conventional wallboard) should be carried out on the storage wallboard and a classification of their effects on the product be made.

v)-Work must be carried out in respect to modification of impregnation technique to suit the requirements of existing wallborad production facilities. This work would be carried out largely at the wallboard plant. Among these impregnations, e.g. are:
(a)- The effects of passing an impregnated wallboard through a drying kiln.
(b)- Stabilizing the setting time to suit the speed in production line.
(c)- The investigation of the immersion technique as a practical manufacturing alternative.

vi)-Undertake full scale tests to evaluate the performance of the system in a representative occupied building. The tests would be conducted using the refined product, absent of any undesireable

properties, in controlled conditions to evaluate the comfort level of human occupants and prove that the product is capable of acquiring market acceptance.

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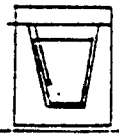
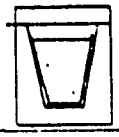
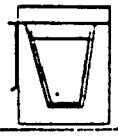
Appendices

Appendix A

The complete results of tests (ASTM C 472) on different specimens of plain as well as BS incorporated gypsum pastes to determine setting time of the thermal storage boards. The position of the thermocouples as well as relative time/temperature graphs are also demonstrated for clarity.

Average of three runs with plain specimens

Time (min)	Temperature (°C)				
					
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2.00	28	28	28	28	26
3.00	29	28	28	28	26
4.00	30	29	29	29	26
5.00	31	30	30	30	26
6.00	33	31	31	30	26
7.00	35	32	33	32	26
8.00	37	34	35	34	26
9.00	40	36	37	35	26
10.00	43	39	40	38	26
11.00	46	41	42	40	26
12.00	48	43	44	42	26
13.00	50	44	46	44	26
14.00	51	46	48	45	26
15.00	52	48	48	46	26
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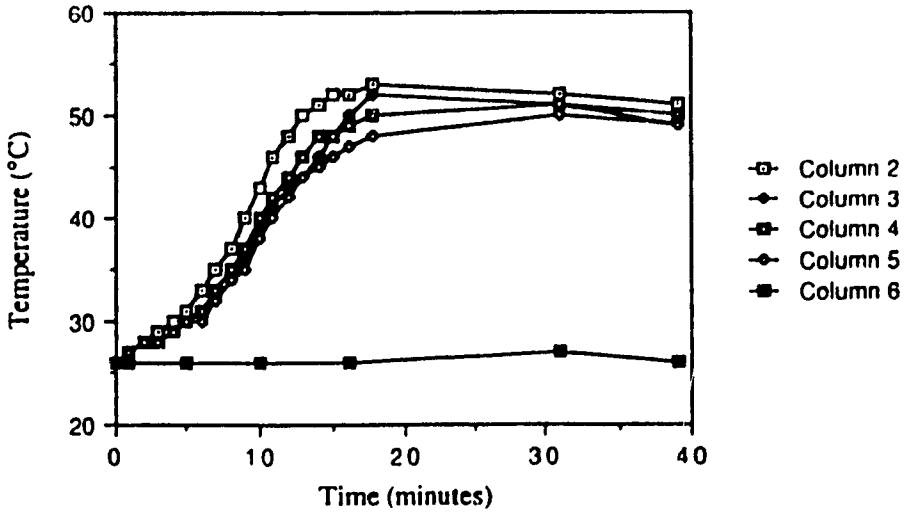
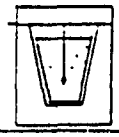
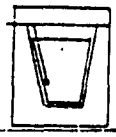
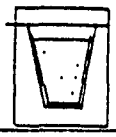
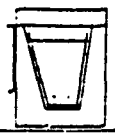
31.00	52	51	51	50	27
39.00	51	49	50	49	26



Average of three runs with WFA specimens

Time (min)	Temperature (°C)				
					
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2.00	28	28	27	27	26
3.00	28	28	28	27	26
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5.00	32	31	30	29	26
6.00	34	33	31	30	26
7.00	36	35	33	32	26
8.00	39	37	36	34	26
9.00	41	39	38	35	26
10.00	43	41	40	37	27
11.00	45	42	42	39	27
12.00	46	43	44	40	27
13.00	46	44	45	41	27
14.00	47	45	46	42	27
15.00	47	45	46	42	27
16.00	48	46	47	43	27
17.00	48	46	47	43	27
18.00	48	46	47	44	27
19.00	48	47	47	44	27
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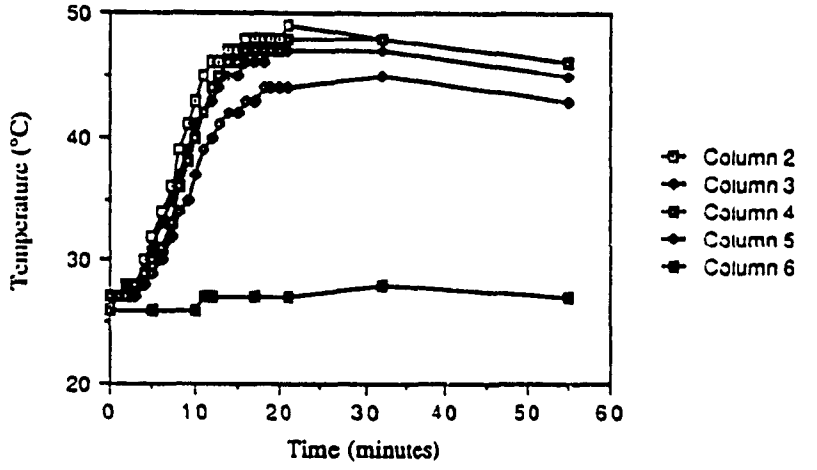
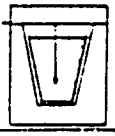
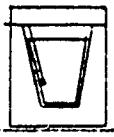
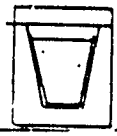
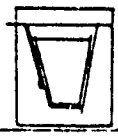
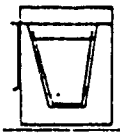
32.00	48	47	48	45	28
55.00	46	45	46	43	27



Average of three runs with PVA specimens

Time (min)	Temperature (°C)				
					
