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STUDY OF REGENERABLE CO_2 SORBENTS FOR EXTRA VEHICULAR ACTIVITY

by
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STUDY OF REGENERABLE CO_2 SORBENTS FOR EXTRAVEHICULAR ACTIVITY

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Abstract

Studies have shown that frequent extravehicular activities planned for future space missions will require regenerable life support systems. The oxides of magnesium, zinc, and silver were tested for their ability to react with CO_2 to form the corresponding carbonates, and subsequent thermal regeneration to the oxides. Catalysts and binders were investigated to enhance CO_2 sorption rates and structural integrity. A silver oxide formulation was developed which rapidly absorbs 95% of its theoretical capacity (0.19#CO_2/# oxide) and has shown no degradation through 28 regenerations.

Introduction

The objective of this program was to select the most promising candidate material for development as a regenerable CO_2 sorbent for EVA. This was accomplished by manufacturing pellets from the candidate materials containing various additives and evaluating their CO_2 sorption capacities under fixed conditions. Detailed chemical and physical analyses of the most promising candidates were conducted in an effort to characterize properties which were conducive to desirable performance. Studies of the regeneration processes were also conducted.

Theoretical Considerations

The NASA-sponsored AEPS studies (References 1 and 2) not only identified the requirements for regenerable CO_2 sorption materials but also identified some of the more promising candidates.

The most promising approach to the development of a high-capacity, regenerable CO_2 sorbent identified by the AEPS studies is the ability of some metallic oxides to react with CO_2 to form the metallic carbonate according to the reaction:

$$MO + CO_2 \quad = \quad MCO_3$$

This reaction is reversible at elevated temperatures, thus thermally decomposing the carbonate, liberating CO_2, and reforming the oxide.

Table 1 lists the properties of interest of the metals comprising Groups I and II of the Periodic Table of Elements. The most attractive metal from the standpoint of potential CO_2 capacity is magnesium, which also has one of the lower regeneration energy requirements. Zinc and silver are attractive because of extremely low energy requirements. Easily eliminated are beryllium and cadmium because they are toxic, and lithium oxide due to its explosive instability in the presence of moisture.

Table 1, Theoretical CO_2 Capacities and Regeneration Energies for Metallic Oxides

Metallic Oxide	CO_2 Capacity (Weight %)	Regeneration Energy (BTU/lb CO_2)
Group IA		
Li_2O	147	2,213
Na_2O	71	3,143
K_2O	47	3,825
Rb_2O	24	3,944
Cs_2O	16	3,990
Group IB		
Ag_2O	19	802
Au_2O	11	
Group IIA		
BeO	176	
MgO	110	1,153
CaO	28	1,738
SrO	42	2,300
BaO	29	2,615
Group IIB		
CdO	34	974
ZnO	54	695

* This study was conducted in the laboratories of the McDonnell Douglas Astronautics Company, Huntington Beach, California. Sponsored by NASA Ames Research Center Contract No. NAS2-6959.

Early work with MgO (Reference 3) indicated the importance of a high moisture content in the reaction between MgO and CO_2. A postulated reaction mechanism of (Reference 1):

$$MgO + H_2O \rightleftharpoons Mg(OH)_2$$

$$Mg(OH)_2 + CO_2 \rightleftharpoons MgCO_3 + H_2O$$

led to the selection of $Mg(OH)_2$ and $Zn(OH)_2$ as additional candidate materials (AgOH) does not exist). The theoretical CO_2 sorption capacities of the five candidate materials are listed in Table 2.

Table 2, Theoretical CO_2 Capacities of Candidate Metallic Oxides and Hydroxides

Candidate	Weight Percent CO_2 Capacity
MgO	1.092
$Mg(OH)_2$	0.760
ZnO	0.540
$Zn(OH)_2$	0.443
Ag_2O	0.189

Figure 1 shows the equilibrium behavior of the candidate materials. It should be noted that these materials behave differently than the more commonly encountered CO_2 adsorbents. The CO_2 partial pressure in equilibrium with the metal oxide is solely dependent on temperature and independent of bed loading as long as any oxide is in contact with the atmosphere.

