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ENCAPSULATION OF PHASE CHANGE MATERIALS IN CONCRETE MASONRY CONSTRUCTION

PROGRESS REPORT NO. 3
(FINAL REPORT)
JUNE 1978-SEPTEMBER 1978

PROCESS SCIENCES DIVISION
DEPARTMENT OF ENERGY AND ENVIRONMENT

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Abstract

Work performed at Brookhaven National Laboratory on Union Carbide Nuclear Division, ORNL contract number 19Y-14279V, "Encapsulation of Phase Change Materials in Concrete Masonry Construction", is summarized in this Third Progress Report for the period of June 1978 - September 1978. Due to program termination, this report also constitutes the final report of the program. Thermal analysis of the system $Na_2CO_3 \cdot 10H_2O$ has shown that after separation upon melting, the heat of fusion is considerably higher than $Na_2SO_4 \cdot 10H_2O$. The system $NaCO_3$-K_2CO_3-H_2O has been found to be unsuitable for PCM use. A "eutectic" in the $MgCl_2$-$CaCl_2$-H_2O is under consideration. It exhibits a high heat of fusion and low transition temperature range. Cylindrical shells have been cast using polymer concrete. The shells were subsequently filled with PCM (phase change material) and thermally cycled. No leaking of PCM occurred. Thermal measurements on PCM composite materials were continued.

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Summary

Work in the area of selection of suitable phase change materials (PCM) was continued. $Na_2CO_3.10H_2O$, which melts incongruently at about 32°C, was observed to have a higher heat of fusion than $Na_2SO_4.10H_2O$ if recombination of water and salt does not occur upon solidification for both PCM's. For $Na_2CO_3.10H_2O$, the heat of fusion goes from 59 to 44 cal/gram while $Na_2SO_4.10H_2O$ goes from 60 to 33 cal/gram. The disadvantages of $Na_2CO_3.10H_2O$ compared with $Na_2SO_4.10H_2O$ are higher cost (about 5¢/lb as opposed to 2¢/lb) and its high alkalinity (pH= 11.7 while $Na_2SO_4.10H_2O$ is neutral). The system Na_2CO_3-K_2CO_3-H_2O was found to be unsuitable for use as a PCM since it forms no eutectics in the low temperature range with high heats of fusion. A promising composition in the $CaCl_2$-$MgCl_2$-H_2O system was found which exhibited a heat of fusion of 42 cal/gram and a broad melting range which extended from 15° to 26°C. This system appears most promising and further work in this area should be undertaken.

Macroencapsulation of PCM in hollow cylindrical shells of polymer concrete was started. This task is much simpler than the "microencapsulation" work which we have previously undertaken. The samples are easily fabricated and no leaking of PCM has occurred.

Encapsulation of a PCM (phenyl ether) in a nylon capsule was performed via a method developed and patented by Pennwalt Corp. (US 3,577,515). The method worked quite well but costs will probably overrule this type of encapsulation procedure. In addition, the capsules were too soft so that a hardener or different polymer system might have to be employed.

Encapsulation of decanoic acid by 3M Corporation was performed and the sample analyzed by DSC. The capsules were too small (10-38 microns) and

supercooling, a problem not heretofore encountered with decanoic acid, was noted.

Thermal measurements via the differential scanning calorimeter (DSC), thermal comparator, and the Seebeck envelope calorimeter (SEC) have been continued.

Encapsulation of Phase Change Materials in
Concrete Masonry Construction

Progress Report No. 3

Final Report

June 1978 - September 1978

Introduction

Space conditioning is the major energy consuming function for both commercial and residential buildings. Low temperature thermal energy storage (LTTES) in the walls, floors, and/or ceilings of these structures can lead to substantial energy savings. One way to increase thermal capacity is via incorporation of phase change materials (PCM's) in the building materials, thus increasing their average heat capacity. The most likely PCM candidates are substances which undergo a solid-liquid transformation at the desired temperature of energy storage. Such materials should have a high heat of fusion, stability with respect to thermal cycling, and chemical compatibility with the encapsulating medium. It is readily calculated that the incorporation of 20% of an active PCM with a heat of fusion of 35 calories/gram and transition temperature of 25°C will raise the stored heat from 2.2 calories/gram to 9.0 calories/gram over the temperature interval 20°-30°C. At 30% PCM loading, 15.7 calories/gram would be stored. Thus substantial increases in thermal energy storage can be achieved with relatively small amounts of PCM.