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1.00	27	27	26	26	26
2.00	27	27	27	26	26
3.00	28	27	27	27	26
4.00	30	29	28	28	26
5.00	31	30	30	39	26
6.00	34	32	31	30	26
7.00	36	34	33	32	26
8.00	38	36	35	34	26
9.00	41	38	37	36	26
10.00	43	40	39	37	26
11.00	44	41	40	39	26
12.00	45	42	41	40	26
13.00	46	43	42	41	26
14.00	47	44	43	42	26
15.00	47	44	44	43	27
16.00	47	44	44	43	27
17.00	48	45	44	44	27
18.00	48	46	45	44	27
19.00	48	46	46	44	27
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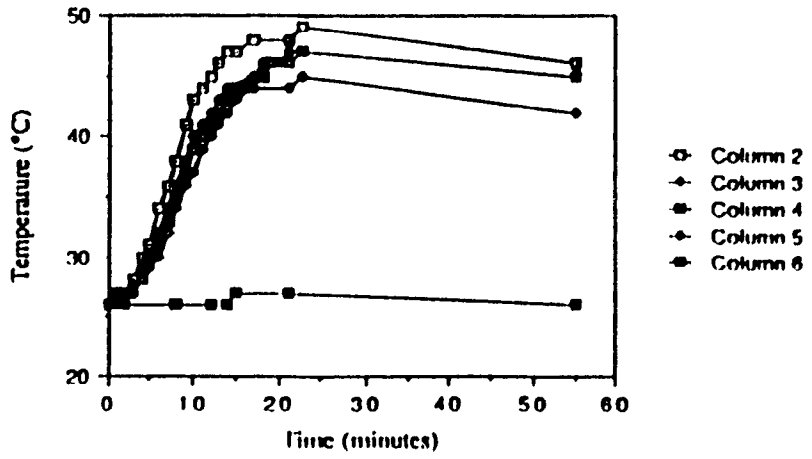
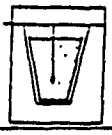
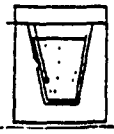
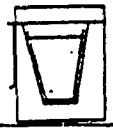
22.66	49	47	47	45	27
55.00	46	45	45	42	26



Average of three runs with SSKL specimens

Time (min)	Temperature (°C)				
					
0.00	26	27	27	27	26
1.00	27	27	27	27	26
2.00	28	28	27	27	26
3.00	28	28	28	28	26
4.00	30	29	29	29	26
5.00	32	31	30	31	26
6.00	34	33	32	32	26
7.00	36	34	33	33	26
8.00	38	35	34	34	26
9.00	39	37	35	34	26
10.00	43	40	39	38	26
11.00	46	43	41	40	26
12.00	47	44	42	42	27
13.00	48	45	43	43	27
14.00	48	46	44	44	27
15.00	49	46	45	44	27
16.00	49	47	45	45	27
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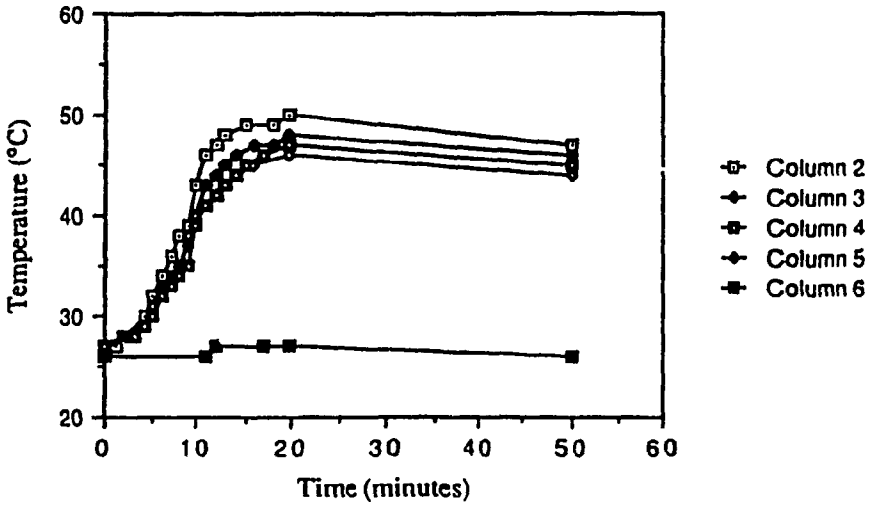
18.00	49	47	46	45	27
19.66	50	48	47	46	27
50.00	47	46	45	44	26



Appendix B

The complete results of the thermal conductivity tests (ASTM C-177) on different specimens of the plain as well as thermal storage gypsum boards for evaluation of the k-factors of the samples under investigation.

Test Data on Thermal Conductivity Measurement (ASTM C177

Report No. CBS-090-GHA.001 Date: 12/14/88

Customer: Mr. Ghanbari
Cntre for Building Studies
Concordia University

Material: gypsum boards
plane reference boards
Sample id: specimens PP7 bottom and PP9 top
Original sample thickness
Thickness of top specimen .013 m
Thickness of bottom specimen .013 m
Weight of sample 395.6 kg

Room conditions: Temperature: 22 °C
Humidity: 50 % R.H.
Coolant temperature: 22 °C

Elapsed time 0 min.

TC 1	19.61033	0.00000	CH19 Volts	2.09700
TC 2	19.61033	0.00000	CH20 Amps	0.29290
TC 3	19.58521	0.00000	K-Factor =	15.21160
TC 4	19.56009	0.00000	R-Value =	0.00085
TC 5	19.61033	0.00000	DT top =	0.10050
TC 6	19.56009	0.00000	DT bot =	-0.03768
TC 7	19.48471	0.00000	Mean temp. =	19.56322
TC 8	19.48471	0.00000		

Elapsed time 15 min.

TC 1	19.92909	0.00000	CH19 Volts	2.09700
TC 2	19.95419	0.00000	CH20 Amps	0.29310
TC 3	20.65620	0.00000	K-Factor =	0.64735
TC 4	20.68124	0.00000	R-Value =	0.02008
TC 5	20.68124	0.00000	DT top =	0.75007
TC 6	20.63115	0.00000	DT bot =	0.72707
TC 7	19.88315	0.00000	Mean temp. =	20.29317
TC 8	19.92909	0.00000		

Elapsed time 30 min.

TC 1	20.16470	0.00000	CH19 Volts	2.09800
TC 2	20.19404	0.00000	CH20 Amps	0.29320
TC 3	21.54153	0.00000	K-Factor =	0.35428
TC 4	21.56651	0.00000	R-Value =	0.03669
TC 5	21.59149	0.00000	DT top =	1.32662
TC 6	21.51654	0.00000	DT bot =	1.37465
TC 7	20.21486	0.00000	Mean temp. =	20.87870
TC 8	20.23994	0.00000		

Elapsed time 45 min.

TC 1	20.37598	0.00000	CH19 Volts	2.09700
TC 2	20.40105	0.00000	CH20 Amps	0.29320
TC 3	22.20542	0.00000	K-Factor =	0.26928
TC 4	22.22613	0.00000	R-Value =	0.04828
TC 5	22.25529	0.00000	DT top =	1.72493
TC 6	22.15554	0.00000	DT bot =	1.82726
TC 7	20.45543	0.00000	Mean temp. =	21.32255
TC 8	20.50555	0.00000		

Elapsed time 60 min.

