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MACRO— ENCAPSULATION OF PCM

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**by
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MACRO-ENCAPSULATION OF HEAT STORAGE
PHASE-CHANGE MATERIALS
for Use in Residential Buildings

Third Quarterly Progress Report

March 29, 1977 - June 29, 1977

by

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16. Abstracts Objectives are to assess the feasibility of macro-encapsulated PCM's for residential solar systems, to develop and evaluate such materials. Encapsulant materials under consideration are multilayer flexible plastic films, steel cans, and plastic bottles. PCM's under study are $Mg(NO_3)_2 \cdot 6H_2O$, naphthalene-benzoic acid eutectic, $Mg(NO_3)_2 \cdot 6H_2O-NH_4NO_3$ eutectic, and $CaCl_2 \cdot 6H_2O$. Compatibility studies between the PCM's and encapsulant materials are continuing. A test device has been constructed, and is ready for use.					
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TABLE OF CONTENTS

	Page
Foreword	1
I. PHASE CHANGE MATERIALS	2
II. ENCAPSULATING MATERIALS.	5
III. MATERIALS COMPATIBILITY.	5
A. Film Evaluation.	5
B. Plastic Bottle Evaluation.	6
C. Steel Can Evaluation	7
D. Material Selection	8
IV. HEAT STORAGE TEST DEVICE	10
V. UTILIZATION ACTIVITY	17
VI. PLANS.	18
VII. CONCLUSIONS.	18
VIII. REPORT DISTRIBUTION LIST	19

FOREWORD

The work described in this report is an extension of a basic effort already performed under contract NSF-C906. The general objectives are: 1) to assess the technical and economic feasibility of encapsulated phase change materials (PCM's) for storing heat in residential solar energy systems, and 2) to develop and evaluate such encapsulated phase change materials.

The project involves three tasks:

Task 1 - Materials selection, including a limited literature search, selection of candidate phase change materials, and selection and characterization of encapsulating materials.

Task 2 - Procurement of phase-change and encapsulating materials, encapsulation studies, and testing of the encapsulated material.

Task 3 - Preliminary design and economic evaluation of a residence-sized heat storage sub-system.

Task 1 has been completed, and Task 2 efforts are in progress.

I. PHASE CHANGE MATERIALS

The PCM's under study are listed in Table I.

TABLE I
PHASE CHANGE MATERIALS
SELECTED FOR STUDY

Materials	m.p., °C	ΔH , cal./g	Cost, ¢/lb.
$Mg(NO_3)_2 \cdot 6H_2O$	89	38 (a)	15 (est.)
Naphthalene + Benzoic Acid	67	29 (b)	15
$Mg(NO_3)_2 \cdot 6H_2O + NH_4NO_3$	52	30 (b)	10
$CaCl_2 \cdot 6H_2O$	27	46 (c)	5

(a) Literature value

(b) New data, Dow Chemical Company

(c) Previously measured, Dow Chemical Company

In addition to the heat of fusion measurements on naphthalene + benzoic acid and $Mg(NO_3)_2 \cdot 6H_2O + NH_4NO_3$ reported above, a value of 26 cal./g was obtained for a sample of technical grade $Mg(NO_3)_2 \cdot 6H_2O$ prepared by Dow's Michigan Division laboratories. This is so much below the literature value for this compound, that the crude material will not be considered as a PCM. A purer "Commercial" grade material will be prepared, and heat of fusion measurements will be done on it, as well as on reagent grade material.

The heat capacities of both liquid and solid PCM's were measured. Data are shown in Figure 1, along with values previously obtained for $CaCl_2 \cdot 6H_2O$.

The thermal conductivities of the crystalline PCM's were also determined, as displayed in Figure 2. For comparison, the thermal conductivity of crystalline $CaCl_2 \cdot 6H_2O$ was previously found to be 26.0×10^{-4} cal. cm.⁻¹ sec.⁻¹ °C.⁻¹.

Figure 1

HEAT CAPACITY OF PCM'S

Figure 2

PCM Solid Phase Thermal Conductivity

II. ENCAPSULATING MATERIALS

The following packaging materials are being evaluated as encapsulants for these PCM's:

R-2 Retort Film, a laminate of 3 mil polyethylene, .35 mil Al foil, and .5 mil polyester.

Glamate GL3002, a laminate of aluminum foil between 2 films of polyethylene.

Glamate #309, similar, but using polypropylene films.

Plastic Bottle, 16 oz. high density polyethylene.

Plastic Bottle, as above, inside surface sulfonated.

Aerosol Can, 2.5 x 6.5 seamed steel.

Aerosol Can, 2.5 x 6.5 drawn steel.

The Glamate films, used for such applications as tooth-past tubes, are thicker than the R-2 Retort Film, and were added to our investigation because of the failure of Retort film packages above 70°C. Sulfonated plastic bottles were prepared by a gas-phase sulfonation process that makes them more resistant to chemical migration.