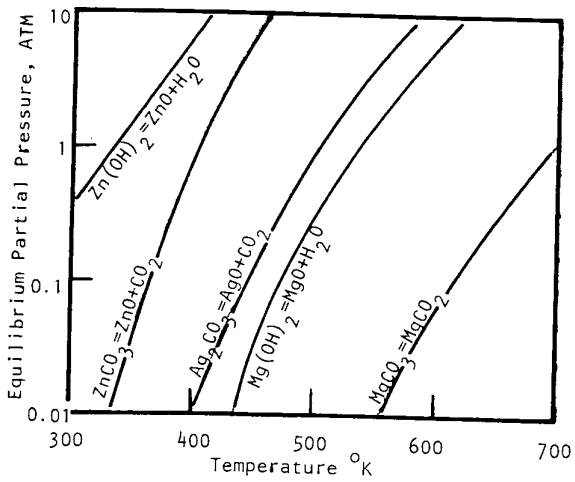

Figure 1, Equilibrium for Carbonates and Hydroxides

The hydroxides decompose well below the carbonates, thus direct regeneration of a hydroxide sorbent is not possible unless the vapor pressure of water is maintained above the equilibrium point. This is feasible with $Zn(OH)_2$ but the pressures required by $Mg(OH)_2$ would be much too high to be practical. The only alternate regeneration process is to decompose the carbonate to the oxide and then hydrate the oxide to the hydroxide with water; however, literature sources show that this does not occur to any appreciable extent and the extensive commercial use of magnesia ceramics substantiates this fact.

Pellet Preparation

The selection of the most promising candidate material was accomplished by manufacturing pellets similar to the configuration in which they would be used in an operational system, and evaluating their performance under identical conditions.

Preparation of Materials

Previous studies (References 3 and 5) have shown that an oxide most suitable for reaction with CO_2 is formed from freshly decomposed carbonate. Consequently, all pellets were manufactured from AR-grade metal carbonate in a water slurry. The addition of catalysts and binders was accomplished before the pellets were made. The most widely used additives were alkali metal hydroxides and sodium silicate. Aqueous solutions of these additives were thoroughly dispensed through the carbonate powder in a mortar and pestle before loading into the pellet maker.

Commercial sources of $Mg(OH)_2$ and $Zn(OH)_2$ could not be located; consequently, they were manufactured by precipitation from alkaline solutions.

Pellet Manufacture

The pellets were produced by extrusion of a slurry on a moving belt which is dried with hot air. The test material in slurry form was loaded into a hopper and forced through a nozzle by a piston-type slurry pump. The belt passed through a 2-inch square aluminum tube 6 feet long through which hot air was forced counter-currently by a heat gun. Thus, the pellets were partially dried as they passed through the tube and easily fell off the belt as it passed over the end roller.

Activation

The newly manufactured pellets were dried overnight at 355°K. Activation was accomplished in a muffle furnace at a temperature sufficient to decompose the carbonate to the oxide. These temperatures were determined by measuring the weight loss from samples of pure carbonate after one-hour exposure in the furnace at increasing increments of 25°K while both zinc and silver carbonates required only 523°K. All pellets were activated by placing them in an alumina crucible and placing it in a preheated furnace at the appropriate temperature for two hours. Cooling was accomplished by placing the crucible in a dessicator. This procedure was also used

when multiple regenerations of a material were accomplished.

Pre-Wetting

Early work with magnesium oxide (Reference 3) showed that significant improvements in sorption rates and capacities could be accomplished by adding moisture in amounts from 10 to 100 percent of the dry weight of the bed. This was accomplished by exposing the pellets to saturated steam.

Test Apparatus

The candidate materials were compared by obtaining CO_2 breakthrough curves under identical test conditions. The regeneration characteristics of the most promising candidate were determined by monitoring the CO_2 evolution as a function of time. The crush strengths of all candidate pellets were also measured.

Screening Test Apparatus

The apparatus used in the screening tests is shown in Figure 2. The candidate sorbent material pellets were placed in a 1-inch diameter glass tube filled to a depth of approximately 4 inches. This bed configuration was found to yield reliable data without appreciable channeling or wall effects. The tube was then mounted vertically (the pellets supported by a layer of glass beads) and connected to the flow apparatus. A humidified stream of 1.1 percent CO_2 in nitrogen, simulating the maximum expected feed concentration, was fed to the tube at a rate of 200 cc/min. Certified gas mixtures provided consistent and reliable feed compositions. The effluent from the tube was continuously monitored for CO_2 concentration with an MSA Model 300 infrared analyzer calibrated at 1.1 percent CO_2 full scale. Continuous water concentration was monitored by a Cambridge Model 880 Dewpointer. The readouts from these two instruments were recorded continuously on strip chart recorders.