TC 1	20.55027	0.00000	CH19 Volts	2.09800
TC 2	20.57532	0.00000	CH20 Amps	0.29310
TC 3	22.74408	0.00000	K-Factor =	0.22413
TC 4	22.77321	0.00000	R-Value =	0.05800
TC 5	22.79810	0.00000	DT top =	2.07264
TC 6	22.74831	0.00000	DT bot =	2.19585
TC 7	20.67552	0.00000	Mean temp. =	21.69880
TC 8	20.72501	0.00000		

Elapsed time 75 min.

TC 1	20.85745	0.00000	CH19 Volts	2.09700
TC 2	20.85320	0.00000	CH20 Amps	0.29320
TC 3	23.17850	0.00000	K-Factor =	0.19850
TC 4	23.15343	0.00000	R-Value =	0.06549
TC 5	23.17850	0.00000	DT top =	2.30826
TC 6	23.10368	0.00000	DT bot =	2.51055
TC 7	20.80770	0.00000	Mean temp. =	21.94873
TC 8	20.85777	0.00000		

Elapsed time 90 min.

TC 1	20.77361	0.00000	CH19 Volts	2.09800
TC 2	20.77786	0.00000	CH20 Amps	0.29320
TC 3	23.47188	0.00000	K-Factor =	0.18536
TC 4	23.47188	0.00000	R-Value =	0.07013
TC 5	23.49251	0.00000	DT top =	2.46672
TC 6	23.42217	0.00000	DT bot =	2.69614
TC 7	20.95309	0.00000	Mean temp. =	22.17389
TC 8	21.02816	0.00000		

Elapsed time 105 min.

TC 1	20.81376	0.00000	CH19 Volts	2.09700
TC 2	20.81376	0.00000	CH20 Amps	0.29320
TC 3	23.68563	0.00000	K-Factor =	0.17432
TC 4	23.68141	0.00000	R-Value =	0.07458
TC 5	23.68563	0.00000	DT top =	2.61753
TC 6	23.63595	0.00000	DT bot =	2.86976
TC 7	21.01824	0.00000	Mean temp. =	22.30033
TC 8	21.06828	0.00000		

Elapsed time 120 min.

TC 1	20.87096	0.00000	CH19 Volts	2.09700
TC 2	20.89599	0.00000	CH20 Amps	0.29320
TC 3	23.81264	0.00000	K-Factor =	0.16912
TC 4	23.84169	0.00000	R-Value =	0.07687
TC 5	23.83747	0.00000	DT top =	2.71231
TC 6	23.76298	0.00000	DT bot =	2.94369
TC 7	21.05039	0.00000	Mean temp. =	22.39969
TC 8	21.12544	0.00000		

Program halted after specified 2 hrs. monitoring operation

Average values at end of test:
K-factor = 0.19029 W/m°C
= 1.31960 Btu-in/ft²,hr,°F

R-value based on thermocouple separation:
R-value = 0.06832 m²°C/W
= 0.38794 ft²,hr,°F/Btu

Test Data on Thermal Conductivity Measurement (ASTM C177

Report No. CBS-090-GHA.002 Date: 12-14-88

Customer: Mr. Ghanbari
Centre for Building Studies
Concordia University

Material: with 21.29% PCM
Sample id: specimens DP10 bottom and DP11 top
gypsum boards with P.V.A.
Original sample thickness
Thickness of top specimen .0129 m
Thickness of bottom specimen .0129 m
Weight of sample 0 kg

Room conditions: Temperature: 22 °C
Humidity: 52 % R.H.
Coolant temperature: 22 °C

Elapsed time 0 min.

TC 1	20.55307	0.00000	CH19 Volts	2.09800
TC 2	20.57812	0.00000	CH20 Amps	0.29320
TC 3	22.97513	0.00000	K-Factor =	0.21981
TC 4	22.95024	0.00000	R-Value =	0.05869
TC 5	23.00425	0.00000	DT top =	1.92330
TC 6	23.00425	0.00000	DT bot =	2.39709
TC 7	21.05381	0.00000	Mean temp. =	21.90337
TC 8	21.10809	0.00000		

Elapsed time 15 min.

TC 1	20.73515	0.00000	CH19 Volts	2.09800
TC 2	20.73515	0.00000	CH20 Amps	0.29330
TC 3	23.20573	0.00000	K-Factor =	0.19839
TC 4	23.20573	0.00000	R-Value =	0.06502
TC 5	23.28033	0.00000	DT top =	2.31791
TC 6	23.23059	0.00000	DT bot =	2.47058
TC 7	20.91040	0.00000	Mean temp. =	22.03347
TC 8	20.96470	0.00000		

Elapsed time 30 min.

TC 1	20.80751	0.00000	CH19 Volts	2.09700
TC 2	20.83255	0.00000	CH20 Amps	0.29310
TC 3	23.40190	0.00000	K-Factor =	0.18732
TC 4	23.40190	0.00000	R-Value =	0.06887
TC 5	23.42676	0.00000	DT top =	2.48359
TC 6	23.45583	0.00000	DT bot =	2.58187
TC 7	20.95770	0.00000	Mean temp. =	22.15523
TC 8	20.95770	0.00000		

Elapsed time 45 min.

TC 1	20.80528	0.00000	CH19 Volts	2.09800
TC 2	20.8[illegible]032	0.00000	CH20 Amps	0.29330
TC 3	23.51972	0.00000	K-Factor =	0.17975
TC 4	23.49910	0.00000	R-Value =	0.07177
TC 5	23.54879	0.00000	DT top =	2.59332
TC 6	23.54879	0.00000	DT bot =	2.69161
TC 7	20.9[illegible]045	0.00000	Mean temp. =	22.20787
TC 8	20.98050	0.00000		

Elapsed time 60 min.

TC 1	20.82760	0.00000	CH19 Volts	2.09900
TC 2	20.82760	0.00000	CH20 Amps	0.29330
TC 3	23.64546	0.00000	K-Factor =	0.17326
TC 4	23.62062	0.00000	R-Value =	0.07445
TC 5	23.67030	0.00000	DT top =	2.68001
TC 6	23.67030	0.00000	DT bot =	2.80544
TC 7	20.97777	0.00000	Mean temp. =	22.28031
TC 8	21.00280	0.00000		

Elapsed time 75 min.

TC 1	20.79133	0.00000	CH19 Volts	2.09800
TC 2	20.81636	0.00000	CH20 Amps	0.29320
TC 3	23.68399	0.00000	K-Factor =	0.16928
TC 4	23.68399	0.00000	R-Value =	0.07621
TC 5	23.70883	0.00000	DT top =	2.72977
TC 6	23.70883	0.00000	DT bot =	2.88015
TC 7	20.96655	0.00000	Mean temp. =	22.29393
TC 8	20.99157	0.00000		

Elapsed time 90 min.

TC 1	20.78384	0.00000	CH19 Volts	2.09800
TC 2	20.80462	0.00000	CH20 Amps	0.29340
TC 3	23.72624	0.00000	K-Factor =	0.16596
TC 4	23.70141	0.00000	R-Value =	0.07773
TC 5	23.80074	0.00000	DT top =	2.80645
TC 6	23.75108	0.00000	DT bot =	2.91959
TC 7	20.97984	0.00000	Mean temp. =	22.31336
TC 8	20.95907	0.00000		

Elapsed time 105 min.

