MASTER

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CHEMICAL AND PHYSICAL STABILITY OF REFRACTORIES FOR USE IN COAL GASIFICATION

Thirteenth Quarterly Progress Report

May 1, 1979 - July 31, 1979

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DISCLAIMER

I. INTRODUCTION

The objectives of this investigation are: (1) to achieve an understanding of the general types of chemical reactions occurring in refractories when exposed to conditions representative of those at the cold face of the refractory lining in coal gasification vessels; (2) to assess the relative importance of these reactions to physical/chemical properties required for long-service life; and (3) to identify those refractory systems providing optimum service performance, particularly in regard to the bond phases.

The basic aim of the investigation is to evaluate the corrosion resistance of refractories, especially the bond phases, to those high pressure/temperature gases and liquids typically present in coal gasification environments.

The present work (1 May 1978 - 30 April 1980) is divided into four tasks given in Figure 1:

TASK I - Degree of Saturation

The aim of Task I is to determine the dependence of the chemical reactions occurring in dense and lightweight castables upon the degree of saturation of the atmosphere and to correlate these reactions to changes in physical and mechanical properties.

TASK II - Liquid versus Vapor Corrosion

The objective of Task II is to determine the corrosion resistance of refractory castables to gas-saturated liquids and vapors and to determine the relative severity of liquid versus vapor corrosion, particularly for lightweight insulating castables, which because of their high porosity, may be most susceptible to chemical attack.

TASK III - Pressure/Temperature Cycling

The aim of Task III is to determine the effect of repeated pressure/temperature cycling upon the mechanical strength of alumina castables containing boehmite; especially the effect upon the mechanical strength and structural integrity of a refractory in which boehmite is formed, but is then subsequently decomposed due to a change in temperature or gas composition. This task is completed and the results have been reported in the Third Annual Report.

TASK IV - Hydrothermal Reactions of CA Compounds

The objective of Task IV is to determine the time, temperature, and pressure dependence for the hydrothermal reactions occurring in calcium aluminate refractory cements and to investigate the reaction of CA, CA_2 and CA_3 individually in hydrothermal atmospheres relevant to coal gasification.

The present report summarizes the experimental and analytical work completed from May 1, 1979 to July 31, 1979 for Tasks I, II and IV.

II. WORK COMPLETED

A. TASK I - Degree of Saturation

A number of exposures in pure steam and other atmospheres are underway for Task I at pressures up to 1000 psia and for 10 days. Three are completed as shown in Table I. The data from Test #1 in pure steam at 410 psia was reported in the Third Annual report. This report describes the results of Test #2 performed in pure steam at 1000 psia. The purpose of these experiments is to find the minimum degree of saturation (or maximum temperature) at which boehmite will form.

A long rectangular bar (18 X 1/4 X 1/4") of neat CA-25 cement (water/cement ratio = 0.3) was used for this test. The chemical composition of

CA-25 cement is given in Table II. The experimental test procedures are given in the Eleventh Quarterly Progress Report. Steam was supplied from a stainless steel vessel in which the Task II experiment was in progress at 545°F (1000 psia). The degree of saturation was varied by imposing a temperature gradient along a horizontal vessel containing the neat cement bar, ~545°F for 100% saturation and ~900°F for minimum saturation.

Every 24 hours, steam was exhausted, condensed, and the PH of the liquid measured. The PH of the condensed water at the end of the first day was ~7. It then decreased gradually every day and reached to a minimum of 5.3 on the tenth day.

The 18" long rectangular specimen was broken in 3-point bending in an Instron machine, using a 2 inch span so that two fracture loads were measured for every inch corresponding to a different percent saturation. The MOR was calculated from the fracture loads and the average of two MOR values corresponding to different % saturation was calculated. The change in the average flexural strength with the degree of saturation is shown in Fig. 2. Those parts of the hydrated CA-25 cement bar where the steam (1000 psia) was >50% saturated were appreciably stronger than those parts exposed to <50% saturation.

The compounds present in the broken specimens as determined by XRD and TGA are given in Table III. As in the Test #1 exposure in 410 psia steam, the amount of boehmite varied with the percent saturation of the steam as shown in Fig. 3. A linear relationship exists between the percent saturation and the amount of boehmite below ~70% saturation. The minimum percent saturation required to form boehmite (in 10 days) at 1000 psia is ~20% which is slightly less than that at 410 psia (~30%). As shown in Table III, boehmite was not detected in sections of the bar which were above 860°F during exposure.

The XRD results in Table III also show that the amount of $C_4A_3H_3$ increases with increasing temperature (i.e., decreasing percent saturation). Calcite ($CaCO_3$) was also found as in the previous exposure, but the reason for its presence is unknown at this time. The atmosphere was presumably free of CO and CO_3. XRD of the unexposed sample of CA-25 cement showed no trace of $CaCO_3$.

B. TASK II - Liquid versus Vapor Corrosion

Similar to Task I, several 10-day exposures are being conducted to evaluate liquid versus vapor corrosion. The test atmosphere and temperature for the Task II experiments are given in Table I. The results of the first 10-day exposure in steam at 410 psia were described in the Third Annual Report. This report summarizes the data for the second exposure in 1000 psia steam/545°F.

The seven commercial and laboratory-prepared cement-bonded castables and three calcium aluminate cements (initially dry powders as well as hydrated neat cement cubes) listed in Table II are being investigated. The experimental procedures are the same as reported previously.

The PH of the water collected from the bottom of the vessel during exposure was ~11 on the first day and then decreased to ~6 where it remained constant. The initial PH of the distilled water used in this test was 7. The high initial PH and its subsequent decrease is consistent with the dissolution of some portion of the CaO from the cement-bond phases and its gradual precipitation as $CaCO_3$.

1. Cement-Bonded Castables

Table IV gives the property data for the castables after exposure to saturated steam (vapor and liquid) at 1000 psia. With the exception of the immersed UMR-1, whose weight increased slightly, and the immersed

UMR-4 whose weight was essentially constant, all castables showed weight losses after exposure. The dimensions of the all castables increased slightly during exposure. The weight and dimensional changes in 1000 psia steam are very similar to those in steam at 410 psia.

The formation of boehmite in the dense castables was confirmed by XRD (Table V) and measured by TGA (see Table VI). Larger amounts of boehmite were found in the dense high alumina castables than in the dense intermediate alumina castables. There was no significant difference in the quantity of boehmite formed in specimens exposed to vapor compared with those immersed in liquid. Calcite was also present in small quantities in all dense castables.

With the exception of Litecast 60-25, less boehmite was formed in the insulating castables compared with the dense castables. Overall, the immersed insulating castables contained more boehmite than those exposed to vapor.

Comparison of liquid versus vapor exposure, in terms of MOR, for various castables is given in Fig. 4. All the dense alumina castables had higher strength after exposure to either the saturated vapor or immersed in liquid, compared with their controls (fired at 500°F in air for 18 h). The large increase in MOR is attributed to the formation of additional bonding phase(s), such as boehmite. Unlike the previous results (410 psia steam), all the dense alumina castables immersed in liquid had either a higher or very similar MOR as compared to their specimens in vapor, Fig. 5. The difference in MOR, however, is not large and cannot be explained by the amount of boehmite formed since the TGA results in Table VI show the boehmite content is nearly the same for both. Dissolution of lime and/or formation of other compounds may contribute to these slight differences in MOR.