The PCM transition temperatures for thermal energy storage are determined by the desired application: hydronic heating, 75°-80°C; hot water, 55°-75°C; hot air, 35°-55°C; wall panel, 20°-35°C; cold storage, 5°-20°C. For the present study, the main concern will be the encapsulation of PCM's which undergo phase transition in the 20°-35°C range and preferably in the 20°-24°C range. This latter range is ideal for a completely passive thermal energy storage system which would require minimal supporting devices for energy delivery.

The encapsulation of phase change material in ordinary concrete offers a low cost method for energy storage. Since normal weight concrete is roughly 75-80% aggregate, a partial replacement of this aggregate with cement-compatible PCM will increase its energy storage capacity. Since the curing of portland cement depends upon hydration reactions, it is imperative that the PCM not interfere with these reactions. The PCM must also not react with the aggregate and must not interfere with the bonding between the cement and aggregate. Our studies thus far have indicated that a prior encapsulation of the PCM will be necessary for use in a concrete matrix, probably through the coating of porous aggregate impregnated with PCM.

Polymer-impregnated concrete (PIC) consists of pre-cast concrete impregnated with a suitable monomer and polymerized <u>in situ</u>. Significant improvements in strength and durability as well as an impermeable material result from this procedure. In order to use PIC, a concrete sample containing PCM must be developed. This sample may be only partially hydrated and of low strength. It might then be possible to achieve both PCM confinement and adequate strength via polymer impregnation.

Polymer concrete (PC) consists of aggregate mixed with suitable monomer instead of portland cement. Subsequent polymerization yields a high strength, durable, and impervious material. The methods for mixing and placement are comparable to ordinary concrete. High strength PC (and PIC) can both be made from relatively low quality, low strength aggregate. At this time, PC seems to offer the best prospect for success.

The objectives of this project were to develop techniques for the encapsulation of phase change materials into concrete, polymer-impregnated concrete, polymer concrete, and/or other building materials and to characterize the physical and thermal properties of these materials. Since the program was unexpectedly terminated, much of the work is in an incomplete stage. However, the program is such that the effort could be renewed in short order if funding should again become available.

Task 1. Identification and Selection of Phase Change Materials

The phase diagrams for $Na_2SO_4.H_2O$ and $Na_2CO_3.H_2O$ (Figures 1 and 2) show that both salts have roughly the same solubility in water, both exhibit retrograde solubility, and the decahydrates melt incongruently. $Na_2CO_3.10H_2O$ is 63% water, 37% anhydrous salt, while $Na_2SO_4.10H_2O$ is 56% water, 44% anhydrous salt. However, since both salts have comparable solubilities, the carbonate leaves behind less undissolved material (∿ 5.3% $Na_2CO_3.H_2O$) than the sulfate (∿ 16.4% Na_2SO_4) after melting at the common melting temperature of 32°C. Thus, if recombination of undissolved solid and saturated solution does not occur during crystallization, then Na_2CO_3 solutions should yield significantly higher heats of fusion. Second meltings of $Na_2SO_4.10H_2O$ have given heats of fusion of about 33 cal/gram while $Na_2CO_3.10H_2O$ yielded 44 cal/gram.

A DSC scan (see Figure 3) of a saturated Na_2SO_4 solution displays two peaks, one at 28°C and the other at -6°C. The higher temperature peak had a heat of fusion of 32 cal/gram. Similar results were obtained for a saturated Na_2CO_3 solution. It is thus apparent that either salt is