TC 1	20.77428	0.00000	CH19 Volts	2.09800
TC 2	20.78436	0.00000	CH20 Amps	0.29320
TC 3	23.72676	0.00000	K-Factor =	0.16312
TC 4	23.72253	0.00000	R-Value =	0.07909
TC 5	23.77642	0.00000	DT top =	2.85659
TC 6	23.75581	0.00000	DT bot =	2.96532
TC 7	20.90953	0.00000	Mean temp. =	22.28990
TC 8	20.90953	0.00000		

Elapsed time 120 min.

TC 1	20.73107	0.00000	CH19 Volts	2.09700
TC 2	20.75611	0.00000	CH20 Amps	0.29330
TC 3	23.72356	0.00000	K-Factor =	0.16205
TC 4	23.72356	0.00000	R-Value =	0.07960
TC 5	23.77323	0.00000	DT top =	2.87933
TC 6	23.79806	0.00000	DT bot =	2.97997
TC 7	20.90632	0.00000	Mean temp. =	22.28978
TC 8	20.90632	0.00000		

Elapsed time 135 min.

TC 1	20.69803	0.00000	CH19 Volts	2.09800
TC 2	20.72733	0.00000	CH20 Amps	0.29320
TC 3	23.69080	0.00000	K-Factor =	0.16177
TC 4	23.69080	0.00000	R-Value =	0.07974
TC 5	23.76530	0.00000	DT top =	2.89211
TC 6	23.74047	0.00000	DT bot =	2.97812
TC 7	20.84826	0.00000	Mean temp. =	22.25428
TC 8	20.87329	0.00000		

Elapsed time 150 min.

TC 1	20.68629	0.00000	CH19 Volts	2.09800
TC 2	20.70707	0.00000	CH20 Amps	0.29330
TC 3	23.72883	0.00000	K-Factor =	0.15935
TC 4	23.72883	0.00000	R-Value =	0.08095
TC 5	23.80332	0.00000	DT top =	2.92925
TC 6	23.80332	0.00000	DT bot =	3.03215
TC 7	20.88659	0.00000	Mean temp. =	22.27573
TC 8	20.86156	0.00000		

Elapsed time 165 min.

TC 1	20.63620	0.00000	CH19 Volts	2.09800
TC 2	20.66124	0.00000	CH20 Amps	0.29330
TC 3	23.67916	0.00000	K-Factor =	0.15972
TC 4	23.67916	0.00000	R-Value =	0.08077
TC 5	23.72883	0.00000	DT top =	2.91732
TC 6	23.73306	0.00000	DT bot =	3.03044
TC 7	20.81149	0.00000	Mean temp. =	22.21811
TC 8	20.81575	0.00000		

Elapsed time 180 min.

TC 1	20.62873	0.00000	CH19 Volts	2.09900
TC 2	20.65377	0.00000	CH20 Amps	0.29330
TC 3	23.69659	0.00000	K-Factor =	0.15814
TC 4	23.69659	0.00000	R-Value =	0.08157
TC 5	23.77109	0.00000	DT top =	2.95455
TC 6	23.77109	0.00000	DT bot =	3.05534
TC 7	20.82906	0.00000	Mean temp. =	22.23137
TC 8	20.80402	0.00000		

Elapsed time 195 min.

TC 1	20.57862	0.00000	CH19 Volts	2.09800
TC 2	20.60368	0.00000	CH20 Amps	0.29330
TC 3	23.67175	0.00000	K-Factor =	0.15729
TC 4	23.67175	0.00000	R-Value =	0.08202
TC 5	23.72565	0.00000	DT top =	2.95918
TC 6	23.72565	0.00000	DT bot =	3.08060
TC 7	20.77899	0.00000	Mean temp. =	22.18875
TC 8	20.75395	0.00000		

Elapsed time 210 min.

TC 1	20.55836	0.00000	CH19 Volts	2.09500
TC 2	20.60847	0.00000	CH20 Amps	0.29340
TC 3	23.67650	0.00000	K-Factor =	0.15722
TC 4	23.62682	0.00000	R-Value =	0.08205
TC 5	23.72617	0.00000	DT top =	2.96744
TC 6	23.72617	0.00000	DT bot =	3.06825
TC 7	20.75874	0.00000	Mean temp. =	22.18000
TC 8	20.75874	0.00000		

Sample has reached steady state in automatic termination mode.

Average values at end of test:
K-factor = 0.15834 W/m°C
= 1.09808 Btu-in/ft²,hr,°F

R-value based on thermocouple separation:
R-value = 0.08147 m²°C/W
= 0.46262 ft²,hr,°F/Btu

Test Data on Thermal Conductivity Measurement (ASTM C177

Report No. CBS-990-GHA.003 Date: 12/19/99

Customer: Mr. Ghanbari
Centre for Building Studies
Concordia University

Material: gypsum board with 21% PCM material
Sample id: specimens BL5 bottom and BL18 top
Ligninsalt type
Original sample thickness
Thickness of top specimen .0129 m
Thickness of bottom specimen .013 m
Weight of sample 0 kg

Room conditions: Temperature: 22 °C
Humidity: 50 % R.H.
Coolant temperature: 22 °C

Elapsed time 0 min.

TC 1	19.67889	0.00000	CH19 Volts	2.09600
TC 2	19.70400	0.00000	CH20 Amps	0.29290
TC 3	20.38143	0.00000	K-Factor =	0.71570
TC 4	20.40650	0.00000	R-Value =	0.01809
TC 5	20.43156	0.00000	DT top =	0.62714
TC 6	20.40650	0.00000	DT bot =	0.70252
TC 7	19.77934	0.00000	Mean temp. =	20.07408
TC 8	19.80444	0.00000		

Elapsed time 15 min.

TC 1	19.95123	0.00000	CH19 Volts	2.09600
TC 2	20.00142	0.00000	CH20 Amps	0.29300
TC 3	21.17884	0.00000	K-Factor =	0.39960
TC 4	21.20809	0.00000	R-Value =	0.03241
TC 5	21.20385	0.00000	DT top =	1.16483
TC 6	21.18309	0.00000	DT bot =	1.21715
TC 7	20.00567	0.00000	Mean temp. =	20.59797
TC 8	20.05160	0.00000		

Elapsed time 30 min.

TC 1	20.17026	0.00000	CH19 Volts	2.09800
TC 2	20.19960	0.00000	CH20 Amps	0.29300
TC 3	21.82603	0.00000	K-Factor =	0.29965
TC 4	21.82603	0.00000	R-Value =	0.04322
TC 5	21.85099	0.00000	DT top =	1.53856
TC 6	21.85099	0.00000	DT bot =	1.64110
TC 7	20.29990	0.00000	Mean temp. =	21.04360
TC 8	20.32497	0.00000		

Elapsed time 45 min.