Comparing the flexural strength of dense castables for the effect of cements and aggregates, the castables containing CA-25 cement (UMR-1 and UMR-8) had generally higher strength than those containing refcon (UMR-4 and UMR-5), see Fig. 4. On the other hand the aggregates seem to have no significant effect on the flexural strength of dense castables, samples made with the same cement, but different aggregate having close to the same MOR.

The insulating castables, generally, showed no large change in MOR after exposure at 1000 psia. The same conclusion was obtained from the previous test at 410 psia.

2. Calcium Aluminate Cements

Table IV summarizes the weight and dimensional changes of hydrated cement cubes and the weight changes of initially dry cement powders. All hydrated and unhydrated cements showed weight increases after exposure compared with their dry (230°F) weights, the weight gain being larger for the cubes exposed to vapor than when immersed in water. Both weight and dimensional increases are expected due to formation of boehmite.

The XRD data (Table V) show that boehmite is the major component in both the hydrated and unhydrated cements after exposure. Except for the hydrated CA-25 cement which showed trace of α-Al_2O_3, no alumina was found after exposure. Essentially all of the alumina in the cements is converted to other compounds, mostly boehmite, during exposure at 1000 psia.

The TGA results showed higher weight losses for cement specimens exposed to vapor compared with those immersed in water. The data also indicated that the amount of boehmite formed in the hydrated cement specimens was more in vapor than in liquid.

C. TASK IV

During the last quarter, the analysis of the XRD and TGA data on CA, CA_2, Secar 250, and CA-25 essentially has been completed. The CA and CA_2 analyses are reported along with those of Secar 250 and CA-25 because both cements are reported by their respective manufacturers to contain CA as the major calcium aluminate phase. Secar 250 contains a minor amount of CA_2 whereas CA-25 contains a significant amount of α-alumina.

All of the samples run in the high pressure reactor were pelletized so that weight and dimensional changes could be evaluated. The pelletized samples were reacted in saturated steam at 100 psi, 500 psi, and 1000 psi for 5, 10, 20, and in some cases 60 hours. After steam exposure all samples were analyzed by XRD and TGA.

At 500 and 1000 psi, the principal reaction products for CA, CA_2, and Secar 250 are AH, C_3AH_6, and $C_4A_3H_3$. As shown in Fig. 6, these form a compatibility triangle. At 100 psi, both CA and CA_2 show an AH_3 phase in addition; however, the phase point for the systems still remains in the $AH-C_3AH_6-C_4A_3H_3$ compatibility triangle.

CA-25 contains AH, AH_3, C_3AH_6, and $C_4A_3H_3$ at 500 psi. At 1000 psi, C_3AH_6 is no longer observed.

Figure 6 shows the reaction points for CA and CA_2. Fig. 7 shows the reaction points for Secar 250 and CA-25.

The data indicate that initially the samples appear to hydrate to a point near the $AH-C_3AH_6$ join, then as time at pressure increases, the samples react to form products on the $AH-C_4A_3H_3$ join. This causes the phase point to move away from the water component.

It is proposed that the following equations reflect the initial stages of hydration of CA and CA_2 respectively:

$3CA + 8H = C_3AH_6 + 2AH$ and

$3CA_2 + 11H = C_3AH_6 + 5AH.$

As the samples are exposed to the high pressure steam for longer time periods, a reaction occurs that yields $C_4A_3H_3$. This phase could be formed directly from CA or CA_2; however, because C_3AH_6 begins to disappear, it is assumed that C_3AH_6 is undergoing a dehydration type reaction to form $C_4A_3H_3$. Two possible reactions are possible in this system;

$4C_3AH_6 + 5AH = 3C_4A_3H_3 + 20H$ or

$3C_3AH_6 = C_4A_3H_3 + 5CH + 10H.$

Figure 8 shows the trends for AH, C_3AH_6, and $C_4A_3H_3$ for various pressures as a function of time, using CA as a starting material. The 1000 psi data best shows the increase in $C_4A_3H_3$ associated with a decrease in AH and C_3AH_6 content.

None of the samples analyzed by XRD or TGA showed any indication of CH. Usually when CH is present in a sample, it can be determined by allowing the sample from the completed TGA run to be exposed to the atmosphere overnight. It has been observed that any free calcia in the sample hydrates readily and a second TGA run shows only a CH decomposition.

Therefore, it appears that the data are best explained by assuming that CA and CA_2 hydrate to AH and C_3AH_6. On continued exposure to high pressure steam, the C_3AH_6 reacts with some of the AH forming $C_4A_3H_3$.

During the hydration-dehydration reactions, significant changes in the volume occupied by the various phases occurs. Using the measured dimensions of the pellets before and after reaction along with the quantitative phase analysis obtained from the TGA, it is possible to calculate sample porosities before and after reaction. The results are presented in Table VII along with the calculated volume changes of the materials and the actual pellet volume changes.

The data indicate that the porosity of the pellets decreases significantly with time at 500 psi. The hydration products must fill voids in the sample. How far this reduction in porosity will proceed was not determined since runs only extended to 20 hours. However Fig. 9 shows the trends for CA, CA_2, Secar 250, and CA-25. The reversal of the trend to reduced porosity in Secar 250 can be observed, but additional data will be needed to confirm the results.

It should be noted that there is a significant increase in the volume of material as the reactions proceed. If very dense samples are prepared, it can be anticipated that large stresses will be built up in the cast pieces as the hydration-dehydration reactions proceed.

III. FUTURE WORK

TASK I - Degree of Saturation

The following experiments are planned for Task I:

1. In the past experiment at 1000 psia steam, the temperature gradient between 50 to 100% saturation occurred within a short length and, therefore, only limited data could be obtained for this interesting region. A second experiment is planned using 1000 psia steam with the temperature gradient for 40-100% saturation covering the entire length (~18 inches) of the specimen. 2. Exposure tests in DOE atmosphere - The first exposure in the DOE atmosphere at 1000 psia was recently completed. From the results of the past two tests in steam at 410 and 1000 psia and the data from the recent test in the DOE atmosphere, the test conditions for future experiments will be decided.

TASK II - Liquid versus Vapor Corrosion

Work will continue along with that for Task I since the same test atmosphere is being used for Task I and II. Furthermore, preliminary experiments of a scouting nature are planned for Task II in coal liquifaction which will be started in September, 1979.

TASK III - Pressure/Temperature Cycling

Work on this task is completed and no further work is planned.

TASK IV - Hydrothermal Reactions of CA Compounds

Only a small amount of further data analysis is required for the samples exposed to saturated steam at 1000 psi and this work will be completed during the next quarter.

An investigation of the reaction of the CA compounds in the DOE atmosphere is proceeding. The exposure should be completed during the next quarter.