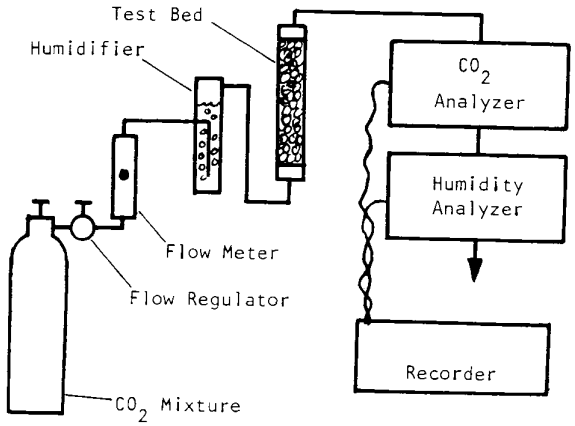

Figure 2, Breakthrough Test Apparatus

Regeneration Test Apparatus

The regeneration characteristics of the materials were studied using the apparatus shown in Figure 3. The pellets were contained in the same glass tube used for the sorption process. This tube fit inside a copper tube which in turn was placed in an electric combustion tube furnace. A thermocouple soldered to the copper tube was connected to a recorder/controller which controlled the tube temperature and provided a time/temperature curve. An auxiliary thermocouple was placed in the center of the bed to provide core temperatures. In most cases, a stream of dry nitrogen was passed through the bed to carry the evolved moisture and CO_2. In one case, a Welch roughing pump was connected upstream of the condenser and the nitrogen purge was stopped to stimulate regeneration to space.

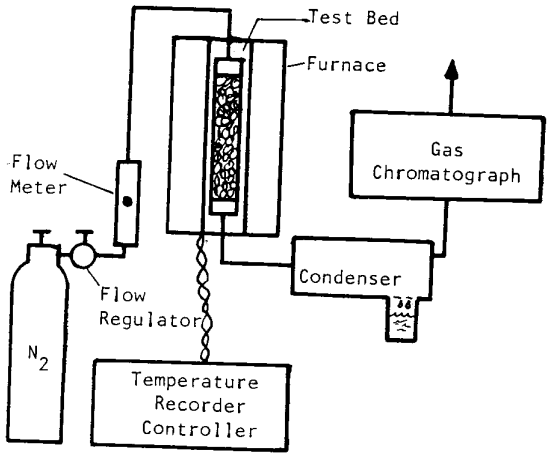

Figure 3, Regeneration Apparatus

Pellet Crush Strength Tester

A pneumatic cylinder with a known cross section piston was connected to a pressure gage and pressure regulator. The test pellet was placed below the piston shaft and the pressure increased until the pellet fractured. The indicated pressure was then used to calculate the force required to crush the pellet.

Screening Results

Selected results from the preliminary screening tests are presented in Table 3. The sample nomenclature has been assigned as follows. The first letter designates the parent oxide; i.e., M for magnesium, Z for zinc, and S for silver. The number following the first letter designates the serial number of the composition of this particular compound. The third digit is either a D or W denoting whether the sample was first tested dry or with water added. The fourth digit denotes whether the sample was tested again wet or dry, or the number of times this sample has been regenerated.

Table 3, Preliminary Screening Test Results

Sample No.	Composition	Time to BT (min)	Time to 80% Feed (hr)	Wt % CO_2 at BT	Wt % CO_2 at 80%	Curve (Shape) (Fig 4)	Crush Strength (Pounds)
M1D	86.2% MgO, 13.8% KOH	105	3.7	2.9	4.6	A	2.0
M1W	68.6% MgO, 11.0% KOH, 20.2% H_2O	210	19	4.0	17.7	A	2.0
M5W	40.4% MgO, 21.6% Na_2SiO_3, 7.8% Asbestos, 30.2% H_2O	60	13	1.1	7.9	A	8.2
M6W	32.5% MgO, 6.2% KOH, 12.9% Na_2SiO_3, 6.1% Asbestos, 42.3% H_2O	225	16.5	3.5	11.1	A	4.0
M6DW5	40.1% MgO, 7.6% KOH, 15.9% Na_2SiO_3, 7.6% Asbestos, 28.9% H_2O	50	14	1.0	9.5	A	4.1
M12W	38.1% MgO, 9.8% LiI, 9.6% KOH, 42.5% H_2O	240	15	3.5	7.0	A	2.0
M13D	100% $Mg(OH)_2$	0	23.5	0	9.6	B	-
Z6D	74.9% ZnO, 11.7% KOH, 13.2% Na_2SiO_3	10	11	-	4.4	B	0.9
Z11D	100% ZnO	5	5.8	-	3.5	B	0.8
Z14D	60.7% ZnO, 11.7% NaOH, 27.6% Na_2SiO_3	30	36	0.6	15.5	B	2.1
Z14D3W2	49.4% ZnO, 9.5% NaOH, 22.4% Na_2SiO_3, 18.7% H_2O	60	20	0.8	9.1	C	2.0
Z19D	100% $Zn(OH)_2$	0	14.5	0	4.0	B	-
S1D	100% Ag_2O	0	14	0	5.4	C	1.0
S4D(1-28)	80.3% Ag_2O, 10.4% KOH, 9.3% Na_2SiO_3	1200	40	8	15	A	0.8-6
S6D3	98.8% Ag_2O, 1.2% Y_2O_3	0	40	0	9.4	C	1.0
S7D(1-22)	Same as S4	1000	36	13	14	A	2-8

For example, sample Z2D2 is the second zinc compound to be manufactured and it has been regenerated and tested twice. Sample M6DW2 is the sixth magnesium compound. It was first tested dry, then tested wet twice.