TC 1	20.33098	0.00000	CH19 Volts	2.09800
TC 2	20.35605	0.00000	CH20 Amps	0.29300
TC 3	22.30604	0.00000	K-Factor =	0.25159
TC 4	22.30604	0.00000	R-Value =	0.05147
TC 5	22.33097	0.00000	DT top =	1.82460
TC 6	22.31027	0.00000	DT bot =	1.96252
TC 7	20.45631	0.00000	Mean temp. =	21.36655
TC 8	20.50573	0.00000		

Elapsed time 60 min.

TC 1	20.51382	0.00000	CH19 Volts	2.09600
TC 2	20.51382	0.00000	CH20 Amps	0.29310
TC 3	22.68717	0.00000	K-Factor =	0.22747
TC 4	22.69139	0.00000	R-Value =	0.05693
TC 5	22.69139	0.00000	DT top =	2.01064
TC 6	22.64158	0.00000	DT bot =	2.17547
TC 7	20.61828	0.00000	Mean temp. =	21.63136
TC 8	20.69342	0.00000		

Elapsed time 75 min.

TC 1	20.59218	0.00000	CH19 Volts	2.09800
TC 2	20.61723	0.00000	CH20 Amps	0.29320
TC 3	22.99332	0.00000	K-Factor =	0.20794
TC 4	22.96843	0.00000	R-Value =	0.06228
TC 5	23.01820	0.00000	DT top =	2.20897
TC 6	22.99332	0.00000	DT bot =	2.37617
TC 7	20.77175	0.00000	Mean temp. =	21.84703
TC 8	20.82183	0.00000		

Elapsed time 90 min.

TC 1	20.67863	0.00000	CH19 Volts	2.09700
TC 2	20.70368	0.00000	CH20 Amps	0.29320
TC 3	23.19934	0.0000	K-Factor =	0.19796
TC 4	23.19934	0.00000	R-Value =	0.06542
TC 5	23.19934	0.00000	DT top =	2.30592
TC 6	23.14960	0.00000	DT bot =	2.50819
TC 7	20.82887	0.00000	Mean temp. =	21.98338
TC 8	20.90822	0.00000		

Elapsed time 105 min.

TC 1	20.75283	0.00000	CH19 Volts	2.09700
TC 2	20.78213	0.00000	CH20 Amps	0.29320
TC 3	23.34761	0.00000	K-Factor =	0.19166
TC 4	23.37669	0.00000	R-Value =	0.06757
TC 5	23.37247	0.00000	DT top =	2.37767
TC 6	23.34761	0.00000	DT bot =	2.59467
TC 7	20.95735	0.00000	Mean temp. =	22.11801
TC 8	21.00740	0.00000		

Elapsed time 120 min.

TC 1	20.78029	0.00000	CH19 Volts	2.09800
TC 2	20.78029	0.00000	CH20 Amps	0.29330
TC 3	23.44944	0.00000	K-Factor =	0.18662
TC 4	23.45366	0.00000	R-Value =	0.06939
TC 5	23.44944	0.00000	DT top =	2.43971
TC 6	23.42459	0.00000	DT bot =	2.67126
TC 7	20.95977	0.00000	Mean temp. =	22.16654
TC 8	21.03484	0.00000		

Elapsed time 135 min.

TC 1	20.82054	0.00000	CH19 Volts	2.09800
TC 2	20.79551	0.00000	CH20 Amps	0.29310
TC 3	23.53909	0.00000	K-Factor =	0.18332
TC 4	23.53909	0.00000	R-Value =	0.07064
TC 5	23.53909	0.00000	DT top =	2.46836
TC 6	23.51424	0.00000	DT bot =	2.73106
TC 7	21.04579	0.00000	Mean temp. =	22.23302
TC 8	21.07081	0.00000		

Elapsed time 150 min.

TC 1	20.75118	0.00000	CH19 Volts	2.09700
TC 2	20.77622	0.00000	CH20 Amps	0.29330
TC 3	23.54479	0.00000	K-Factor =	0.17997
TC 4	23.54479	0.00000	R-Value =	0.07195
TC 5	23.54479	0.00000	DT top =	2.51615
TC 6	23.54479	0.00000	DT bot =	2.78110
TC 7	21.00149	0.00000	Mean temp. =	22.22048
TC 8	21.05579	0.00000		

Elapsed time 165 min.

TC 1	20.74517	0.00000	CH19 Volts	2.09900
TC 2	20.77021	0.00000	CH20 Amps	0.29330
TC 3	23.56368	0.00000	K-Factor =	0.17847
TC 4	23.56368	0.00000	R-Value =	0.07256
TC 5	23.56368	0.00000	DT top =	2.54111
TC 6	23.54306	0.00000	DT bot =	2.80599
TC 7	20.97472	0.00000	Mean temp. =	22.22175
TC 8	21.04979	0.00000		

Elapsed time 180 min.

TC 1	20.71739	0.00000	CH19 Volts	2.09800
TC 2	20.74243	0.00000	CH20 Amps	0.29330
TC 3	23.58581	0.00000	K-Factor =	0.17585
TC 4	23.56096	0.00000	R-Value =	0.07364
TC 5	23.61065	0.00000	DT top =	2.58054
TC 6	23.56096	0.00000	DT bot =	2.84347
TC 7	20.99276	0.00000	Mean temp. =	22.22359
TC 8	21.01778	0.00000		

Elapsed time 195 min.

TC 1	20.71466	0.00000	CH19 Volts	2.09800
TC 2	20.71466	0.00000	CH20 Amps	0.29330
TC 3	23.55826	0.00000	K-Factor =	0.17623
TC 4	23.50856	0.00000	R-Value =	0.07348
TC 5	23.53341	0.00000	DT top =	2.59352
TC 6	23.50856	0.00000	DT bot =	2.81874
TC 7	20.88992	0.00000	Mean temp. =	22.17413
TC 8	20.96500	0.00000		

Elapsed time 210 min.

TC 1	20.67038	0.00000	CH19 Volts	2.09800
TC 2	20.72047	0.00000	CH20 Amps	0.29330
TC 3	23.56401	0.00000	K-Factor =	0.17344
TC 4	23.56401	0.00000	R-Value =	0.07466
TC 5	23.61370	0.00000	DT top =	2.63056
TC 6	23.56401	0.00000	DT bot =	2.86859
TC 7	20.94578	0.00000	Mean temp. =	22.20165
TC 8	20.97080	0.00000		

Elapsed time 225 min.