IV. PERSONNEL

During this quarter the following personnel worked on this project:

1. Delbert E. Day, Principal Investigator
2. Gordon Lewis, Co. Investigator
3. Abbas Fakhr, Postdoctoral Fellow (Full time)
4. Syed Rahman, Research Assistant Professor
5. Robert D. Karp; Student Technician (Part time)

Table I - Tests Conditions and Status for Task I

Test #	Atmosphere	Temp. (°F)	Pressure (psia)	Duration (Days)	Status
1	steam	447	410	10	complete
2	steam	545	1000	10	Complete, temp. gradient 540-900°F
3	DOE*	447	1000	10	Exposure complete, sample characterization in progress
4	steam	545	1000	10	Starts in August 1979, temp. gradient 540-650°F

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* DOE composition is 18 CO, 12 CO_2, 5 CH_4, 24 H_2, 41 H_2O (Vol %) plus 1% H_2S

Table II. Refractory Materials Used in Task II Exposure Test

Trade Name or Identification	Manufacturer	Composition and Remarks
Dense, High Alumina Castables		
UMR-1	Univ. of Missouri	93.4% Al_2O_3, 5.4% CaO (70% Tabular Al_2O_3* + 30% CA-25 Cement**)
UMR-4	Univ. of Missouri	87.4% Al_2O_3, 10% CaO, 1.7% SiO_2 (70% Tabular Al_2O_3* + 30% Refcon Cement**)
Dense, Intermediate Alumina		
UMR-8	Univ. of Missouri	63.8% Al_2O_3, 4.8% CaO, 28.3% SiO_2 (75% Calcined Kaolin Aggregate*** + 25% CA-25 Cement**)
UMR-5	Univ. of Missouri	58.6% Al_2O_3, 8.6% CaO, 29.6% SiO_2 (75% Calcined Kaolin Aggregate*** + 25% Refcon Cement**)
Insulating Castables		
Cerlite #75	C. E. Refractories	53.8% Al_2O_3, 2.9% CaO, 40.5% SiO_2
Litecast 60-25	General Refract.	46.7% Al_2O_3, 9.1% CaO, 40.2% SiO_2
VSL-50	A. P. Green Ref.	34.5% Al_2O_3, 9.3% CaO, 52.5% SiO_2
Calcium Aluminate Cements		
CA-25	ALCOA	79% Al_2O_3, 18% CaO, 0.2% SiO_2
Secar 250	Lone-Star Lafarge	72% Al_2O_3, 26% CaO, 0.2% SiO_2
Refcon	Universal Atlas	58% Al_2O_3, 33.5% CaO, 5.6% SiO_2

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*Aluminum Company of America, Bauxite, Arkansas. Tabular alumina T-61, used as grog in the high alumina castables, contained the following size fraction (wt%):

-8 to 14 mesh = 20%
-14 to 28 mesh = 18%
-28 to 48 mesh = 16%
-48 mesh = 46%

**Information on various cements appears elsewhere in this Table.

***C. E. Minerals, King of Prussia, Pennsylvania. Mulcoa 60 (4 and 8 mesh sizes) and Mulgrain M47 (20 mesh size) calcined Kaolin aggregate used as grog contained the following size fraction (wt%):

-4 to 8 mesh = 27%
-8 to 20 mesh = 36%
-20 to 60 mesh = 13%
-60 mesh = 24%

Table III. MOR and Compounds Present in CA-25 hydrated cement exposed to pure steam at 1000 psia and various degrees of saturation (TASK I).

Sample No.	Temp °F	% Saturation	MOR (PSI)	DTA/TGA DATA			X-RAY DATA Compounds*			
				% W. L.	% AH	% $CaCO_3$				
1	500	100	6670	17.73	55	8.58	AH	Calcite	$C_4A_3H_3$	α-Al_2O_3 (tr)
2	540	100	6828	16.8	53	8.13	AH	Calcite	$C_4A_3H_3$	α-Al_2O_3
3	635	50	5287	15.33	38	13.22	AH	Calcite	α-Al_2O_3	$C_4A_3H_3$ (tr)
4	645	47	1860	13.82	15.15	22.76	AH	Calcite	α-Al_2O_3	$C_4A_3H_3$ (tr)
5	730	27	1176	11.87	3.66	22.8	Calcite	α-Al_2O_3	AH	$C_4A_3H_3$ (tr)
6	765	22	1185	9.75	1.85	15.50	α-Al_2O_3	Calcite	$C_4A_3H_3$	AH (tr)
7	825	16.9	1240	8.02	0.58	5.8	α-Al_2O_3	$C_4A_3H_3$	Calcite	AH (tr)
8	845	14.9	1227	7.9	0.4	5.3	α-Al_2O_3	$C_4A_3H_3$	Calcite	AH (tr)
9	860	13.9	1565	7.68	0	4.66	α-Al_2O_3	$C_4A_3H_3$	Calcite	---------
10	870	13.3	1320	7	0	4.5	α-Al_2O_3	Calcite	$C_4A_3H_3$	---------

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* In decreasing order of their abundance; (tr) represents trace amount.

Table IV. Properties of castables and pure cements after exposure to saturated steam at 1000 psia/545°F, Task II.

Trade name or identification	% Weight Change		% Dimensional change		MOR (PSI)		Density (gm/cc)		% Porosity	
	vapor	liquid (immersed)	vapor	liquid (immersed)	vapor	liquid (immersed)	vapor	liquid (immersed)	vapor	liquid (immersed)
Dense High Alumina										
UMR-1 (Tabular + CA-25)	-0.75	+0.81	+0.32	+0.12	3500±215	4895±240	2.70	2.67	16.53	10.16
UMR-4 (Tabular + Refcon)	-1.01	+0.01	+0.16	+0.14	2145±145	2515±165	2.57	2.54	21.80	14.57
Dense, Intermediate Alumina										
UMR-8 (Kaolin + CA-25)	-1.82	-2.18	+0.1	+0.1	4365±270	4180±820	2.37	2.36	12.37	13.17
UMR-5 (Kaolin + Refcon)	-1.58	-1.07	+0.2	+0.17	2535±290	3210±345	2.39	2.36	16.02	15.02
Insulating Castables										
Cerlite #75	-1.85	-3.67	+0.72	+1.23	510±50	485±100	1.49	1.46	51.28	53.14
Litecast 60-25	-3.88	-3.2	+0.6	+0.21	540±25	660±100	1.16	1.18	56.11	55.70
VSL-50	-9.05	-9.28	+0.53	+0.46	95±35	190±20	0.88	0.95	65.26	63.95

Table IV. Continued

Trade name or identification	% Weight Change		% Dimensional Change		MOR (PSI)		Density (gm/cc)		% Porosity	
	vapor	liquid (immersed)	vapor	liquid (immersed)	vapor	liquid (immersed)	vapor	liquid (immersed)	vapor	liquid (immersed)
<u>Hydrated Cements</u>										
CA-25	+5.66	+3.82	+0.38	+0.77	Not Measured		Not measured		Not measured	
Secar 250	+7.27	+4.91	+0.28	+0.49	Not Measured		Not measured		Not measured	
Refcon	+2.92	+2.73	+0.24	+0.77	Not Measured		Not measured		Not measured	
<u>Unhydrated Cements</u>										
CA-25	+9.7									
Secar 250	+21.3									
Refcon										

+ represents weight or dimensional gain.

- represents weight or dimensional loss.

± represents standard deviation.

Table V. X-ray data for castables and cements after exposure at 1000 psia pure steam for 10 days (Task II, Liquid vs. Vapor).