The second column in the table gives the sample composition in the as-tested form. The BT time in column 3 is the time in minutes before CO_2 first appeared in the bed effluent. The "time to 80%" is the time required for the CO_2 concentration in the effluent to reach approximately 80 percent of the feed concentration.

The "wt % CO_2 at BT" column is the weight of CO_2 absorbed by the bed up to the time of breakthrough expressed as a percent of the initial bed weight, and the "wt % CO_2 at 80%" is the weight of CO_2 absorbed when the effluent concentration of CO_2 reached 80 percent of the feed concentration.

The curve shape column specifies the general shape of the experimental breakthrough curve. The letters correspond to the general curve shapes shown in Figure 4. In general, results with magnesium and zinc compounds were disappointing from either capacity, rate, or strength standpoints.

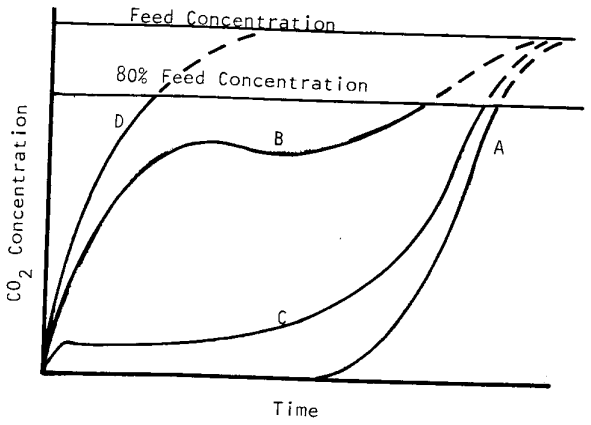

Figure 4, Typical Breakthrough Curves

Early results with silver oxide using potassium hydroxide and sodium silicate individually as additives showed both give rather good total capacities and promising structural properties but poor sorption rates. Compound S4 includes both additives and excellent results were obtained. This material has shown no tendency to degrade in activity through 28 regenerations. In all cases, regeneration was carried out by removing the pellets from the tube, placing them in an evaporating dish, and heating in a preheated oven at 523°K for two hours. The regenerated pellets have a very dark brown color, while the expended material has a greenish tint.

The sorption rate showed a marked improvement during the first few regenerations. This was probably due to some structural changes occuring, since after the fifth regeneration some degradation in pellet hardness was noted. This process apparently continued since the bed volume increased in spite of loss of material due to spillage. Some dusting during handling became apparent sometime after 10th regeneration.

Runs 15 through 21 were conducted at increasing flow rates in an effort to get some idea of the sorption rate under conditions expected in an AEPS. Breakthrough curves for these runs are shown in Figure 5.

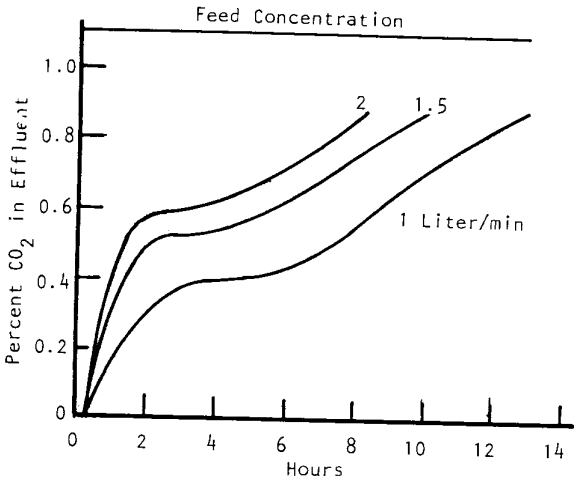

Figure 5, Breakthrough Curves for S4

Some indication of rate-controlling mechanisms may be deduced from these data. At any point in time, doubling the flow rate does not double the effluent concentration. Thus, the major factor in controlling the CO_2 sorption rate is probably mass transfer at the surface of the pellet.

Sample S6 was manufactured by the method described by Culbertson in Reference 5. He found the presence of 1 percent Y^{+++} ion to greatly enhance the performance of silver oxide. The data for S6 showed quite good CO_2 capacities, which increased with each regeneration. However, the rate was very poor as evidenced by immediate breakthroughs.