TC 1	20.63359	0.00000	CH19 Volts	2.09700
TC 2	20.65437	0.00000	CH20 Amps	0.29310
TC 3	23.52752	0.00000	K-Factor =	0.17394
TC 4	23.50267	0.00000	R-Value =	0.07445
TC 5	23.52752	0.00000	DT top =	2.60599
TC 6	23.52752	0.00000	DT bot =	2.87111
TC 7	20.90901	0.00000	Mean temp. =	22.15203
TC 8	20.93404	0.00000		

Elapsed time 240 min.

TC 1	20.63088	0.00000	CH19 Volts	2.09800
TC 2	20.65593	0.00000	CH20 Amps	0.29330
TC 3	23.49999	0.00000	K-Factor =	0.17421
TC 4	23.49999	0.00000	R-Value =	0.07434
TC 5	23.54968	0.00000	DT top =	2.61843
TC 6	23.52484	0.00000	DT bot =	2.85658
TC 7	20.90632	0.00000	Mean temp. =	22.14987
TC 8	20.93134	0.00000		

Sample has reached steady state in automatic termination mode.

Average values at end of test:
K-factor = 0.17473 W/m°C
= 1.21175 Btu-in/ft²,hr,°F

R-value based on thermocouple separation:
R-value = 0.07411 m²°C/W
= 0.42085 ft²,hr,°F/Btu

Test Data on Thermal Conductivity Measurement (ASTM C177

Report No. CBS-090-GHA.004 Date: 12-19-88

Customer: Mr. Ghanbari
Centre for Building Studies
Concordia University

Material: gypsum boards containing 22% PCM material
Sample id: specimens DP3 bottom and DP5 top
PVA type
Original sample thickness
Thickness of top specimen .0135 m
Thickness of bottom specimen .0132 m
Weight of sample 0 kg

Room conditions: Temperature: 22 °C
Humidity: 50 % R.H.
Coolant temperature: 22 °C

Elapsed time 0 min.

TC 1	20.35150	0.00000	CH19 Volts	2.09900
TC 2	20.35150	0.00000	CH20 Amps	0.29340
TC 3	22.42191	0.00000	K-Factor =	0.26762
TC 4	22.39699	0.00000	R-Value =	0.04988
TC 5	22.45108	0.00000	DT top =	1.61315
TC 6	22.47600	0.00000	DT bot =	2.05795
TC 7	20.87755	0.00000	Mean temp. =	21.51872
TC 8	20.82322	0.00000		

Elapsed time 15 min.

TC 1	20.30241	0.00000	CH19 Volts	2.09800
TC 2	20.32322	0.00000	CH20 Amps	0.29320
TC 3	22.77172	0.00000	K-Factor =	0.20713
TC 4	22.77596	0.00000	R-Value =	0.06445
TC 5	22.82152	0.00000	DT top =	2.28108
TC 6	22.79662	0.00000	DT bot =	2.46103
TC 7	20.52799	0.00000	Mean temp. =	21.60593
TC 8	20.52799	0.00000		

Elapsed time 30 min.

TC 1	20.42401	0.00000	CH19 Volts	2.09800
TC 2	20.42401	0.00000	CH20 Amps	0.29330
TC 3	23.07092	0.00000	K-Factor =	0.19015
TC 4	23.07092	0.00000	R-Value =	0.07021
TC 5	23.12068	0.00000	DT top =	2.52127
TC 6	23.12068	0.00000	DT bot =	2.64691
TC 7	20.62446	0.00000	Mean temp. =	21.80376
TC 8	20.57436	0.00000		

Elapsed time 45 min.

TC 1	20.42028	0.00000	CH19 Volts	2.09700
TC 2	20.44534	0.00000	CH20 Amps	0.29320
TC 3	23.29106	0.00000	K-Factor =	0.17760
TC 4	23.26619	0.00000	R-Value =	0.07517
TC 5	23.31592	0.00000	DT top =	2.68276
TC 6	23.29106	0.00000	DT bot =	2.84582
TC 7	20.62073	0.00000	Mean temp. =	21.90891
TC 8	20.62073	0.00000		

Elapsed time 60 min.

TC 1	20.46719	0.00000	CH19 Volts	2.09500
TC 2	20.46719	0.00000	CH20 Amps	0.29330
TC 3	23.46188	0.00000	K-Factor =	0.16843
TC 4	23.43703	0.00000	R-Value =	0.07926
TC 5	23.48674	0.00000	DT top =	2.84406
TC 6	23.51158	0.00000	DT bot =	2.98226
TC 7	20.66762	0.00000	Mean temp. =	22.01773
TC 8	20.64257	0.00000		

Elapsed time 75 min.

TC 1	20.46293	0.00000	CH19 Volts	2.09800
TC 2	20.48799	0.00000	CH20 Amps	0.29320
TC 3	23.55705	0.00000	K-Factor =	0.16305
TC 4	23.55705	0.00000	R-Value =	0.08188
TC 5	23.58190	0.00000	DT top =	2.94348
TC 6	23.60674	0.00000	DT bot =	3.08159
TC 7	20.66336	0.00000	Mean temp. =	22.06942
TC 8	20.63831	0.00000		

Elapsed time 90 min.

TC 1	20.50132	0.00000	CH19 Volts	2.09800
TC 2	20.50132	0.00000	CH20 Amps	0.29330
TC 3	23.64479	0.00000	K-Factor =	0.15982
TC 4	23.64479	0.00000	R-Value =	0.08353
TC 5	23.69447	0.00000	DT top =	3.00537
TC 6	23.66963	0.00000	DT bot =	3.14347
TC 7	20.67668	0.00000	Mean temp. =	22.12621
TC 8	20.67668	0.00000		

Elapsed time 105 min.

TC 1	20.47199	0.00000	CH19 Volts	2.09800
TC 2	20.47199	0.00000	CH20 Amps	0.29320
TC 3	23.71930	0.00000	K-Factor =	0.15536
TC 4	23.73991	0.00000	R-Value =	0.08593
TC 5	23.76474	0.00000	DT top =	3.06515
TC 6	23.76474	0.00000	DT bot =	3.25761
TC 7	20.72250	0.00000	Mean temp. =	22.16648
TC 8	20.67668	0.00000		

Elapsed time 120 min.

TC 1	20.46826	0.00000	CH19 Volts	2.09700
TC 2	20.49332	0.00000	CH20 Amps	0.29310
TC 3	23.75681	0.00000	K-Factor =	0.15408
TC 4	23.76104	0.00000	R-Value =	0.08664
TC 5	23.78165	0.00000	DT top =	3.09216
TC 6	23.78587	0.00000	DT bot =	3.27814
TC 7	20.69373	0.00000	Mean temp. =	22.17877
TC 8	20.68947	0.00000		

Elapsed time 135 min.

TC 1	20.49758	0.00000	CH19 Volts	2.09800
TC 2	20.49758	0.00000	CH20 Amps	0.29330
TC 3	23.81493	0.00000	K-Factor =	0.15156
TC 4	23.81493	0.00000	R-Value =	0.08808
TC 5	23.86458	0.00000	DT top =	3.16659
TC 6	23.86458	0.00000	DT bot =	3.31734
TC 7	20.72304	0.00000	Mean temp. =	22.21877
TC 8	20.67295	0.00000		

Elapsed time 150 min.