Trade name or Identification	VAPOR Compounds*				LIQUID Compounds*				
Dense High Alumina									
UMR-1	α-$\mathrm{Al_2O_3}$	AH	Calcite		α-$\mathrm{Al_2O_3}$	AH	Calcite		
UMR-4	α-$\mathrm{Al_2O_3}$	AH	Calcite		α-$\mathrm{Al_2O_3}$	AH	Calcite(tr)		
Dense Intermediate Alumina									
UMR-8	$\mathrm{A_3S_2}$	α-$\mathrm{Al_2O_3}$	AH	Calcite	$\mathrm{A_3S_2}$	α-$\mathrm{Al_2O_3}$	AH	Calcite	
UMR-5	$\mathrm{A_3S_2}$	AH	$\mathrm{C_2AS}$(tr)	Calcite(t)	$\mathrm{A_3S_2}$	AH	$\mathrm{C_2AS}$	Calcite(tr)	$\mathrm{C_4A_3H_3}$(tr)?
Insulating Castables									
Cerlite #75	α-Quartz	α-$\mathrm{Al_2O_3}$	AS		α-Quartz	AS	α-$\mathrm{Al_2O_3}$	$\mathrm{CAS_4H_2}$(tr)	
Litecast 60-25	α-Quartz	AS	AH		α-Quartz	AS	$\mathrm{CAS_4H_2}$	AH(tr)	
VSL-50	α-Quartz	$\mathrm{CAS_4H_2}$	α-$\mathrm{Al_2O_3}$	Bayerite	α-Quartz	$\mathrm{CAS_4H_2}$	α-$\mathrm{Al_2O_3}$	Bayerite	AH(tr)
Hydrated pure Cements									
CA-25	AH Calcite	$\mathrm{C_3AH_6}$	α-$\mathrm{Al_2O_3}$	$\mathrm{C_4A_3H_3}$(tr)	AH	Calcite	316	α-$\mathrm{Al_2O_3}$	$\mathrm{C_4A_3H_3}$
Secar	AH	Calcite	$\mathrm{C_3AH_6}$		AH	Calcite	$\mathrm{C_3AH_6}$		
Refcon	AH	$\mathrm{C_3AH_6}$	Calcite		AH	$\mathrm{C_3AH_6}$	Calcite	$\mathrm{CAS_4H_2}$(tr)?	

Table V. Continued.

Trade Name or Identification	VAPOR Compounds*	LIQUID Compounds*
Unhydrated Cements		
CA-25	AH Calcite C_3AH_6(tr) $C_4A_3H_3$(tr)	
Secar 250	AH Calcite $C_4A_3H_3$ C_3AH_6	
Refcon	AH C_3AH_6 Calcite	

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* In the decreasing order of their abundance.

(tr) represents trace amount.

? represents possible presence.

Table VI. % Weight lost, boehmite and calcite for castables and cements after exposure at 1000 psia pure steam for 10 days (Task II) as determined by TGA.

Trade Name or Identification	Vapor % W.L.	Vapor % AH	Vapor % $CaCO_3$	Liquid % W.L.	Liquid % AH	Liquid % $CaCO_3$
<u>Dense High Alumina</u>						
UMR-1	6.2	22.5	5.2	6.8	23.5	2.3
UMR-4	7.1	20.0	5.7	7.0	18.3	3.9
<u>Dense Intermediate Alumina</u>						
UMR-5	5.2	11.1	3.2	4.6	10.3	3.0
UMR-8	3.7	11.4	1.7	3.4	10.8	1.5
<u>Insulating Castables</u>						
Cerlite #75	3.8	3.6	---	6.2	6.1	---
Litecast 60-25	6.2	12.8	---	5.5	13.0	---
VSL-50	4.0	8.2	---	4.7	9.5	---
<u>Hydrated Cements</u>						
CA-25	19.34	67.4	9.4	17.12	59.3	6.8
Secar 250	22.6	57.3	15.7	20.9	53.9	11.7
Refcon	18.8	43.9	6.6	17.6	40.6	5.4
<u>Unhydrated Cements</u>						
CA-25	17.8	63	12.9			
Secar 250	19.2	43	9.3			
Refcon						

Table VII. Porosity and Volume Expansion of Samples After Exposure to Saturated Steam at 500 psi.

500 psi

	Time (hrs.)	%Porosity before reaction	%Porosity after reaction	Calculated %Volume expansion (materials)	Measured %Volume expansion (pellet)
CA	5	33.0	22.7	23.7	7.1
	10	31.7	17.4	30.0	4.2
	20	34.7	15.7	36.2	5.6
CA_2	5	31.2	23.0	19.1	6.4
	10	34.9	23.3	22.3	3.8
	20	31.6	19.4	23.6	5.0
Secar 250	5	30.8	13.5	22.5	3.1
	10	33.7	12.0	36.0	2.4
	20	31.5	13.0	30.7	3.0
CA-25	5	37.0	26.4	23.0*	5.2
	10	39.8	23.9	28.0*	1.2
	20	36.8	20.1	31.4*	4.0