Sample S7 was manufactured with exactly the same composition as S4 to provide verification of S4's attractive behavior and a source of specimens for detailed analyses. The regeneration process was modified somewhat in that the pellets were retained in the sorption tube during regeneration. In addition, the tube was placed in a cold furnace and allowed to heat up with the furnace. This change in procedure is assumed to be the cause of extremely stable pellet structure through 22 regenerations. No dusting or crumbling was observed, in contrast with S4 which began to dust after 12 regenerations. Several runs were made at high flow rates verifying the curves for S4 in Figure 5. Some of these are shown in Figure 6. In addition, water breakthrough curves are shown.

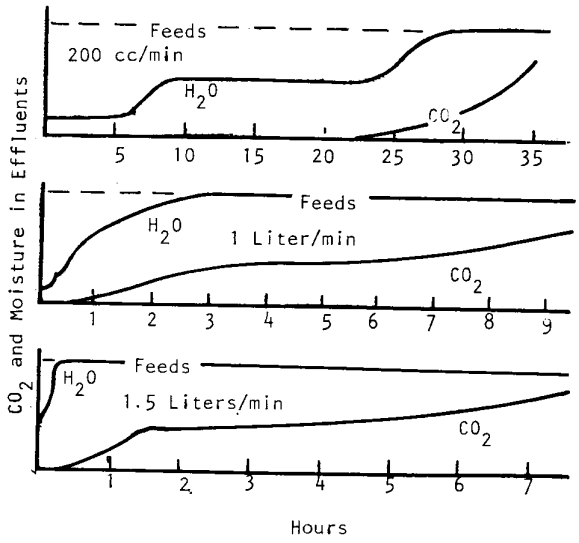

Figure 6, Breakthrough Curves for S7

If one looks closely at the point where the CO_2 breakthrough occurs, it appears to correspond to the second knee in all three water curves. It has been postulated that the potassium present in this composition is present as K_2CO_3. If the K_2CO_3 reacts with H_2O and CO_2 forming $KHCO_2$, it may explain the correlation between these curves since the exhaustion of K_2CO_3 would then lower both CO_2 and H_2O sorption rates.

Runs 20 and 21 were made with a CO_2 concentration of 3 mm Hg. The resulting capacities, approximately 12 percent, show a potential use for this material for the control of CO_2 in space cabins in light of the newer low CO_2 partial pressure requirements. Breakthrough curves are shown in Figure 7.

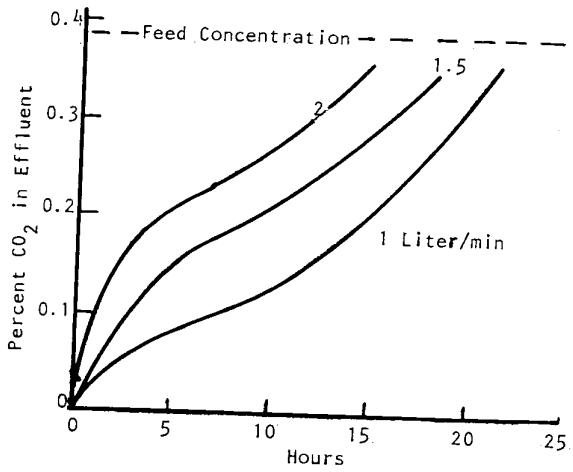

Figure 7, Breakthrough Curves for Space Cabin Atmosphere

Figure 8 shows a compilation of the capacity data for samples S4 and S7. No degradation in performance is evident (within the scatter of the data) through more than 20 regenerations. Some of the lower points were from the high flow runs which is understandable since the tests were terminated at 80 percent breakthrough.

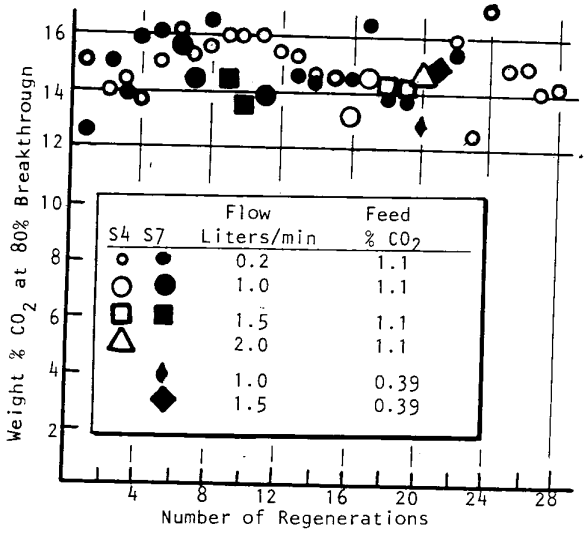

Figure 8, Regeneration Histories for S4 and S7

Regeneration Studies

The regeneration characteristics of compounds S4 and S7 were studied using the apparatus shown in Figure 3.

