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DEGRADATION OF DESICCANTS UPON CONTAMINATION: AN EXPERIMENTAL STUDY

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ABSTRACT

Experiments were conducted to quantify the effects of thermal cycling and exposure to contamination on solid desiccant materials that may be used in desiccant cooling systems. A test apparatus was used to thermally cycle several desiccant samples and expose them to "ambient" or "contaminated" humid air. The source of contamination was cigarette smoke. Six different solid desiccants were tested: two types of silica gel, activated alumina, activated carbon, molecular sieves, and lithium chloride. The exposed desiccant samples were removed after 0.5, 1, 2, 4, or 11 months of exposure and their moisture capacities were measured. Other tests were conducted to characterize pollutants deposited on the exposed samples or to evaluate impact of exposure on internal structure of the samples. Compared to fresh samples, the capacity loss due to thermal cycling with ambient air was generally 10% to 30%. The capacity loss due to only cigarette smoke was generally between 20% to 50%.

INTRODUCTION

Desiccant cooling systems thermally regenerated with solar energy, natural gas, or other thermal sources are gaining acceptance for space air-conditioning applications. In a typical desiccant cooling system, a desiccant dehumidifier removes the moisture from the process air. Then the air is cooled to the desired conditions by a set of regenerative evaporative coolers or by vapor compression coolers. The desiccant material in a dehumidifier adsorbs (or absorbs) moisture from the process air to be dried. Later, the desiccant material is regenerated with hot air (generated by a thermal source) to drive the moisture from the desiccant for the next adsorption cycle.

In addition to moisture, a desiccant material may co-sorb pollutants from the air. Although the co-sorption process may be used for air cleaning, it may interfere with the moisture sorption process and degrade the performance. Co-sorption of pollutants may be an irreversible process and reduces the useful life of desiccants.

Hydrothermal cycling of a desiccant (a process occurring many times in a dehumidifier) may breakdown its internal structure, reducing performance and useful life. Based on hypothetical degradation scenarios, our preliminary system performance calculations showed that desiccant degradation can significantly reduce the performance of a desiccant cooling system.

The life of a desiccant affects the life of a desiccant dehumidifier. Because the useful life of desiccant dehumidifiers is a concern of manufacturers and end users, experimental data are needed to quantify the magnitude of degradation. Some data exist on degradation of desiccants for industrial applications. However, for residential and commercial air-conditioning applications, such data did not exist.

The objective of this research work was to obtain comprehensive experimental data on degradation of desiccants under conditions experienced in solar-regenerated desiccant cooling applications. Our desiccant contamination research strategy was based on the recommendations of a Desiccant Contamination Workshop sponsored by U.S. Department of Energy and conducted in Washington, D.C. in June 1987. The major recommendations of the workshop were that controlled contamination experiments should be conducted to obtain the time history of desiccant degradation due to thermal cycling and airborne contaminants. For initial screening, cigarette smoke in a proper mix with humid air was recommended as the first airborne contaminant to be studied. Cigarette smoke, a mixture of gaseous and particulate contaminants, was considered to be the worst contaminant commonly found in residential and commercial buildings. Note that the above experiment was considered an extreme-case screening mechanism, i.e., if the desiccants did *not* sufficiently degrade with cigarette smoke, then desiccant contamination would not be an issue.

Based on the workshop recommendations, a test apparatus was built and used to expose 200 samples of six different solid desiccant materials for several months. Then the sorption capacities of the exposed desiccants, as well as virgin samples, were measured. The purpose of this paper is to describe the contamination experiment and to present the test results on some of desiccant samples.

THE EXPERIMENTAL APPARATUS

The experimental apparatus called the Desiccant Contamination Test Facility (DCTF) was built to thermally cycle and contaminate multiple solid desiccant samples under carefully controlled conditions found in the cyclic operation of desiccant dehumidifiers (Pesaran and Bingham 1988). Experiments and analyses to characterize the degree and cause of degradation were done elsewhere as described later. The purpose of the DCTF was to provide data on the degradation of desiccant materials with time because of hydrothermal cycling and airborne contaminants. Specifically, the DCTF was designed to

Cycle desiccant samples between a hot and a warm stream of humid air.

Expose desiccant samples to airborne contaminants such as cigarette smoke.

Simulate the operation of a desiccant dehumidifier in a solar-regenerated desiccant cooling system.

Note that the desiccant samples in the contaminated test cell of the DCTF represent worst-case scenarios for desiccant materials found in each of the following cases:

- The desiccant cooling cycle is in the ventilation mode and the outside air is very polluted.
- The desiccant cooling cycle is in the recirculation mode and the return air from the conditioned space is very polluted.
- The desiccant cooling cycle is in any mode and the regeneration air is very polluted.