*Incomplete reaction

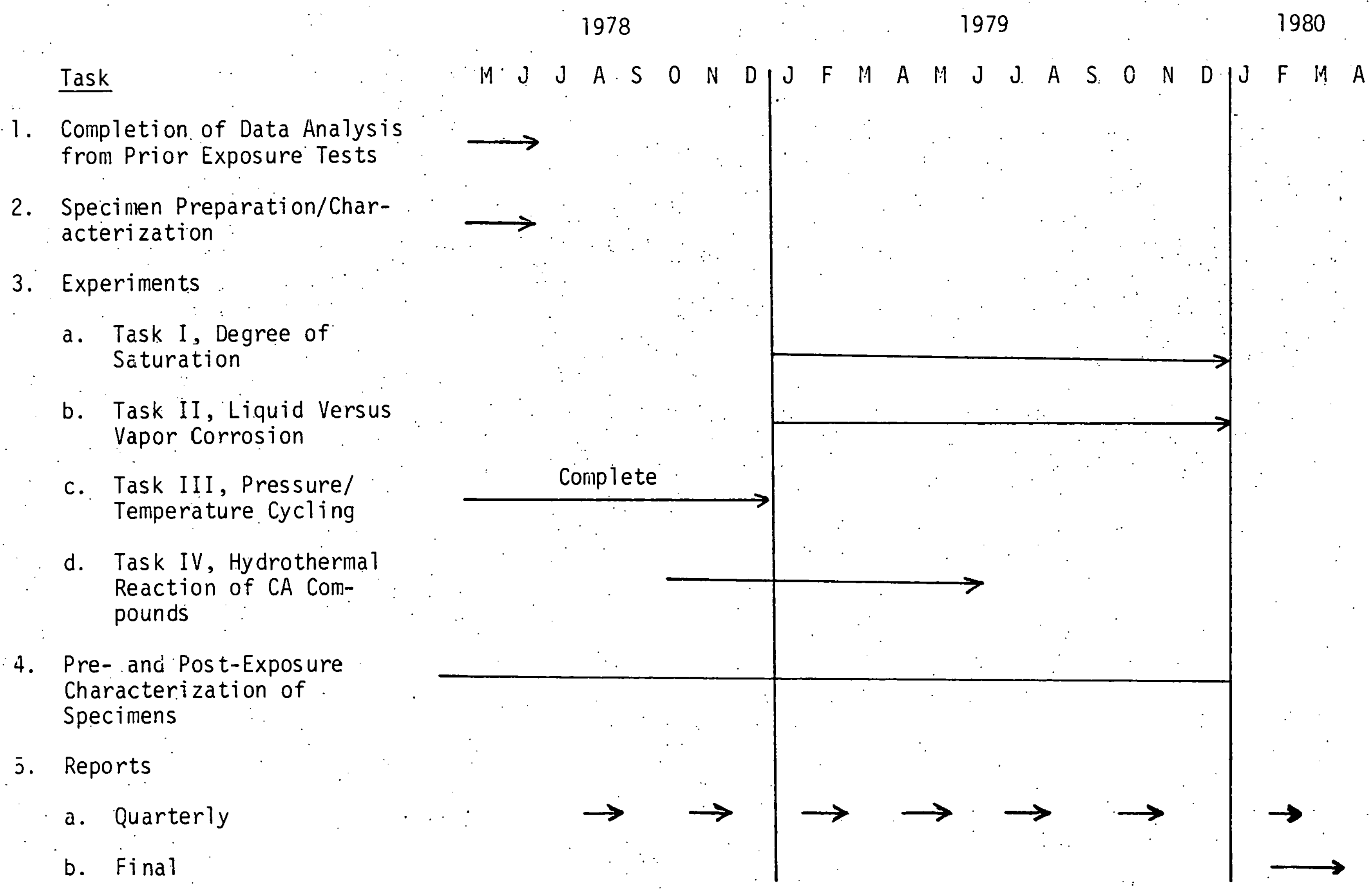


Figure 1. Work Schedule, 1 May 1978 to 30 April 1980

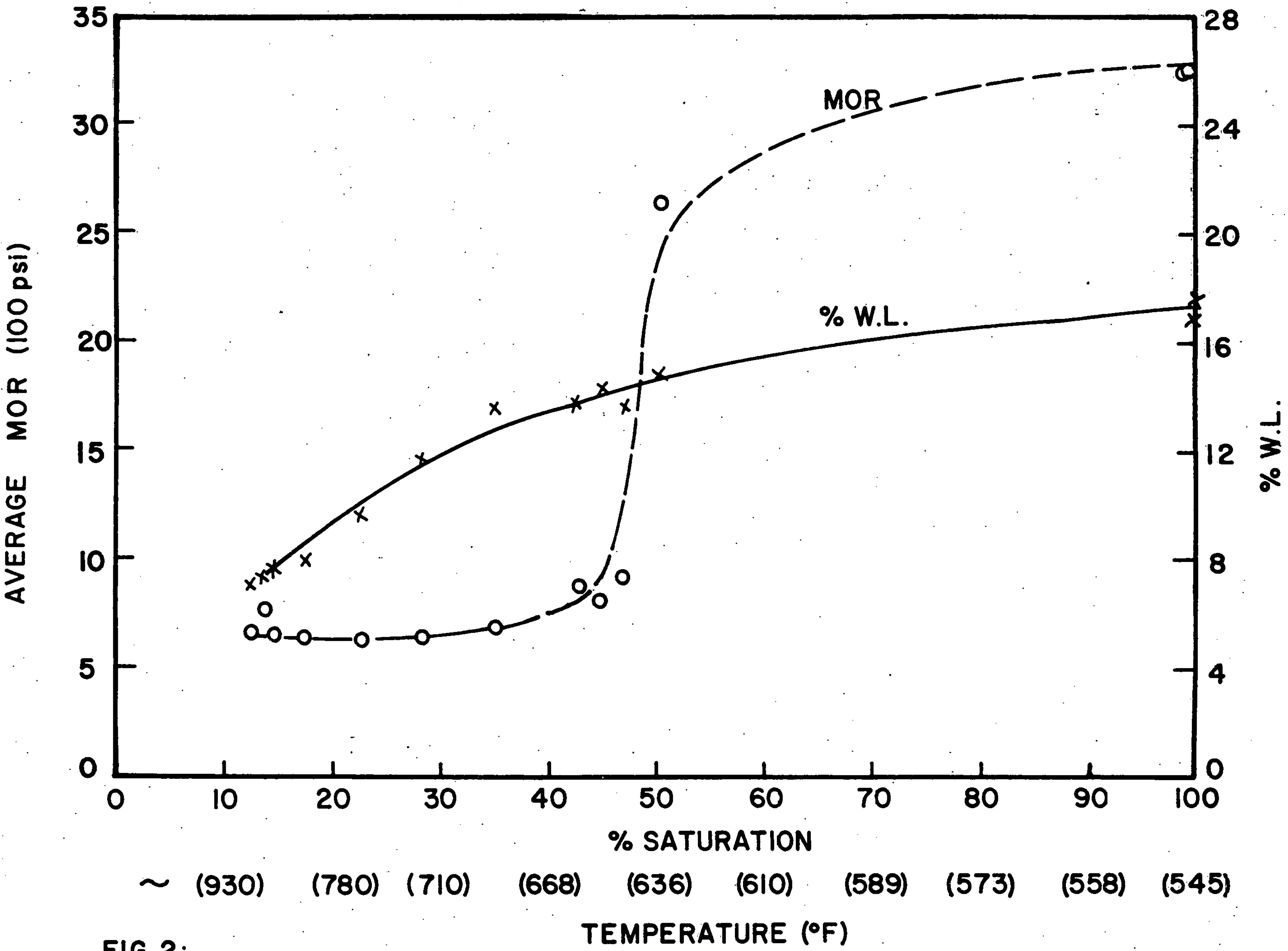


FIG. 2:

VARIATION OF MOR AND % WEIGHT LOST OF HYDRATED CA-25 CEMENT EXPOSED AT 1000 psia PURE STEAM WITH THE DEGREE OF SATURATION