III. MATERIALS COMPATIBILITY

A. Film Evaluation

The compatibility of PCM's with Glamate films was tested by filling 3/4" x 5" (#309) or 1 11/16" x 6 1/2" (GL3002) cylinders with the molten PCM's, and storing them at a temperature 5-15°C above the melting point of the PCM. These films are lap-sealed, rather than butt-sealed as with the Retort film. The $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ tests are complete, but the other PCM exposures are still underway. Tensile test specimens were prepared and evaluated before exposure, after dry exposure for 2 weeks, and after exposure to the molten PCM (100°C) for 2 weeks. Failure under tensile test invariably occurred not at the seam, but in the film. The $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ - exposed samples failed by breaking cleanly, while in the other specimens the foil broke

first, followed by elongation and then rupture of the plastic. The force required at failure was 32.4 lb./in. for the unexposed GL3002 film, 37.6 lb./in. for unexposed #309 film, 43.2 lb./in. for dry-exposed #309 film, and 42.6 lb./in. for PCM-exposed #309 film. Exposure at 100°C embrittles the film, and molten $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ yellows and further embrittles it. We observed no attack on the seam, the weak point of Retort film capsules, however.

B. Plastic Bottle Evaluation

High density polyethylene bottles were found unsuitable for use with naphthalene-benzoic acid eutectic or stearic acid because of migration of the PCM into the plastic. With $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, embrittlement occurred. Sulfonated bottles were tested, as the surface layer of sulfonated plastic resists chemical migration. Compatibility was studied by cutting 4" long "dog bone" test bars 1/8" wide in the test area. These specimens were formed from the sides of the bottles and selected to be similar in thickness. Tensile strength was measured before exposure, after dry exposure to the test temperature, and after 4 weeks exposure at the test temperature filled with the liquid PCM. Results are outlined in Table II.

TABLE II
EVALUATION OF SULFONATED
PLASTIC BOTTLES WITH PCM'S

Materials	Storage Temp., °C	Tensile Strength Psi	Wt. Change, %
None	20	2610	0.00
None	85	2720	-0.09
Naphthalene + Benzoic Acid	85	2750	+0.09
Stearic Acid Mix	85	2750	+0.09

The sulfonated bottles showed negligible uptake of naphthalene-benzoic acid eutectic or stearic acid mixture, in contrast to weight gains of 5-13% observed for the unsulfonated bottles. In spite of the low uptake of PCM, a few tiny crystals of naphthalene were found on the outside of the bottle in an area where sulfonation apparently was spotty. These bottles should be satisfactory for encapsulation of the two organic PCM's.

C. Steel Can Evaluation

Through a computation error, the corrosion rates reported for steel cans in our previous report are erroneous. Corrected figures are presented, along with new data, in Table III.

TABLE III
EVALUATION OF STEEL CANS WITH PCM'S

Materials	Temp. °C	Corrosion Rate, lbs./ft. ² /day	
		Seamed Cans	Drawn Cans
Mg(NO ₃) ₂ ·6H ₂ O + NH ₄ NO ₃	70	9.1 x 10 ⁻⁶	6.6 x 10 ⁻⁶
CaCl ₂ ·6H ₂ O	50	1.2	1.0
CaCl ₂ ·6H ₂ O*	50	1.1	0.8
Mg(NO ₃) ₂ ·6H ₂ O	100	3.1	9.6
Mg(NO ₃) ₂ ·6H ₂ O**	100	17.5	16.5
Stearic acid	75	21.3	14.3
Naphthalene/benzoic acid	90	123.	113.

*0.2% corrosion inhibitor added.

**Technical grade material.

To obtain these data, 1" x 3" coupons were cut from the sides of seamed and drawn aerosol cans. No surface treatment, other than washing with acetone, was used. The coupons were immersed in molten PCM's for 31 days and the corrosion rate was determined by weight loss.

Either type of can is suitable for encapsulating calcium chloride hexahydrate, if air is excluded during filling. The corrosion rates for reagent grade magnesium nitrate hexahydrate and its eutectic with ammonium nitrate are higher, but should be acceptable for either type of can. The technical grade magnesium nitrate hexahydrate and the stearic acid mixture are marginal, and further tests should be run, if encapsulation in steel cans is to be recommended. The corrosion rate for the eutectic of naphthalene and benzoic acid is too high for this PCM to be used in steel cans. A corrosion rate of 15×10^{-6} lbs./ft.²/day is equal to about 0.1 mil/yr., or 2 mils in 20 years. The seamed aerosol can is 10 mil thick, and the drawn can 7.5 to 8.5 mil thick. In actual use, corrosion will be non-uniform, and further tests with cans filled with molten PCM's should be run.