Configuration and Components

The DCTF, shown schematically in Figure 1, consists of two test cells (each can hold 100 desiccant sample tubes), two duct heaters, two external booster heaters, air filters, a humidifier, a smoking machine, 10 globe valves, and an air blower. The components are connected via 3.8-, 5-, and 7.6-cm galvanized steel pipes. After the air is humidified to 0.012 to 0.016 kg water/kg dry air, it is divided into two branches.

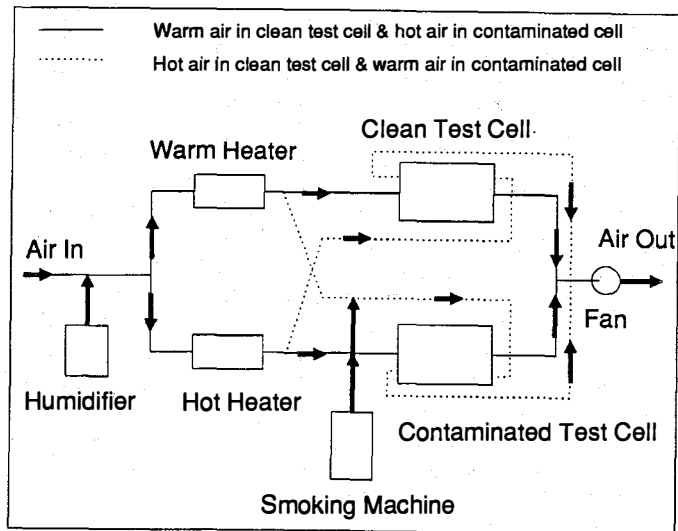


Fig. 1. Schematic of the Desiccant Contamination Test Facility

In each branch, air passes through a heater and a test cell. The hot heater (2 kW) heats the air in one air stream to about 100°C; the warm heater (0.5 kW) warms the air in the second branch to about 38°C. The heat from the booster heaters compensates for the heat losses between the heaters and the test cells. Depending on whether the valves in each branch are in the open or closed position, hot air can go in only one test cell (for desiccant regeneration) while warm, humid air is going in the other test cell (for desiccant adsorption). After coming out of the two test cells, the air streams in the two branches join in one stream that is exhausted outside the building with the air blower. Cigarette smoke is injected through a pipe connected to only one of the test cells.

The smoking machine was designed and fabricated by a manufacturer to generate fresh smoke. The machine consists of a cigarette hopper, a hopper drum, a smoking drum, an electric lighter, an ash receptacle, two motor drives, a timing circuit with a photoelectric sensor, and associated electronics and circuitry.

Instrumentation, Control, and Data Acquisition

Temperatures and humidities of air at the entrance and exit of each test cell were monitored and recorded during the experiment by a set of thermocouple arrays and chilled-mirror dewpoint hygrometers. The flow rate to each test cell was measured with a turbine flowmeter. The pressure drops across each test cell and absolute pressure were measured with pressure transducers. The analog signals from all the sensors were measured with a voltmeter/scanner. The analog signals were then converted to digital signals and transferred to an IBM-XT personal computer for calculation, display, and storage. A data acquisition software was used to collect raw data, analyze them, and graphically display the engineering parameters in real time. The collected data were stored in either 15-s or 1-min intervals.

The duct heaters were controlled with two remote thermostats. The outlet air temperatures from each heater were successfully controlled at desired levels uniformly with fluctuations of less than 0.5°C. Figure 2 shows typical air temperatures at the inlet of the two test cells as a function of time. The inlet air temperature for each test cell cycles between an adsorption and a regeneration temperature to simulate typical dehumidifier operation. The outlet air temperatures from each test cell are also shown in Figure 2. Notice how the ambient and contaminated test cells are identically out of phase with each other because of the designed cyclic nature of the facility. During the experiment, the air temperature going into each test cell was between 41°C and 44°C during adsorption and between 80°C and 84°C during regeneration.

To control the humidifier to obtain a uniform level in moisture in the main air stream, we implemented a feedback control mechanism with proportional, integral, and derivative (PID) features. As a result, the air humidity going into each test cell could be maintained at about 0.014 with ± 0.001 kg water/kg dry air fluctuations. This fluctuation was smaller than levels that a dehumidifier may experience in a field operation.