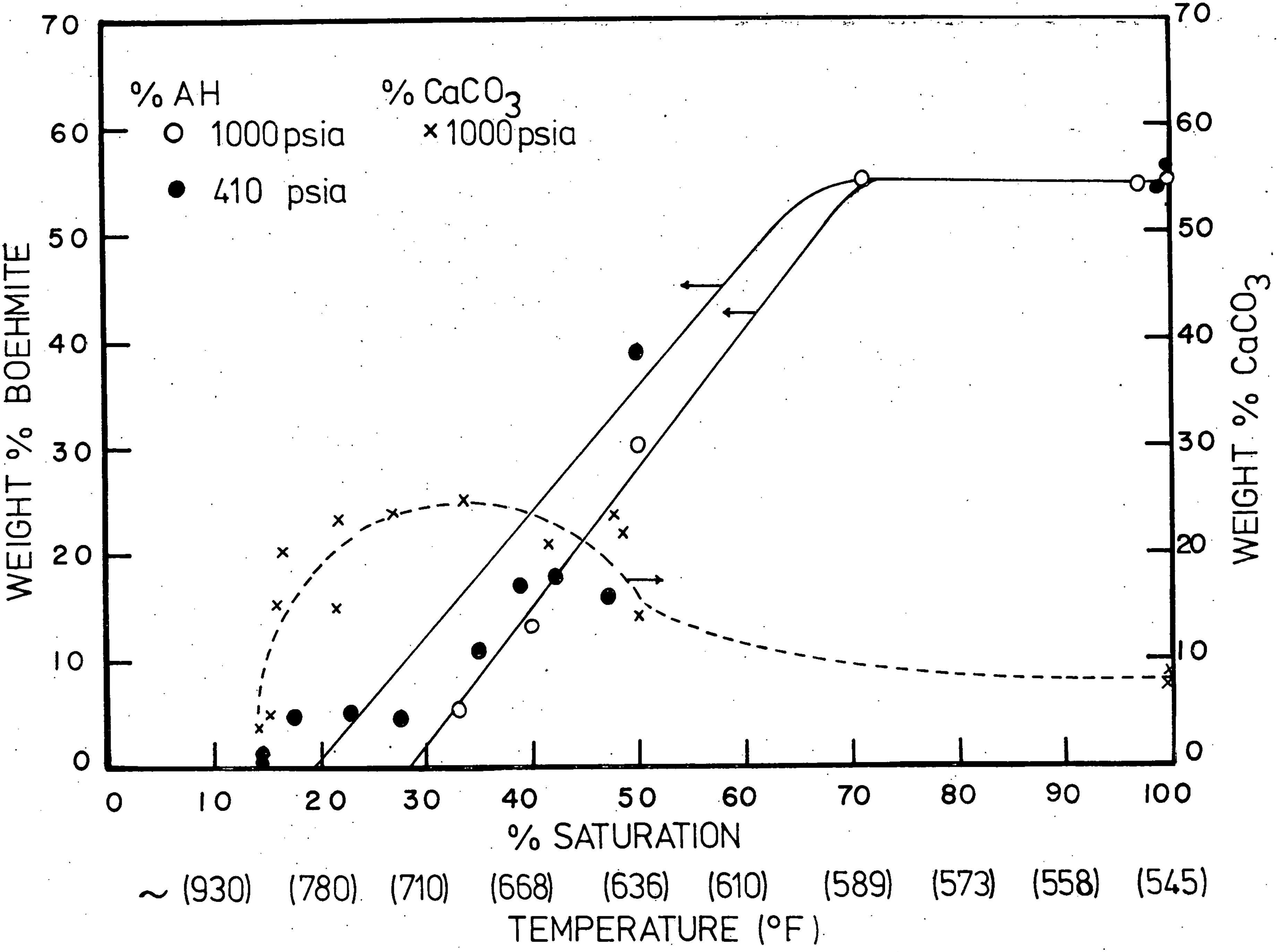


Fig. 3. Weight % boehmite and CaCO$_3$ as a function of the degree of saturation for CA-25 exposed to pure steam for 10 days.

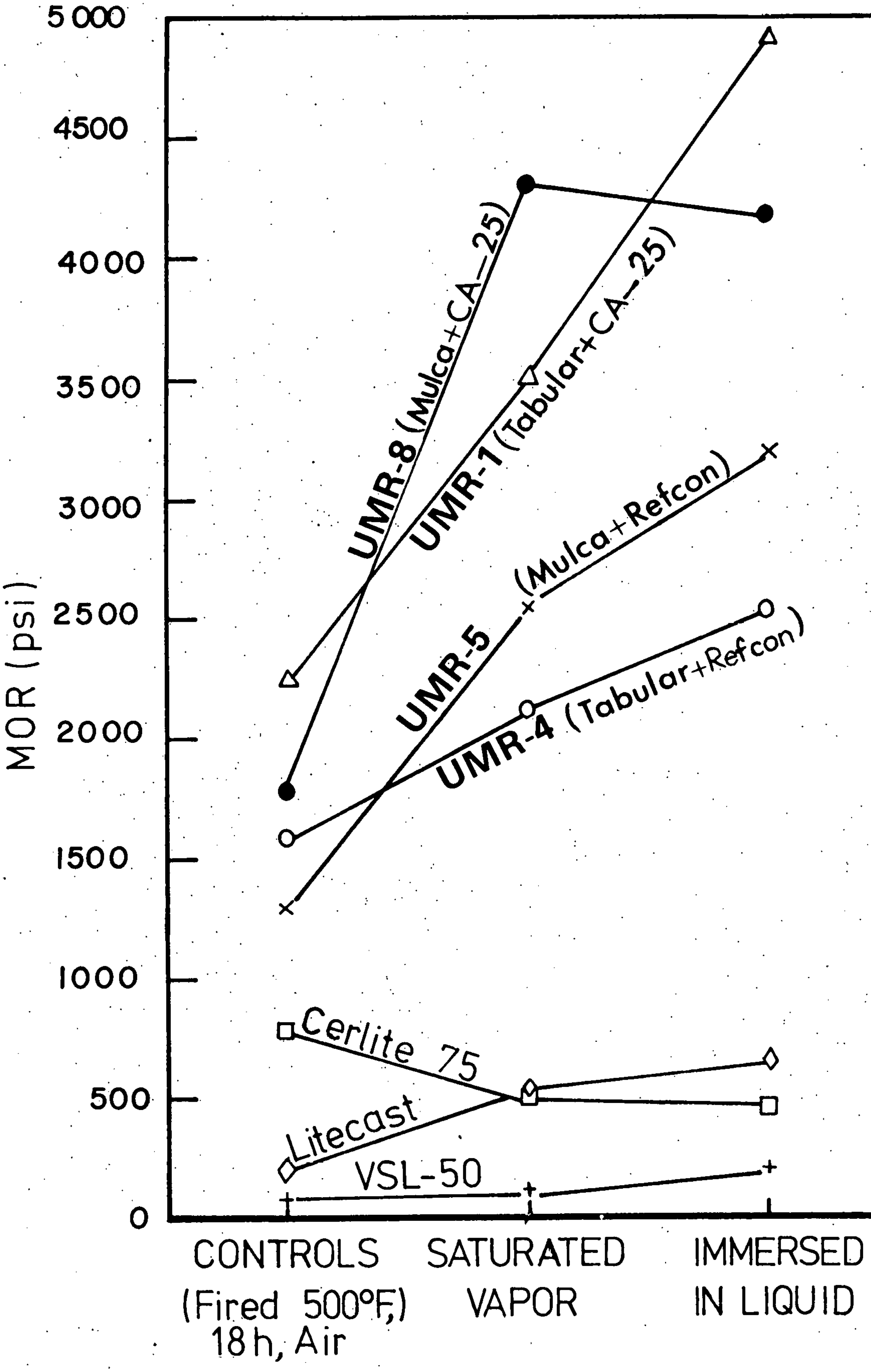


Fig. 4. Room Temperature Modulus of Rupture of Castables After 10-Day Exposure to Saturated Steam, 1000 psia/545°F.