Figure 9 shows a regeneration of sample S7D4 with the oven control set at its lowest power setting. Approximately 40 minutes were required for the oven to reach 500°K. An early low temperature spike is evident followed by the majority of CO_2 evolving when the bed temperature went past 450°K. An extremely slow trailoff was encountered until the temperature was increased to 525°K. The rate then went directly to zero.

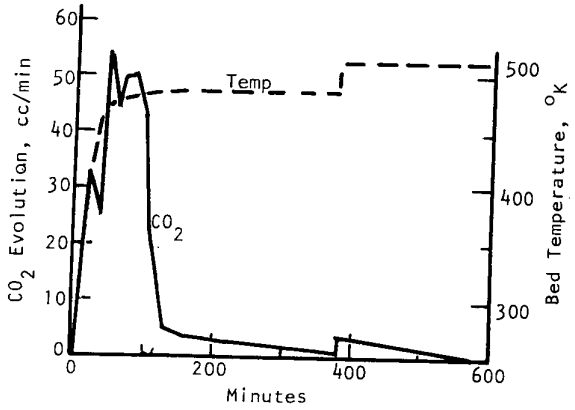

Figure 9, Regeneration of Sample S7D4

Detailed Analysis of Samples

Some of the more promising samples were subjected to detailed chemical and physical analyses in an attempt to identify those properties which could be correlated to desirable sorption and physical characteristics.

Thermogravimetric Analysis (TGA)

This technique involves monitoring the weight loss of a small sample as a function of temperature in any desired atmosphere environment. The equipment also measures the instantaneous rate of change at a given point.

Figure 10 shows the decomposition characteristics of the three carbonates of interest. The rate of change of basic magnesium carbonate shows two distinct decompositions, probably Mg $(OH)_2$ and H_2O at 500°K and then $MgCO_3$ around 675°K. The curves for silver carbonate show two decompositions and the percent lost is greater than theoretical for going to the oxide. Apparently the oxide decomposition to metallic silver begins near 500°K.

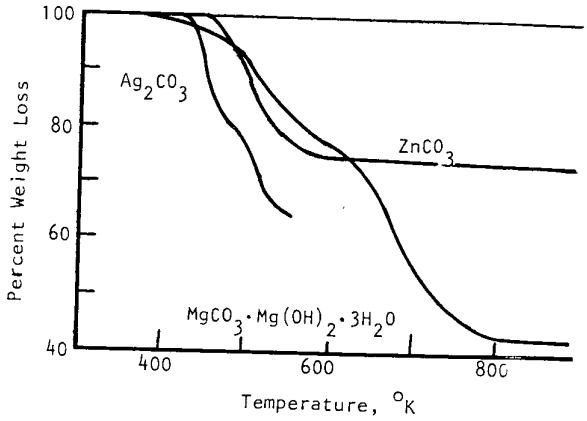

Figure 10. Thermogravimetric Analysis of Carbonates

The decomposition of compound S4 was studied in several environments as shown in Figure 11. A

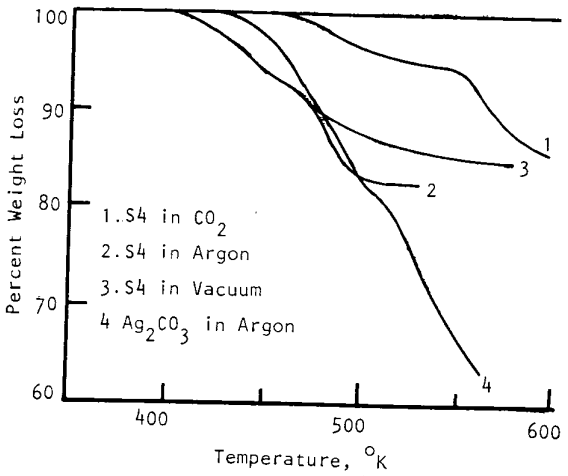

Figure 11, Thermogravimetric Analysis of S4

comparison of S4 in argon with pure silver carbonate shows a very different process. The very early decomposition correlates with the early peaks observed during the large-scale regenerations. The decomposition to Ag_2O appears to stabilize after 525°K as evidenced by the rate returning to zero. Complete decomposition in the vacuum test appears to require higher temperatures for completion. However, it is probable that this is an instrumentation error since the thermocouple was shielded from the furnace wall by the sample holder and relied on secondary radiation for its energy. In gaseous environments, the thermocouple received its heat by conduction and probably came closer to matching the specimen temperature. The upper curve shows the effect of CO_2 partial pressure compared with the test run in argon. Complete decomposition did not occur until 600°K; however, it appears to have stabilized as the oxide even at this temperature.

Physical Properties of Pellets

The surface and porosity characteristics of the best magnesium and silver pellets were measured.