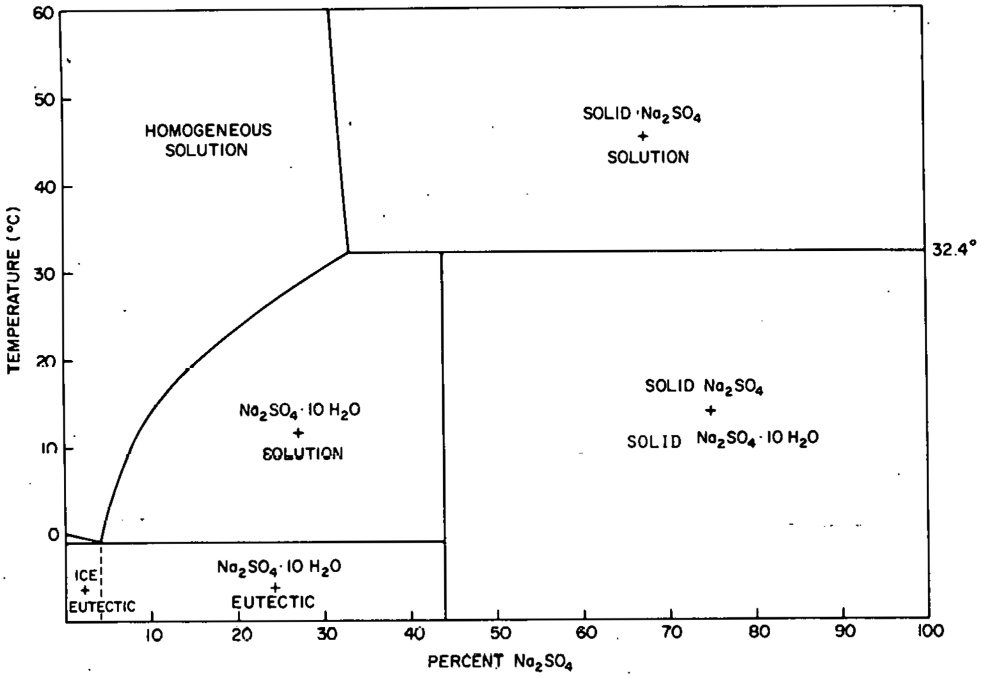

Figure 1. Phase diagram for $Na_2SO_4 \cdot H_2O$ system

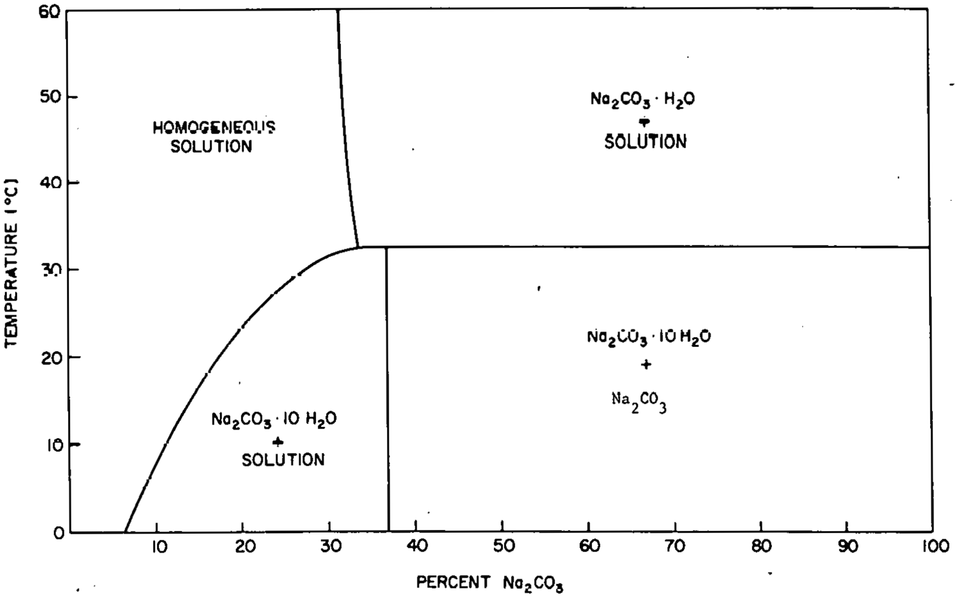

Figure 2. Phase diagram for $Na_2CO_3 \cdot H_2O$ system

potentially useful as a PCM in the form of its saturated solution. This might find application in such systems as Sunteck's Thermocrete[1] or in our own porous aggregate system, where it is difficult or impossible to inject slurries into the systems.