TC 1	20.45173	0.00000	CH19 Volts	2.09800
TC 2	20.47679	0.00000	CH20 Amps	0.29320
TC 3	23.84821	0.00000	K-Factor =	0.15000
TC 4	23.81915	0.00000	R-Value =	0.08900
TC 5	23.86880	0.00000	DT top =	3.17929
TC 6	23.81915	0.00000	DT bot =	3.36942
TC 7	20.67721	0.00000	Mean temp. =	22.20165
TC 8	20.65217	0.00000		

Elapsed time 165 min.

TC 1	20.49439	0.00000	CH19 Volts	2.09800
TC 2	20.49439	0.00000	CH20 Amps	0.29330
TC 3	23.85719	0.00000	K-Factor =	0.14933
TC 4	23.86141	0.00000	R-Value =	0.08940
TC 5	23.91106	0.00000	DT top =	3.21626
TC 6	23.91106	0.00000	DT bot =	3.36491
TC 7	20.71985	0.00000	Mean temp. =	22.23989
TC 8	20.66976	0.00000		

Elapsed time 180 min.

TC 1	20.47733	0.00000	CH19 Volts	2.09800
TC 2	20.47733	0.00000	CH20 Amps	0.29330
TC 3	23.86933	0.00000	K-Factor =	0.14898
TC 4	23.86933	0.00000	R-Value =	0.08961
TC 5	23.89416	0.00000	DT top =	3.20387
TC 6	23.89839	0.00000	DT bot =	3.39201
TC 7	20.70705	0.00000	Mean temp. =	22.23383
TC 8	20.67775	0.00000		

Elapsed time 195 min.

TC 1	20.43628	0.00000	CH19 Volts	2.09800
TC 2	20.48640	0.00000	CH20 Amps	0.29330
TC 3	23.85772	0.00000	K-Factor =	0.14893
TC 4	23.82867	0.00000	R-Value =	0.08964
TC 5	23.88255	0.00000	DT top =	3.21642
TC 6	23.90314	0.00000	DT bot =	3.38186
TC 7	20.66603	0.00000	Mean temp. =	22.21845
TC 8	20.68681	0.00000		

Elapsed time 210 min.

TC 1	20.49655	0.00000	CH19 Volts	2.09800
TC 2	20.51734	0.00000	CH20 Amps	0.29330
TC 3	23.93802	0.00000	K-Factor =	0.14806
TC 4	23.91320	0.00000	R-Value =	0.09016
TC 5	23.93802	0.00000	DT top =	3.21804
TC 6	23.96284	0.00000	DT bot =	3.41866
TC 7	20.74278	0.00000	Mean temp. =	22.27884
TC 8	20.72200	0.00000		

Sample has reached steady state in automatic termination mode.

Average values at end of test:
K-factor = 0.14906 W/m°C
= 1.03371 Btu-in/ft²,hr,°F

R-value based on thermocouple separation:
R-value = 0.08956 m²°C/W
= 0.50857 ft²,hr,°F/Btu

Test Data on Thermal Conductivity Measurement (ASTM C177

Report No. CBS-090-GHA.005 Date: 12-19-88

Customer: Mr. Ghanbari
Centre for Building Studies
Concordia University

Material: gypsum boards containing 21% PCM material
Sample id: specimens BL6 bottpm and BL9 top
WFA type
Original sample thickness
Thickness of top specimen .0138 m
Thickness of bottom specimen .0138 m
Weight of sample 0 kg

Room conditions: Temperature: 22 °C
Humidity: 50 % R.H.
Coolant temperature: 22 °C

Elapsed time 0 min.

TC 1	20.34463	0.00000	CH19 Volts	2.09900
TC 2	20.34463	0.00000	CH20 Amps	0.29330
TC 3	22.79299	0.00000	K-Factor =	0.23128
TC 4	22.71829	0.00000	R-Value =	0.05967
TC 5	22.74319	0.00000	DT top =	1.98510
TC 6	22.76809	0.00000	DT bot =	2.41101
TC 7	20.79558	0.00000	Mean temp. =	21.65661
TC 8	20.74551	0.00000		

Elapsed time 15 min.

TC 1	20.28595	0.00000	CH19 Volts	2.09800
TC 2	20.31102	0.00000	CH20 Amps	0.29320
TC 3	22.96300	0.00000	K-Factor =	0.19580
TC 4	22.95876	0.00000	R-Value =	0.07048
TC 5	23.03765	0.00000	DT top =	2.52603
TC 6	23.06253	0.00000	DT bot =	2.66239
TC 7	20.56164	0.00000	Mean temp. =	21.70838
TC 8	20.48647	0.00000		

Elapsed time 30 min.

TC 1	20.35262	0.00000	CH19 Volts	2.09900
TC 2	20.37769	0.00000	CH20 Amps	0.29330
TC 3	23.12445	0.00000	K-Factor =	0.18984
TC 4	23.07470	0.00000	R-Value =	0.07269
TC 5	23.17420	0.00000	DT top =	2.62127
TC 6	23.12445	0.00000	DT bot =	2.73442
TC 7	20.55311	0.00000	Mean temp. =	21.78553
TC 8	20.50300	0.00000		

Elapsed time 45 min.

TC 1	20.42465	0.00000	CH19 Volts	2.09800
TC 2	20.42465	0.00000	CH20 Amps	0.29330
TC 3	23.19592	0.00000	K-Factor =	0.18589
TC 4	23.19592	0.00000	R-Value =	0.07424
TC 5	23.27053	0.00000	DT top =	2.69553
TC 6	23.27053	0.00000	DT bot =	2.77128
TC 7	20.57499	0.00000	Mean temp. =	21.86652
TC 8	20.57499	0.00000		

Elapsed time 60 min.

TC 1	20.40757	0.00000	CH19 Volts	2.09900
TC 2	20.45343	0.00000	CH20 Amps	0.29340
TC 3	23.35304	0.00000	K-Factor =	0.17900
TC 4	23.32818	0.00000	R-Value =	0.07710
TC 5	23.37790	0.00000	DT top =	2.77200
TC 6	23.37790	0.00000	DT bot =	2.91011
TC 7	20.63308	0.00000	Mean temp. =	21.93873
TC 8	20.57871	0.00000		

Elapsed time 75 min.

TC 1	20.41556	0.00000	CH19 Volts	2.09800
TC 2	20.46568	0.00000	CH20 Amps	0.29340
TC 3	23.36096	0.00000	K-Factor =	0.17710
TC 4	23.33610	0.00000	R-Value =	0.07792
TC 5	23.41068	0.00000	DT top =	2.83224
TC 6	23.41068	0.00000	DT bot =	2.90791
TC 7	20.59096	0.00000	Mean temp. =	21.94457
TC 8	20.56591	0.00000		

Elapsed time 90 min.