The air flow in each test cell was about 9.5 L/s. The cycle time of 7.4 min between regeneration and adsorption in each test cell was achieved by simultaneous opening and closing of a set of valves energized and de-energized by an electrical repeat-cycle timer. The total cycle time of 14.8 min is typical of the rotational speed of commercial dehumidifiers.

To obtain design concentration of cigarette smoke in the air stream going through the contaminated test cell, cigarette smoke was generated by the continuous smoking machine. The smoking

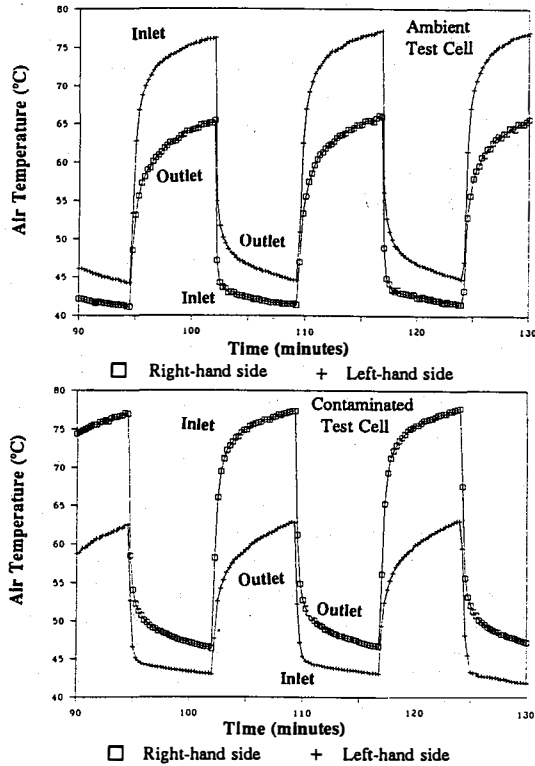
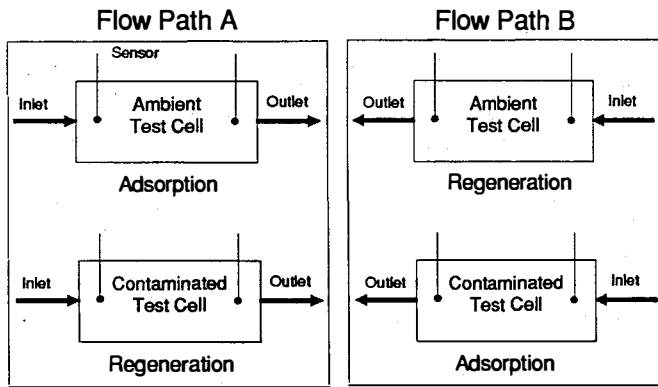


Fig. 2. Air temperature response at the inlet of the test cells for the two flow path configurations

machine injected fresh smoke at a rate of six cigarettes per hour into one of the air streams of the DCTF, where it was diluted with air (at about 9.5 L/s) to the desired concentration (about 6 mg/m³ of "total particulate matter"). This injection rate was based on twice the upper concentration of "total particulate matter" found in smoking residences, the air flow rate through a test cell, and the emission and burning rates of common cigarettes. According to Wadden and Scheff (1983), the case of 15-425m³ homes with 1 to 3 air changes per hour and 7 to 35 cigarettes burned per hour represents smoking residences. The concentration of "total particulate matter" of smoke in this case is 1.1 to 3.0 mg/m³.

Each test cell holds 100 sample tubes containing desiccants. The sample tubes are held by individual cylindrical brass sleeves. The sample tubes were made of brass, with an outer diameter of 8.7 mm and a length of 7.6 cm. The test cells were insulated during the experiment.

EXPERIMENTAL PROCEDURE

Sample Preparation

We prepared 10 sample tubes per desiccant for each test cell. Sample tubes were loaded with six different solid desiccants. When possible, two different batches of each desiccant were used to account for batch-to-batch or lot-to-lot variations caused by changes during manufacturing. The desiccant materials suitable for solar cooling applications are usually coated on or impregnated into the walls of a dehumidifier matrix. In this investigation, we considered desiccant materials in both bulk form (i.e., particulate) and matrix form. For example, silica gel was tested both in bulk form in a packed-bed configuration and in a matrix form. In matrix form, fine silica gel particles were coated on a double-sided tape, wrapped in a sinusoidal shape, and then inserted into a tube to form parallel passages in the tube sample. Lithium chloride was tested only in corrugated matrix form.