The Dow Chemical Co.[2] report lists several "eutectic" systems of Na_2CO_3-K_2CO_3-H_2O as potentially useful phase change materials. The compositions and temperatures of transition are:

	22°C	25°C	30°C
K_2CO_3	21.3	48.8	45.6
Na_2CO_3	16.0	4.8	6.9
H_2O	62.7	46.4	47.5

The entries are in weight percent. No phase change was observed for the 25°C and 30°C compositions over the range of 10°C to 30°C where the systems are solutions. The 22°C system exhibited two peaks on the DSC, one below zero and the other a broad melting peak beginning at about 17°C. The heat of fusion was recorded as 19.7 cal/gram which is far too low to be useful. Work on these systems will be discontinued.

The Dow Report also lists several "entectics" of $CaCl_2$-$MgCl_2$-H_2O as potentially useful PCM's.

	I	II	III
$CaCl_2$	31.2	38.3	52.2
$MgCl_2$	14.2	9.4	3.4
H_2O	54.6	52.3	44.4

These systems are not true eutectics but two of them are of interest since they have reasonably high heats of fusion. System I exhibits two peaks in the DSC thermogram, one at -23°C and the other at 10°C. The higher melting

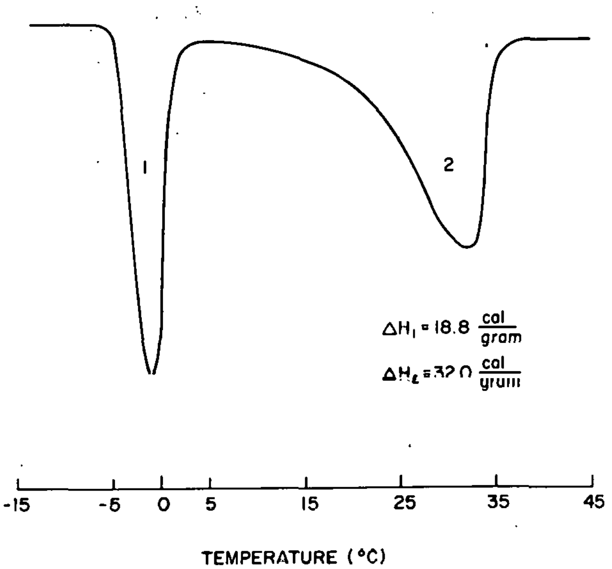

Figure 3. DSC thermogram saturated Na_2SO_4 solution

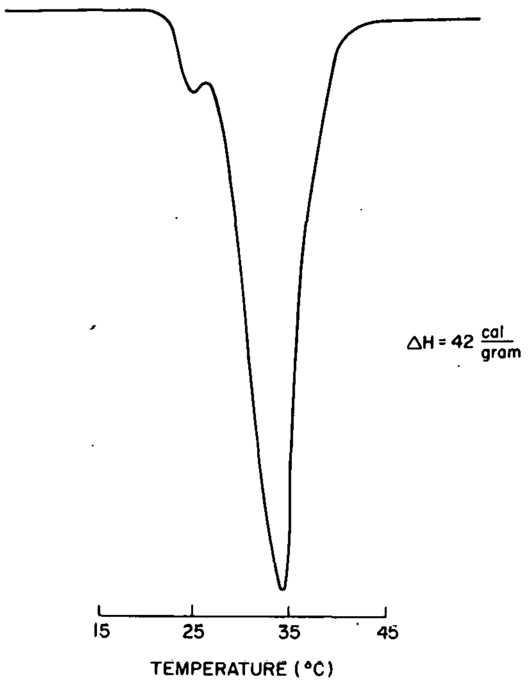

Figure 4. DSC thermogram .522 $CaCl_2$-.034 $MgCl_2$-.444 H_2O

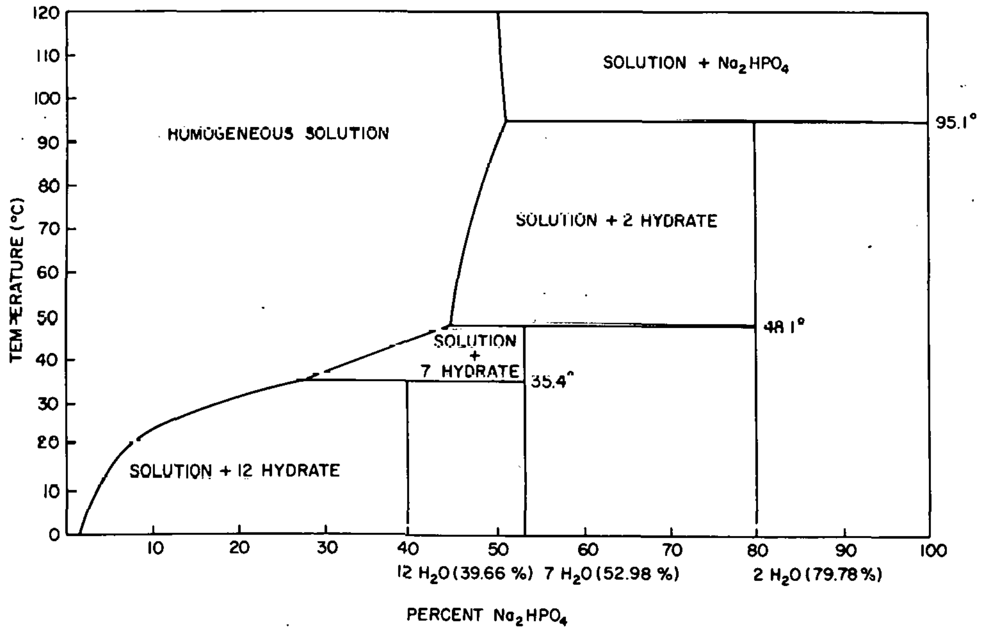

Figure 5. Phase diagram for $Na_2HPO_4 \cdot H_2O$ system

peak is quite broad with the onset of melting occurring at 0°C. The heat of fusion was measured at 13.2 cal/gram. System II exhibits two peaks in the thermogram, although two of five samples developed a third peak after roughly four or five thermal cycles. The higher melting peak is broad with a melting onset temperature of -15°C. The heat of fusion is 31.5 cal/gram. The most promising system is III which exhibited a phase transition beginning at about 24°C. The heat of fusion was determined by DSC (see Figure 4) to be 42 cal/gram. This material is considered to be one of the most promising candidates for use as a PCM. The melting temperture is ideal for passive applications.

Disodium phosphate dodecahydrate, $Na_2HPO_4.12H_2O$, was briefly considered as a potential phase change material. Its biggest drawback is its high melting temperature of 35.4°C. The phase diagram is shown in Figure 5. The dodecahydrate is about 60% water, 40% Na_2HPO_4. Na_2HPO_4 forms a number of hydrates including a dihydrate and a heptahydrate. Upon heating the dodecahydrate to its melting point, a two phase region of solution plus heptahydrate