Table 4 presents the surface area and percent open porosity of various forms of compound S7 and M7. Compound M7 is a new batch of pellets of composition identical to the best magnesium compound tested, M6. The specific area data for the S7 samples is somewhat unexpected since the area of S7D4 oxide is less than S7D1; this in spite of the fact that S7D4 carbonate is greater than the oxide in spite of a significantly lower open porosity. However, since the areas are all within 10 percent of an average (the level of precision expected as reported in Reference 6), the differences may be ignored as experimental error.

Table 4, Physical Properties of Pellets

Compound	Area[1] m^2/gm	Percent Open Porosity[2]
S7D1 oxide	3.31	67.16
S7D4 oxide	3.02	50.37
S7D4 carbonate	3.41	53.88
M7D oxide	17.8	80.68
M7W oxide	8.4	68.05
M7DW5 oxide	6.8	-
M7W carbonate	-	59.73

(1) As measured by BET method.
(2) As measured by mercury intrusion porosimitry.

The areas and porosities of the M7 compounds are considerably greater than those of the silver compounds. The addition of water causes a large loss in area although it greatly enhances the CO_2 sorption properties of the material.

Scanning Electron Microscope Studies

Detailed photographs of the surfaces and fractured cross sections of various pellet configurations were obtained using a scanning electron microscope. Samples were prepared by vacuum depositing a thin layer of gold on the pellets to provide an electrically conductive surface.

Figure 12 shows the surface of an M7W pellet in carbonate form. The carbonate form shows a

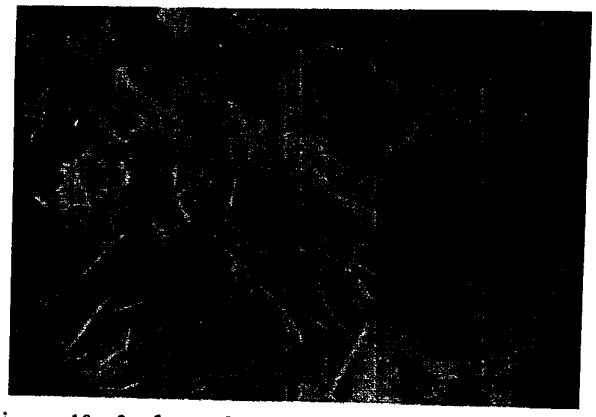

Figure 12, Surface of M7W Carbonate X 1500

number of spiny-appearing clumps on the surface. The clumps are actually made up of fine plates of carbonate which have grown from the surfaces of some of the oxide particles. Energy-dispersible x-ray analysis of these clumps show them to contain a higher potassium content than the granular particles and essentially no silicon. Thus, it appears that these carbonate growths are selectively concentrating the potassium, and repeated regenerations will eventually lead to a separation of the active ingredients. This will eventually lead to loss of activity and/or structural integrity.

Figure 13 shows the surface of S7D12 in the carbonate form. The carbonate seems to grow throughout the structure and retains its amorphous properties. This is ideal behavior for a regenerable system since no localized changes take place.

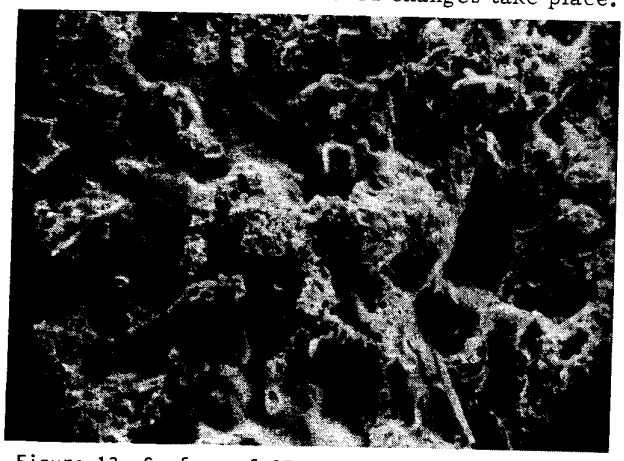

Figure 13, Surface of S7D12 X 1500

Apparently a carbonate skin forms on the pellets. It appears to be quite impervious and one would expect it to form early in the sorption process and hinder complete utilization of the pellet. However, sorption data show greater than 90-percent theoretical conversion with good sorption rates. Therefore, either the skin has sufficient holes, including those that are not obvious, to permit good mass transfer, or solid diffusion in the matrix is very rapid. The relatively low surface area of this material--3 m^2/gm compared to common adsorbents such as molecular sieves--suggests that solid diffusion is likely the dominant mechanism.

Cross sections of both forms, shown in Figure 14, show an almost glassy continuity in the structure while maintaining some open structure.