Visual observation can be misleading in evaluating corrosion. For example, the naphthalene/benzoic acid samples remain bright and appear not to be corroding, though rapid, uniform corrosion is occurring. The coupons in $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ discolor and the reagent grade of material appears worse than the technical grade. Just the opposite is actually true.

D. Material Selection

Table IV summarizes the present status of the materials compatibility work. For $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ and the $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ - NH_4NO_3 eutectic, all the encapsulating materials appear usable. For naphthalene/benzoic acid and stearic acid, the sulfonated polyethylene bottle is the only fully satisfactory encapsulant. For magnesium nitrate hexahydrate, steel cans are the encapsulating material of choice.

TABLE IV

SUMMARY OF MATERIAL COMPATIBILITY WITH PCM'S

Encapsulant Material	PCM MATERIAL				
	CaCl ₂ ·6H ₂ O	Mg(NO ₃) ₂ ·6H ₂ O + NH ₄ NO ₃	Stearic Acid	Naphthalene + Benzoic Acid	Mg(NO ₃) ₂ ·6H ₂ O
Polyethylene Bottle	OK	OK	Weight Gain	Weight Gain	Embrittles
Sulfonated Polyethylene Bottle	-	-	OK	OK	-
R-2 Retort Film	OK	OK	Seam Leaks	Seam Migration	Seam Leaks
Alure LD Film	OK	OK	-	-	Seam Leaks
Alure MD Film	-	-	Seam Rupture	-	-
Glamine GL3002	-	-	-	-	-
Glamine #309	-	-	-	-	Marginal
Drawn Steel Can	OK	OK	Marginal	NG	OK*
Seamed Steel Can	OK	OK	Marginal	NG	OK*

*OK with reagent grade material, marginal with technical grade.

On the basis of the compatibility data, we have altered the tentative pairing of PCM's with encapsulant materials, as given in Table V. Calcium chloride hexahydrate in high density polyethylene bottles will be the first encapsulated PCM evaluated in the heat storage test device. Further tests are in progress with the Glamine films, and a choice will be made among the several films under study before the magnesium nitrate hexahydrate-ammonium nitrate eutectic is evaluated in the test device.

TABLE V

TENTATIVE SELECTION OF PCM-ENCAPSULANT PAIRS

Phase Change Material	Encapsulating Medium
$\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$	High Density Polyethylene Bottle
$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} / \text{NH}_4\text{NO}_3$ Eutectic	R-2 Retort Film
Naphthalene/Benzoic Acid Eutectic	Sulfonate Polyethylene Bottle
$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	Drawn Steel Aerosol Can

IV. HEAT STORAGE TEST DEVICE

The thermal energy storage (TES) test device is complete and ready to start testing macro-encapsulated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$. In shake-down runs using empty 16 oz. plastic bottles (see Figure 3), a constant air flow rate and inlet temperature were achieved at both the high and low set points. The time required to step from the low to the high set point is short (10 minutes), but the step from high to low set point is too slow (30 minutes). The step-down time will be improved by modifying the chiller circuit. Leakage measurements indicate that at 64 scfm total air flow thru the system there will be 1.5 scfm of leakage. As all or most of the leaks are thought to be in the air reconditioning unit, between the outlet temperature and flow rate measurement areas, the effect will be a proportional reduction (2.3%) in the measured capacity of the unit. Pressure drop across the storage unit was measured as a function of flow rate, as shown in Figure 4. One-half inch of water may be regarded as the practical upper limit for pressure drop across a TES unit.

In order to obtain a meaningful measurement of storage capacity, the procedure of ASHRAE 94-77 will have to be modified. It is misleading when applied to a latent heat TES unit of the type to be tested under this contract. Test time is defined in ASHRAE 94-77 as $\tau_D = \frac{T_{SC}}{C_p \dot{M} \Delta T_{IN}}$. This is the time for an ideal

Figure 3

Calcium Chloride Hexahydrate Storage Medium,
Air - Heat Transfer Fluid, Thermal Energy Storage Unit

Cabinet 1/8" Steel - 125 Kg

Containers .061M Dia .180 HT Bottles
Walls .74 mm HDPE

Layout .024M Spacing Triangular Pitch
Minimum Possible Void Fraction .876

Figure 4

Pressure Drop Across Storage
Vs. Flow Rate

plug flow sensible heat TES unit to discharge. The problem can be illustrated by comparing an ideal plug flow sensible heat device with an ideal latent heat device (all stored heat is latent and delivered at the freezing point). In evaluating a latent heat TES unit the high and low set points are chosen to bracket the freezing point of the storage material so the unit would discharge at one-half the initial step temperature change. As shown in Figure 5, for units with the same theoretical storage capacity (TSC), an ideal latent TES would take twice as long to discharge. Since no practical storage unit will behave in an ideal manner, it is even more difficult to justify this definition of test time (T_D).