is encountered (incongruent melt). At about 45°C, a single solution phase remains. It would thus be possible to utilize this material with a system such as the foamed glass aggregate or the Sunteck system which are difficult to fill with incongruently melting materials such as $Na_2SO_4.10H_2O$ or $Na_2CO_3.10H_2O$ by heating $Na_2HPO_4.12H_2O$ above 45°C before impregnation. The heat of fusion, as one would expect from the number of water molecules bound in the hydrate structure, is high, about 60 cal/gram. The material has a tendency to supercool as do all the inorganic salt hydrates and no nucleating agent is known.

The investigation of polyethylene glycols as potential PCM's was continued. These materials are low molecular weight polymers of ethylene glycol and have a broad melting range. They do not require nucleating agents since they do not supercool to any appreciable extent. Their broad melting range allows them to function continuously, storing and releasing heat over the melting interval. The heats of fusion are somewhat low, 30 cal/gram being a representative value. Their major drawback is a low thermal conductivity ($\sim$ 0.19 watts/meter-^{o}C) which could make charging and discharging a problem. The DSC thermogram of a 50/50 mixture of PEC 600 and PEG 1000 is shown in Figure 6. The heat of fusion was determined to be 31.5 cal/gram.

It is clear from the above discussion and from our previous progress reports[3] that there is no single phase change material which is clearly superior to the others. The inorganic salt hydrates are generally cheaper than the organic PCM's. The least expensive of the inorganics are $Na_2SO_4.10H_2O$, $Na_2CO_3.10H_2O$, and $CaCl_2.6H_2O$. These range in price from 2 to 5 cents per pound. It would thus appear that if an inorganic system is chosen, it should be one of these three. The decahydrates both melt incongruently which might pose a problem for some applications. Sodium carbonate is highly basic (pH $\sim$12) and might be chemically incompatible in some systems while sodium sulfate is neutral in solution. Calcium chloride has the advantage of melting congruently but is hygroscopic. On balance, we feel that $CaCl_2.6H_2O$ is probably the best system overall and that $Na_2SO_4.10H_2O$ is a very close second. The system $CaCl_2$-$MgCl_2$-H_2O (System III above) should be examined more carefully if a melting temperature around 24^{o}C is necessary. It is also recommended that further work on the $Na_2CO_3.10H_2O$ material be conducted.