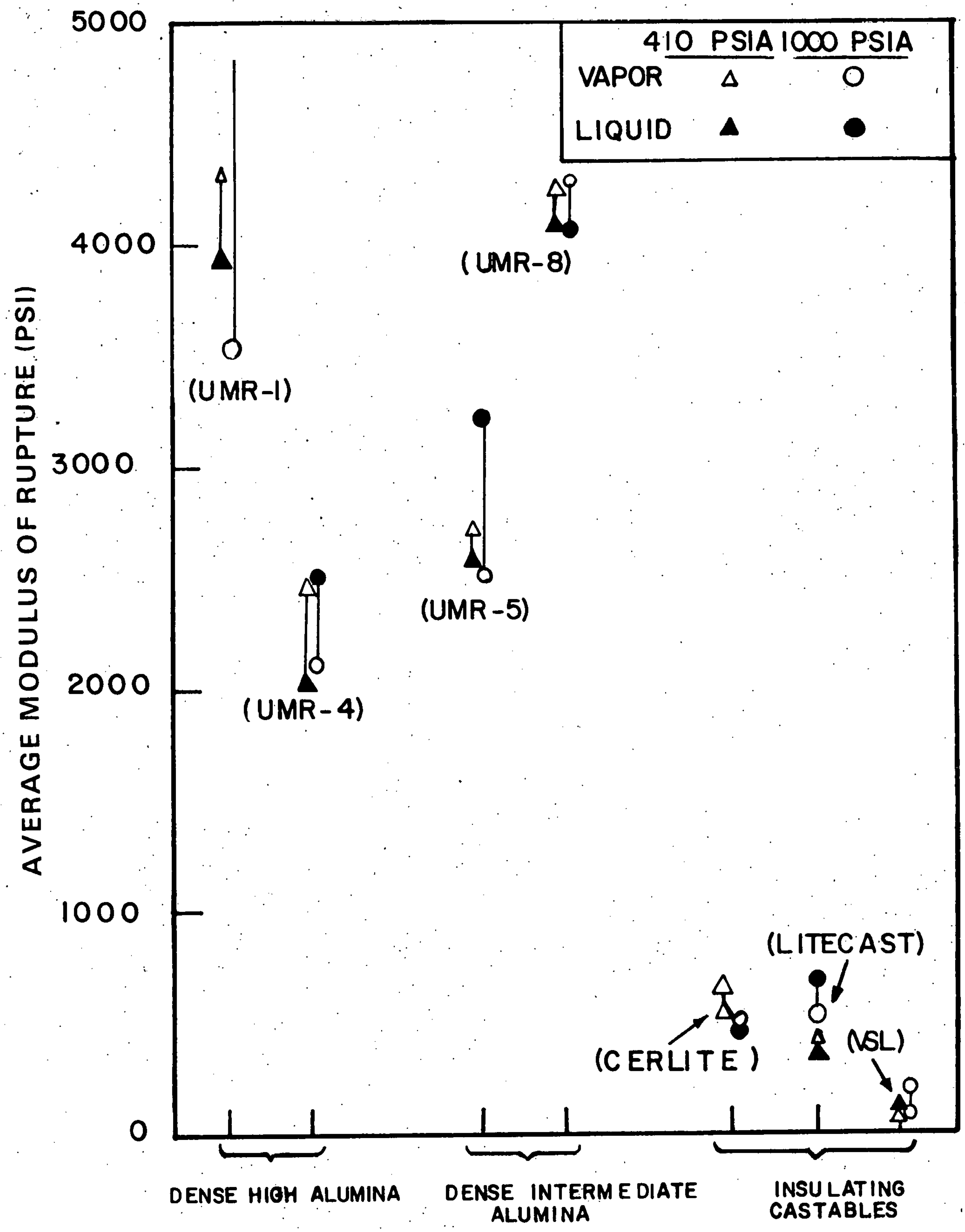


FIG. 5:
COMPARISON OF THE MOR OF CASTABLES EXPOSED IN LIQUID AND VAPOR AT 1000 PSIA/545°F WITH THOSE EXPOSED AT 410 PSIA/447°F PURE STEAM FOR 10 DAYS.

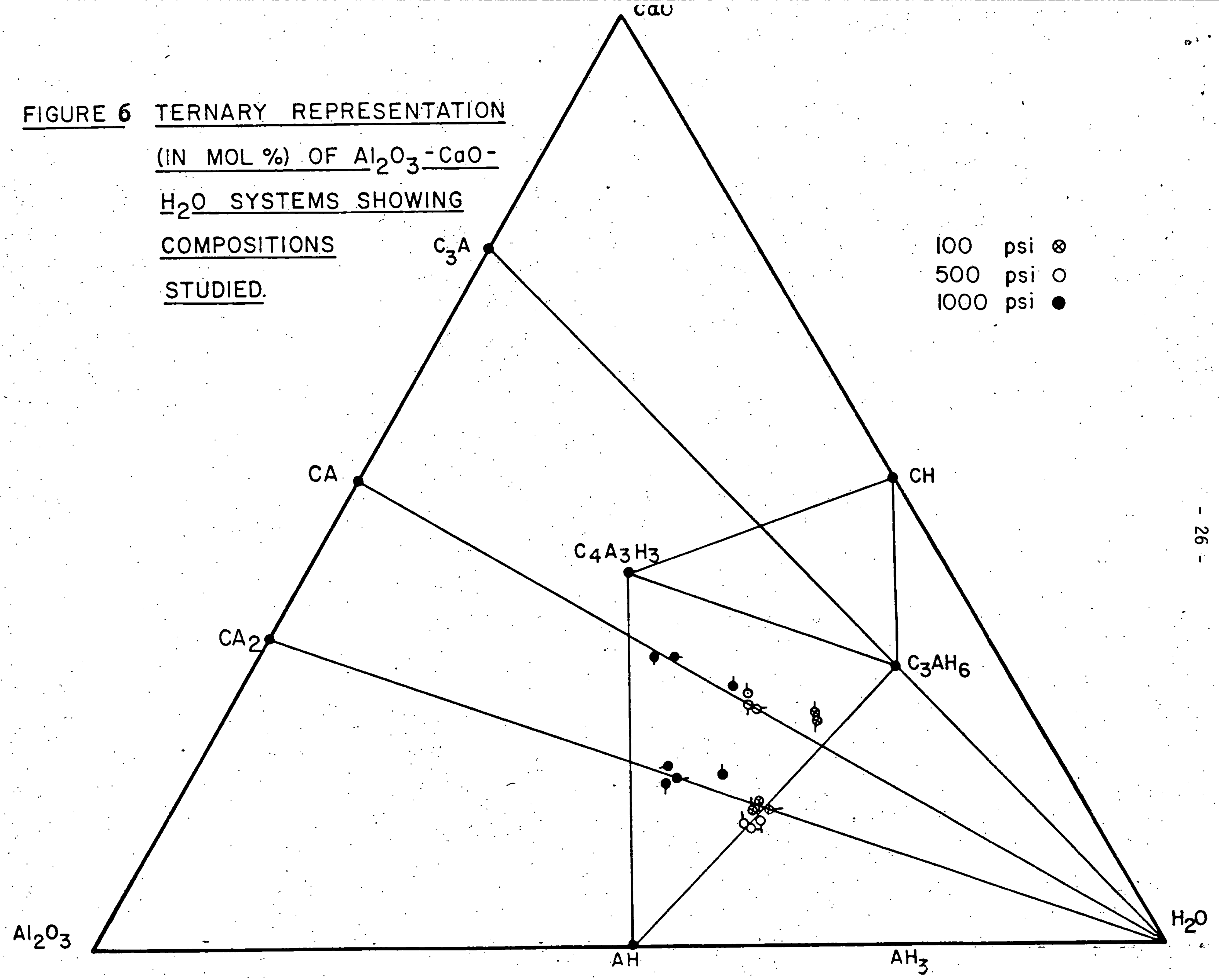
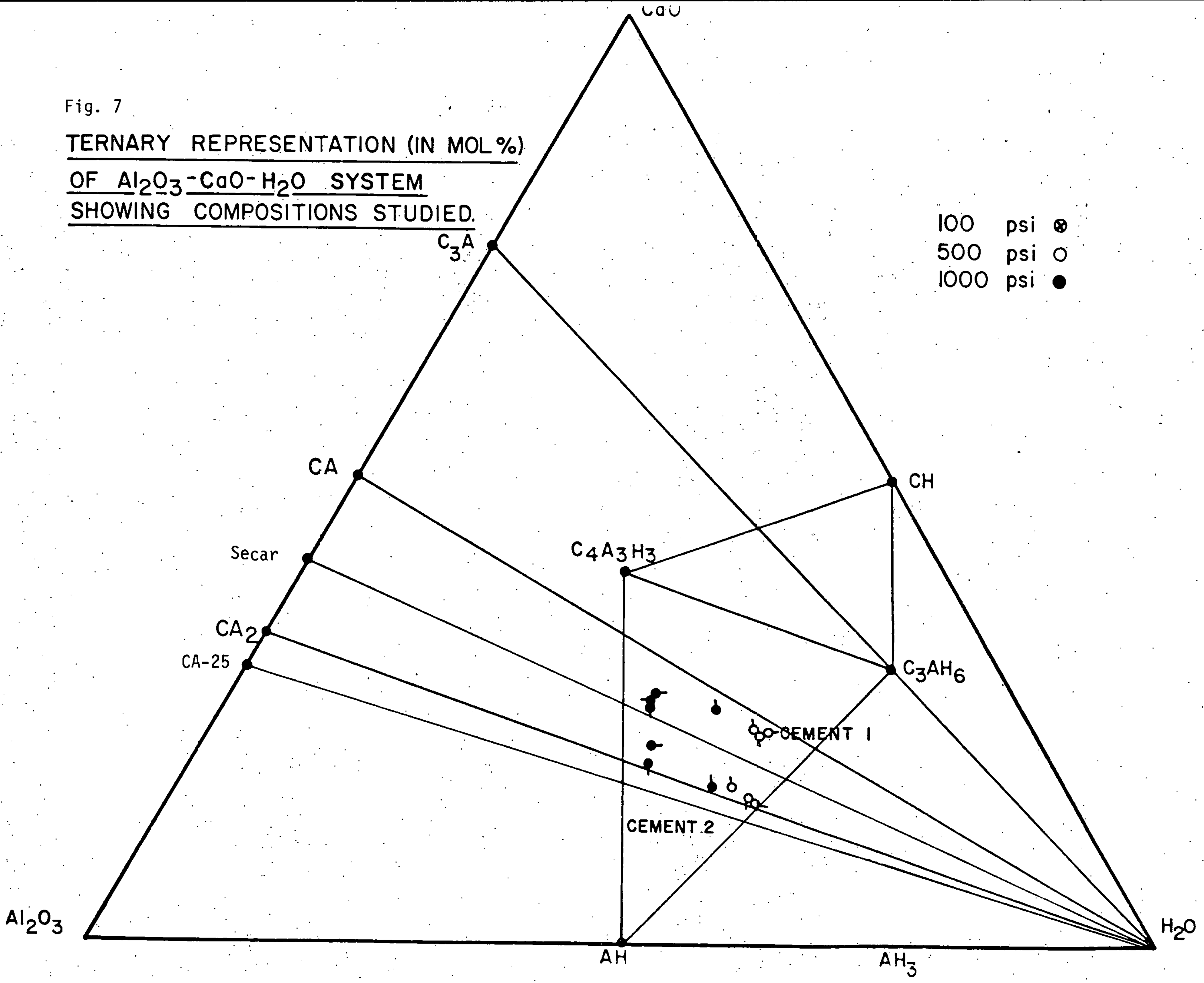