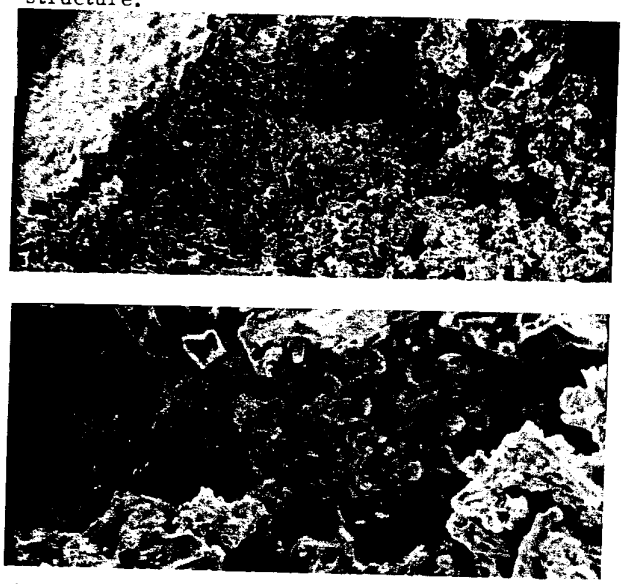

Figure 14, Cross Section of S7D12 X 1500

Figure 15 shows the surface of a zinc-oxide pellet. The light rod-shaped structures are assumed to be zinc carbonate formed by exposure to the air during sample handling. The profusion of these structures on the surface of the carbonate form indicates that zinc carbonate grows from particles in a manner similar to that of magnesium carbonate and thus will eventually lead to selective separation of components.

Figure 15, Surface of Z14D3W3 X 375

Discussion of Results

The objective of this program was to select the most promising candidate for development into a regenerable CO_2 sorbent for EVA. The first conclusion that may be drawn is that magnesium and zinc hydroxides may be eliminated from further considerations. Since the hydroxides decompose at a temperature lower than the carbonates, regenerability is impossible since the oxides do not rehydrate. In addition, the hydroxides showed little affinity for CO_2. This also discounts the previously assumed mechanisms which involved the hydroxides as intermediates when wet oxides react with CO_2.

The potential development of zinc oxide is also doubtful based on the results discussed earlier. All sorption capacities are quite low, based on theoretical analysis, and the binder which was successful with other materials was ineffective with zinc oxide compounds. In addition, SEM studies indicate the selective separation of materials during the carbonation reaction thus eventually limiting regenerability.

Magnesium oxide compounds, although theoretically having the highest sorbent capacity, could not be induced to absorb CO_2 beyond approximately 15 weight percent. In addition, they require the highest regeneration temperature and must be prewetted before they become active. A binder system was developed which yielded very strong stable pellets. The SEM photographs show a granular consistency with the carbonate growing away from the particle surfaces. This process was found to selectively concentrate the catalyst

in the carbonate and reject the binder. Repeated regenerations will obviously lead to decomposition of the structure.

The results from the tests of silver oxide compounds are most encouraging. Compounds S4 and S7, with identical compositions, were found to absorb essentially 95% of their theoretical capacity through 28 regenerations without any noticeable loss of efficiency. Although the weight percent attained is approximately the same as attained in some of the better magnesium oxide compounds, the density differences between silver oxide and magnesium oxide makes silver oxide much more efficient based on volume requirements. In addition, the regeneration temperature for silver oxide is much lower and no pre-wetting is required. Silver oxide is clearly the most promising candidate of the five materials specified in the contract for further development.

A rough scale-up of the high flow breakthrough curves for S4 shown in Figure 5 was calculated in an effort to determine the bed size required for an actual AEPS. A direct proportional scale-up shows that 17 lbs of material will maintain the CO_2 partial pressure below 4 mm Hg for 10 hours at a metabolic rate of 1200 Btu/hr. The dimensions of this bed would be 7 inches in diameter and 10 inches long. This scale-up is very conservative since the feed concentration used in the test is the maximum allowable, and an actual system will start at zero and build up to that point. In addition, a larger bed will be more efficient due to increased depth and smaller wall effects. The Apollo LiOH cartridge weighs only 4 lbs (Reference 4). However, it is only rated for six hours and the maximum allowable CO_2 concentration is almost four times higher (15 mm Hg). A LiOH cartridge sized to these AEPS specifications may not be much smaller than an Ag_2O unit.

Conclusions

- Magnesium hydroxide and zinc hydroxide show no potential for development into regenerable CO_2 sorbents.
- Zinc oxide shows a poor affinity for CO_2 and no satisfactory structural binder has been identified.
- A good binder system has been developed for magnesium oxide; however, the CO_2 capacity has not been improved over previous results.
- A silver oxide formulation has been developed which results in essentially theoretical CO_2 capacity at a high rate with good regenerability. This material should be developed further.

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