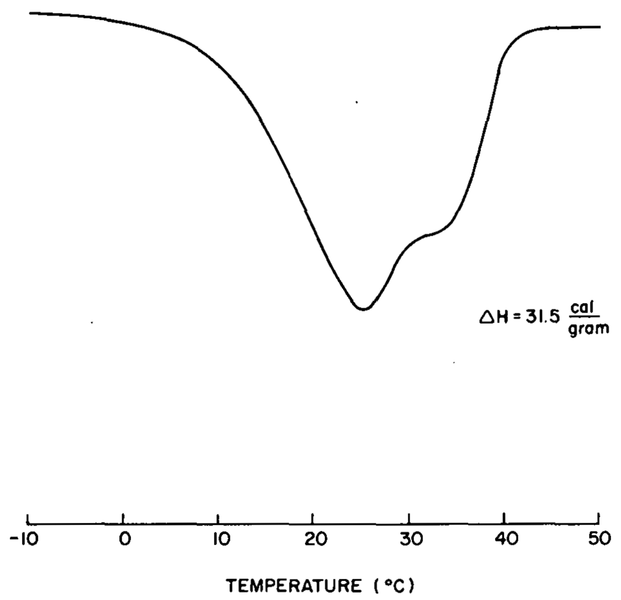

Figure 6. DSC thermogram .50 PEG 600-.50 PEG 1000

The organic PCM's have the major disadvantage of costing several times more than the cheapest inorganic systems. In general they also have lower heats of fusion so that for a given heat storage capacity, they are five to ten times more expensive. The fatty acids (decanoic, octanoic, palmitic and their eutectics) have the distinct advantage of not supercooling. But this is somewhat offset by their poorer thermal conductivities (a factor of 2-4 times lower than the salt hydrates). On balance we feel that these materials are too expensive to see any application in the area of residential thermal storage.

Task 2. Encapulation Techniques

An alternative to the microencapsulation methods which had been previously attempted in this program is the confinement of phase change materials in polymer concrete shells of various geometries. The impetus for this change of emphasis is the realization that macroencapsulation would be more likely to attain near term success. The shells can find application in both load bearing and non-load bearing walls. The latter application is ideal for retrofitting existing structures with thermal storage capacity. In load bearing applications, the polymer concrete would be fashioned as bricks or blocks of sufficient wall thickness with or without internal webbing for additional strength. Alternatively, for non-load bearing applications, the PC shells would have a much lower strength requirement. The shells could be built into a decorative firewall, a storage wall in the basement, or fashioned as wall and ceiling tiles.

Standard concrete blocks can be fabricated with any desired number, shape, and distribution of holes into which pre-cast materials can be inserted. It is clearly possible to increase the heat capacity of these blocks by using pre-cast polymer concrete shells filled with PCM as inserts.

Cylindrical shells have been cast using polymer concrete. The PC used a 1/1 ratio of #30 and #100 sand. The polymer comprised 12% of the composite weight. The monomer consisted of 85% methyl methacrylate, 15% TMPTMA, and 5% polymethyl methacrylate initiated with 2% benzoyl peroxide and promoted with 1% dimethyl aniline. Glass molds coated with a mold release agent (Frekote) and equipped with a 1" diameter teflon rod were used to cast the cylinders. The wall thickness was about 1/8". The cylinders were sealed with PC caps which were held in place with epoxy or other resins. About fifteen samples were made and filled with various PCM's for subsequent thermal cycling.