FIGURE 6 TERNARY REPRESENTATION (IN MOL %) OF Al_2O_3-CaO-H_2O SYSTEMS SHOWING COMPOSITIONS STUDIED.

Fig. 7

TERNARY REPRESENTATION (IN MOL %) OF Al_2O_3-CaO-H_2O SYSTEM SHOWING COMPOSITIONS STUDIED.



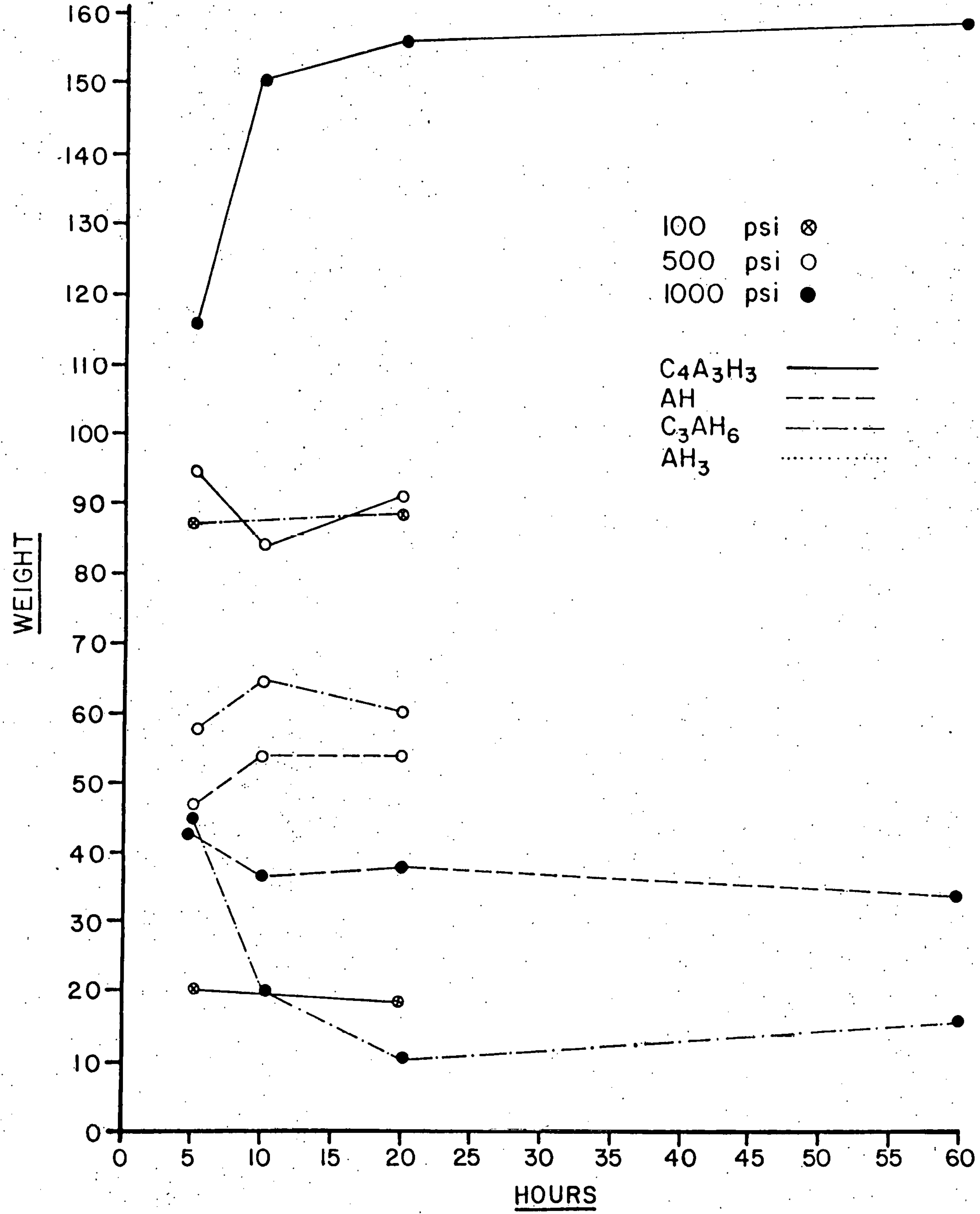


FIGURE 8: TIME RELATIONSHIP AS A FUNCTION OF PRESSURE FOR CA

FIGURE 9: Relative Porosity Changes at 500 psi