The ASHRAE 94-77 definition of test time will be modified to give consideration to design rate (KJ/hr.). At a constant flow rate, the design rate can be used to determine a minimum useful temperature out of storage. This minimum useful temperature will be used as the criterion for terminating a test, and τ_D will be the time it takes to discharge the TES to the minimum useful temperature. In the recommended graphical presentation of a TES test the temperature axis is normalized by dividing ($T_{OUT} - T_{IN}$) by ΔT_{IN} . The time axis can also be made non-dimensional by dividing by the test time τ_D . Figure 6 shows the result of a computer simulation of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ TES unit discharge tests at three air flow rates. Lacking other criteria, the minimum discharge temperature was defined as $.25\Delta T_{IN}$. For the 75 scfm (153 kg/hr.) simulation, the design rate can be calculated from the equation:

$$Q_D = C_P \dot{M} \Delta T_{MIN} = 1350 \text{ KJ/hr.}$$

Capacity can be calculated from the integration:

$$C = \tau_D C_P \dot{M} \Delta T_{IN} \int_0^1 \frac{(T_{OUT} - T_{IN})}{\Delta T_{IN}} dt = 21,350 \text{ KJ}$$

Storage capacity in hours discharge at the design rate is then:

$$SC = C/Q_D = 16 \text{ hours}$$

This is the treatment that will be used in tests under this contract.

Figure 5

Comparison Of "Ideal" Storage Systems.
ASHRAE 94-77 Specifies Testing Until $t/\tau_D = 1$.
By This Test The Shaded Area Would Be Missed.

Figure 6

Computer Prediction of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ Test Unit
Performance At Different Flow Rates

SYMBOL DEFINITIONS

C = Practical Capacity of a storage unit - KJ.

C_p = Heat Capacity of the heat transfer fluid.
KJ/Kg°C

cfm = Air flow rate - cubic feet per minute.

$\dot{M}$ = Mass flow rate of heat transfer fluid - Kg/hr.

Q_D = Design rate - KJ/hr.

SC = Storage Capacity - hr.

TSC = Theoretical Storage Capacity - hr.

T_{IN} = Inlet Air temperature to storage unit - °C.

T_{OUT} = Outlet Air temperature from storage unit - °C.

ΔT_{IN} = Initial ($T_{OUT} - T_{IN}$) step change - °C.

ΔT_{MIN} = Minimum useful ($T_{OUT} - T_{IN}$) - °C.

t = Time - hr.

T_D = Test time in ASHRAE 94-77 - hr.

τ_D = Design time (time to discharge storage unit to ΔT_{MIN} at a given $\dot{M}$) - hr.

V. UTILIZATION ACTIVITY

Several possible applications for macro-encapsulated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ have surfaced. The freezing point, 27°C (80°F), is too low for standard home heating applications, but the material may be useful in grain drying, heating systems that use night-time set back, greenhouses, and air-to-air, water-to-air, and water-to-water heat pump systems, with or without solar collection.

We have discussed with Jay Kress, Indianapolis, a manufacturing representative for Solar, Inc., storage for a night-time set back system using macro-encapsulated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$.

H. M. Keener, Assistant Professor OARDC (Ohio Agricultural Research and Development Center), expressed interest in developing a computer model of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ TES for grain drying applications. He has been experimenting with low temperature solar grain drying for several years, and if the modeling results look good, full scale experiments might be undertaken.

Discussions were held with Dr. T. Short of OARDC, concerning testing $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ TES for solar heated greenhouse applications. Professor Aldrich of Penn State also expressed interest in $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ TES in greenhouses. We discussed the possible replacement of a rock pile in one of his solar heated greenhouses next summer.

We have also talked with Dr. M. W. Wildin, who plans to outfit a building at the University of New Mexico with an experimental solar assisted water-to-water heat pump system. Preliminary design will start this fall with final design next year. He is interested in including $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ TES in the preliminary design.

We are currently interacting with both Singer and American Air Filter concerning $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ TES for application with their water-to-air heat pumps.

The primary advantage of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ over water or rock bed storage is volume/weight reduction. If long period storage capacity is required, isothermal storage also would become an advantage.

VI. PLANS

The following work is planned for the next quarter:

1. Further materials compatibility tests will be completed.
2. Further tests will be run on potential hazards of nitrate materials.
3. Long-range melt-freeze cycling tests will continue.
4. Encapsulated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ will be evaluated in the heat storage test device.
5. Quantities of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} + \text{NH}_4\text{NO}_3$ will be prepared and encapsulated in plastic composite film.

VII. CONCLUSIONS

Compatible encapsulating materials are available for the PCM's being studied. Construction of the energy storage device is complete, and shake-down runs are under way.

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