Since chemical compatibility between the supporting matrix and the PCM is generally the most difficult problem, the advantage of prior encapsulation of phase change materials either as liquids or solids is obvious. Microcapsules of phenyl ether, a potential PCM which melts at 27°C, were prepared using a nylon coating. Phenyl ether was chosen solely on the basis of chemical compatibility with the encapsulating material to demonstrate feasibility of the method. The procedure was basically that of patent example #1 according to the Pennwalt patent (US 3,577,515) except that diethylene triamine was substituted for 1,3,5 benzenetriamine trihydrochloride. The procedure consisted of suspending an organic phase composed of PCM (in this case phenyl ether), sebacoyl chloride, and trimesoyl trichloride (crosslinking agent) in distilled water containing 1/2% of an oil in water emulsifier such as Gelvatol. To this suspension was added an aqueous solution of ethylene diamine, diethylene triamine (crosslinking agent) and base (NaOH and Na_2CO_3). The addition of this latter solution was made with agitation so that as the interfacial polycondensation of the di-acid chloride and diamine proceeded, the organic phase (PCM) was encapsulated within the nylon wall. The resulting capsules were well formed and ranged in size from about 0.1 to 2 mm. However, the shells were too soft for blending into concrete or other masonry materials. A stronger polymer or an additional coating would be required.

A sample of decanoic acid was sent to 3M Corporation for microencapsulation. Their procedure required that the internal phase be water immisible. The capsules which they prepared had a payload of 76% (percent PCM) and were in the range of 10-38 microns which was one order of magnitude below the size requested due to technical problems. The encapsulating material is a urea-formaldehyde resin. A DSC scan of this material showed that, while the melting temperature and heat capacity had not been affected, the PCM now displayed supercooling, a characteristic not heretofore observed with decanoic acid. It can be concluded that microencapsulation at these dimensions is detrimental to the freezing behavior of PCM's. Further work in this area is definitely needed.

Since microencapsulation in the 1-500 micron range will probably prove to be too expensive for application to low temperature thermal energy storage systems, the best prospects for immediate success probably reside with utilizing existing technologies. The following work is thus proposed:

1) Encapsulation of PCM's by a Southwest Research Institute (SwRI) process. The process centers on a high capacity device for microencapsulation. The device consists of a nozzle of two concentric tubes mounted axially in a tubular duct. The internal phase (PCM) is pumped through the center tube while the shell forming material flows through the annular

region. The outer duct transports a non-reactive immiscible carrier fluid in the direction of the extrusion. It serves to support the extruded material which tends to break up into spheres of internal phase surrounded by shell material. These spheres are swept away in the carrier stream to a hardening bath where the walls are hardened by chemical reaction, cooling, or solvent extraction depending on the wall material. SwRI recommended shell formulations consisting of mixtures of parafin wax and hydrocarbon resins and feels that PCM encapsulation by their process is feasible.

2) Encapsulation of PCM-impregnated porous aggregate by Wurster process. This process is used for the encapsulation of solids. It is a one-step process which combines coating, drying, and blending in one vessel. Solid particles are fluidized in an air stream which produces a cyclic flow. Each time the particles pass through the coating zone, they receive a small increment of coating. The residence time is of such duration to ensure the desired thickness of coating. The prospects for the encapsulation of the impregnated foamed glass aggregate from Teledyne via this process are quite good.

Task 3. Thermal and Physical Properties Measurement

Calorimetry on composite samples which are generally far too large for DSC analysis was conducted with a Seebeck Envelope Calorimeter (SEC) purchased from Thermonetics Corporation of San Diego. The calorimeter consists of a 2-1/4" cube enclosed in a jacket through which a constant temperature fluid can be circulated. The calorimeter walls consist of thermoelectric transducers that relate thermal flux to a DC voltage output signal. (The thermal flux is proportional to the small temperature difference across the thermopile transducers.) The generation of a potential difference in this manner is known as the Seebeck effect. Thus, the calorimeter measures heat flow through its walls directly. The total heat is obtained by calculating the area under the heat flow versus time curve. From this quantity, the specific heat or heat of fusion can be readily determined. The calorimeter constant was found to be 0.0272 cal/(sec-mv) which is equivalent to 39.716 cal/in^2 at a chart speed of 20 cm/hr and 540 mv (millivolts) full scale deflection. The peak areas were determined by means of an electronic planimeter (Numonics Corp., Lansdale, Pa.). Results obtained with the calorimeter are presented in Table 1. It is apparent that the PCM's are undergoing phase transition and exhibiting the expected increase in heat capacity. The question of whether or not acceptably high heat transfer rates through macroencapsulated phase change materials can be achieved is a subject requiring further investigation.