TC 1	20.45824	0.00000	CH19 Volts	2.09900
TC 2	20.45824	0.00000	CH20 Amps	0.29330
TC 3	23.45724	0.00000	K-Factor =	0.17443
TC 4	23.40753	0.00000	R-Value =	0.07911
TC 5	23.48209	0.00000	DT top =	2.85461
TC 6	23.47786	0.00000	DT bot =	2.97414
TC 7	20.63790	0.00000	Mean temp. =	21.99900
TC 8	20.61285	0.00000		

Elapsed time 105 min.

TC 1	20.46197	0.00000	CH19 Volts	2.09900
TC 2	20.48702	0.00000	CH20 Amps	0.29340
TC 3	23.48155	0.00000	K-Factor =	0.17346
TC 4	23.45670	0.00000	R-Value =	0.07956
TC 5	23.50640	0.00000	DT top =	2.86895
TC 6	23.53125	0.00000	DT bot =	2.99463
TC 7	20.68744	0.00000	Mean temp. =	22.02808
TC 8	20.61230	0.00000		

Elapsed time 120 min.

TC 1	20.39903	0.00000	CH19 Volts	2.09800
TC 2	20.47422	0.00000	CH20 Amps	0.29330
TC 3	23.46885	0.00000	K-Factor =	0.17142
TC 4	23.44400	0.00000	R-Value =	0.08050
TC 5	23.54340	0.00000	DT top =	2.90856
TC 6	23.51855	0.00000	DT bot =	3.01980
TC 7	20.64959	0.00000	Mean temp. =	22.01161
TC 8	20.59523	0.00000		

Elapsed time 135 min.

TC 1	20.48331	0.00000	CH19 Volts	2.09700
TC 2	20.48331	0.00000	CH20 Amps	0.29320
TC 3	23.50271	0.00000	K-Factor =	0.17094
TC 4	23.50271	0.00000	R-Value =	0.08073
TC 5	23.57725	0.00000	DT top =	2.92069
TC 6	23.59148	0.00000	DT bot =	3.01941
TC 7	20.68372	0.00000	Mean temp. =	22.05602
TC 8	20.63363	0.00000		

Elapsed time 150 min.

TC 1	20.47849	0.00000	CH19 Volts	2.09800
TC 2	20.49928	0.00000	CH20 Amps	0.29330
TC 3	23.51855	0.00000	K-Factor =	0.16943
TC 4	23.51855	0.00000	R-Value =	0.08145
TC 5	23.61793	0.00000	DT top =	2.96834
TC 6	23.61793	0.00000	DT bot =	3.02966
TC 7	20.67464	0.00000	Mean temp. =	22.06874
TC 8	20.62454	0.00000		

Elapsed time 165 min.

TC 1	20.44489	0.00000	CH19 Volts	2.09800
TC 2	20.46995	0.00000	CH20 Amps	0.29330
TC 3	23.53916	0.00000	K-Factor =	0.16965
TC 4	23.48947	0.00000	R-Value =	0.08134
TC 5	23.56401	0.00000	DT top =	2.93332
TC 6	23.56824	0.00000	DT bot =	3.05690
TC 7	20.64533	0.00000	Mean temp. =	22.04267
TC 8	20.62028	0.00000		

Elapsed time 180 min.

TC 1	20.48331	0.00000	CH19 Volts	2.09500
TC 2	20.50410	0.00000	CH20 Amps	0.29350
TC 3	23.57302	0.00000	K-Factor =	0.16814
TC 4	23.54817	0.00000	R-Value =	0.08207
TC 5	23.62694	0.00000	DT top =	2.97253
TC 6	23.62694	0.00000	DT bot =	3.06689
TC 7	20.67946	0.00000	Mean temp. =	22.08391
TC 8	20.62936	0.00000		

Elapsed time 195 min.

TC 1	20.47422	0.00000	CH19 Volts	2.09900
TC 2	20.49928	0.00000	CH20 Amps	0.29350
TC 3	23.56401	0.00000	K-Factor =	0.16829
TC 4	23.54340	0.00000	R-Value =	0.08200
TC 5	23.61370	0.00000	DT top =	2.97866
TC 6	23.63854	0.00000	DT bot =	3.06696
TC 7	20.67037	0.00000	Mean temp. =	22.07851
TC 8	20.62454	0.00000		

Sample has reached steady state in automatic termination mode.

Average values at end of test:
K-factor = 0.16929 W/m°C
= 1.17400 Btu-in/ft²,hr,°F

R-value based on thermocouple separation:
R-value = 0.08152 m²°C/W
= 0.46289 ft²,hr,°F/Btu

Appendix C

Here are the calculated standard deviations of the results of the tests for evaluation of the Foam densities (T, 5.5-5.12), Compressive strength (T, 6.1-6.4), Paste densities (T ,6.5-6.9), Flexural strength (T, 6.10-614) and Thermal setting time (T, 7.1) of the specimens under investigation.

The calculation was done using the following familiar formula.

$$s.d = \sqrt{\Sigma (\Delta x_i)^2 / (n-1)}$$

where

xi is independent reading.

(Δxi) is the difference between any individual reading (xi) and the Mean or Average $\bar{x}$. i.e. $(\bar{x} = 1/n \sum_{i=1}^{n} xi)$.

n is the number of reading for each categories of the tests.

Therefore our results in the abovementioned tables will be $(\bar{x} \pm s.d)$.

Table c.1

Calculated standard deviations

Table	s.d.	Table	s.d.
5.5	± 0.001	5.11	±0.002
	± 0.001		± 0.001
	± 0.002	5.12	± 0.001
	± 0.001		± 0.002
5.6	± 0.001		± 0.001
	± 0.001		± 0.001
	± 0.001	6.1	± 6.3
	± 0.001	6.2	± 4.6
5.7	± 0.001	6.3	± 10.6
	± 0 002	6.4	± 8.7
	± 0.001	6.5	± 0.035
	± 0.001	6.6	± 0.008
5.8	± 0.001	6.7	± 0.02
	± 0.001	6.8	± 0.008
	± 0.001	6.9	± 0.007
	± 0.001		± 0.002
5.9	± 0.001		± 0.005
	± 0.001		± 0.012
	± 0.001	6.10 (para)	± 3.9
	± 0.002	(perp)	± 6.2
5.10	± 0.001	(plain)	± 1.2
	± 0.001	6.11 (para)	± 0.88
	± 0.001	(perp)	± 1.6
	± 0.001	6.12	± 0.58
5.11	± 0.001		± 2.2
	± 0.001	6.13	± 0.64
			± 1.6
6.14 (para)	± 0.58	6.14 (perp)	± 1.6
7.1 All four sets , i.e. (plain), (WFA), (PVA), and (SSKL)			± 0.58