Conclusions and Recommendations

Since this report also represents the final report for this program, it is appropriate at this point to summarize the major conclusions and make recommendations for future work. It must be remembered that the program life was only one year with an unexpected and unforwarned demise so that much of our work was in an incompleted stage.

1) Ordinary portland cement concrete and all the phase change materials examined in the program were incompatible. The cement hydration reactions did not proceed when the materials were simply mixed and the material did not set up. The PCM's used were inorganic salt hydrates ($CaCl_2.6H_2O$, $Na_2SO_4.10H_2O$, $Na_2CO_3.10H_2O$, $Na_2HPO_4.12H_2O$, $Zn(NO_3)_2.6H_2O$, $Na_2S_2O_3.5H_2O$) and organic materials (decanoic acid, PEG 600 and PEG 1000). Thus if PCM's are to be used with ordinary portland cement, prior encapsulation of the PCM is required.

Table 1

Heat Capacity of PCM's Encapsulated in Polymer Concrete Shells

Sample No.	PCM	Total Heat[a] (cal)	Ti(^{o}C)	Tf(^{o}C)	T(^{o}C)	Wt PCM, g	Wt shell, g	Wt total[b], g	Cp (actual) cal g^{-1} $^{o}C^{-1}$	Cp (calculated) cal g^{-1} $^{o}C^{-1}$
29	$CaCl_2.6H_2O$ 1% $Ba(OH)_2$	1469	23.8	40.8	17	23.43	46.65	70.08	1.23	1.19
32	PEG 600	745	9.7	30.5	20.8	14	42.7	56.7	0.63	0.685
38	$CaCl_2.6H_2O$	1228	9.2	35.1	25.9	16.14	46.41	67.28	0.705	0.696
38A	$CaCl_2.6H_2O$	1116	19.6	35.4	15.8	20.42	42.04	66.73	1.06	1.16

[a] The calorimeter heat capacity is roughly 13.57 cal/^{o}C and has been accounted for.

[b] In samples 38 and 38A, the shells also include impregnated foamed glass aggregate so that wt PCM + wt shell + wt aggregate = total weight.

2) Direct microencapsulation of PCM into polymer concrete wherein the PCM is dispersed throughout the supporting PC matrix will probably be unsuccessful for several reasons. The PCM is admixed as a solid. Upon melting there is a volume increase ranging from 8 to 15% depending on PCM. After a number of thermal cycles, the internal pressure generated is sufficient for the liquid to find its way through the polymer concrete matrix to the surface resulting in a loss of heat capacity and potential damage to the residence. In addition, polymer concrete is more expensive than ordinary concrete and attempts to confine the PCM by an additional coating to the outside surface will further increase costs. All samples of polymer concrete made by simply substituting part of the aggregate with PCM ultimately leaked. Thus, as with an ordinary concrete support matrix, prior encapsulation is required.

3) Since 400,000 Btu's of stored thermal energy is required for typical residential storage, the cost of the phase change material per Btu is a prime consideration. By far the least expensive materials are $CaCl_2.6H_2O$, $Na_2SO_4.10H_2O$, and $Na_2CO_3.10H_2O$. The first is hygroscopic and acidic (pH =3) while the two latter materials melt incongruently. All these materials have a tendency to supercool. These are the preferred materials for low temperature storage arranged in order of our preference. It is unlikely that the organic PCM's will be competitive since they are at least 10 times more expensive.

4) Prior encapsulation of phase change material is a promising method for solving the problem of "microencapsulated" PCM storage. It also lends flexibility to the final system since the encapsulated PCM can be utilized in a number of different support matrices such as concrete or gypsum wallboard. Firms such as 3M or Pennwalt and laboratories such as SwRI or

Wurster (see body of this report) should be contracted for this task.

5) Macroencapsulation of phase change material in polymer concrete shells of various geometric configurations is another promising method. The PCM is added as a liquid and the problem of sufficient room for thermal cycling is thereby eliminated. Since PC is very strong (compressive strengths of 15,000-20,000 psi being typical), the walls can be fairly thin so that material costs can be reduced. The material has low water permeability and would serve as an excellent container for PCM. Samples made at the end of the program have not leaked to date.

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