The following solid desiccants were used for the first phase of testing:

- Two lots of microporous silica gel particles, Grade 40, average size 1.5 mm
- Microbead silica gel on tape in a parallel-passage configuration, particle size 75-105 μm
- Fine-particle silica gel on tape in a parallel-passage configuration, particle size 150-300 μm
- Two lots of molecular sieve particles, 13X, average size 1.5 mm
- Activated alumina particles, average size 1.5 mm
- Activated carbon particles, average size 1.5 mm
- Two lots of lithium chloride corrugated matrix.

We prepared 200 sample tubes (20 tubes from each desiccant identified above) by loading desiccants and placing metal screens at both ends of each tube to hold the desiccant in tubes. The samples were dried with flowing air at 1.5% relative humidity and 100°C for more than 72 hours in another test apparatus called the heat and mass transfer test facility (Pesaran and Bingham 1989). The desiccant tubes were sealed on both ends with plastic caps to prevent the desiccant from interacting with the ambient air when not in the test cells.

Using a balance with a precision of 0.1 mg, we measured the weight of each tube, the weight of the screens, the weight of the dried desiccants, and the weight of the plastic caps. The average weight of the test tubes with two screens was 6.0 g. The weight of dry particulate desiccant samples varied from 1.5 to 1.8 g. The average weight of the lithium chloride corrugated desiccant was about 0.54 g. The average weight of microbead silica gel on tape was 0.28 g, with a tape weight of 0.08 g. The average weight of fine-particle silica gel on tape was 0.68 g, with a tape weight of 0.06 g. The average weight of two plastic caps was 1.24 g. In addition, 20 other desiccant sample tubes were prepared in the same manner as the virgin baseline for comparing with samples exposed in the ambient and contaminated test cells.

Test Procedure

We took the following steps to operate the DCTF:

1. One hundred desiccant sample tubes were installed in each test cell at the appropriate test tube holder. To keep track of where each desiccant sample was placed the tube holders were

- identified with a number (column identifier) and a letter (row identifier). Each test tube was also identified with a number (1 to 10 for the ambient test cell and 11 to 20 for the contaminated test cell) and a letter (A to J). The desiccant sample tubes were randomly placed in the test cell.
2. The test cells were closed and insulated. The data acquisition system, air blower, heaters and humidifier were turned on.
 3. The air flow rate, air humidities, and air temperatures were adjusted to desired set points.
 4. The smoking machine was loaded and turned on. Cigarette smoke was injected only into airstream of the contaminated test cell.
 5. The hot regeneration and warm adsorption air streams were cycled between the two test cells every 7.4 min by simultaneous opening and closing of globe valves.
 6. The experiment was started in the middle of August 1989 and was continued 24 hours a day until the middle of July 1990. After 0.5, 1, 2, 4, and 11 months, the apparatus was shut down and appropriate sample tubes from each test cell were removed. At each time, 20 samples were removed from each test cell and replaced with tubes containing glass beads or desiccant materials.
 7. Regularly, the status of the apparatus was monitored. The collected data were stored on 3.5-in. floppy diskettes every day.
 8. The above steps were repeated until all the first 200 samples were processed in the apparatus.

Note that each month of testing in the DCTF is equivalent to 6 months of field operation, assuming that a desiccant dehumidifier works 8 hours a day for 6 months a year and that the concentration in the field and in the experiment is the same. Accounting for the higher concentration in this experiment, we expect one month of testing in the DCTF to be equivalent to 1 to 2 years of field operation.

EXPERIMENTAL RESULTS

After samples were removed from the ambient and contaminated test cells, their moisture capacity was measured by a gravimetric technique. A set of the silica gel samples (virgin, ambient, and contaminated) was sent to a commercial laboratory for internal pore structure analysis. Another set of samples was evaluated using scanning electron microscopy and x-ray photoelectron spectroscopy. The contaminants deposited on some of the samples were extracted using a solvent and analyzed using the gas chromatography/mass spectroscopy technique. In this section, the results of some these efforts are presented. Further details and data can be found in Pesaran and Dresler (1990).

It should be noted that because of differences in densities of desiccant materials or porosity of test tubes, unequal pressure drop may exist through sample tubes. This may lead to channeling, i.e., more air may flow through one desiccant type than the other. This would result in different effective exposure time for each material. The nominal pressure drop for each test tube is being measured and we will adjust effective exposure time. Although we have presented the results in experiment time, the effective exposure time of each desiccant is different, and any comparison on time basis should be regarded as relative.

Visual Observation

The colors of virgin, ambient, and contaminated samples of several desiccants were compared. We noted that the color of

virgin silica gel changed from clear white (glasslike) to tan, amber or brown upon exposure in the ambient test cell to ambient air and thermal cycling. The contaminated silica gel samples exposed to cigarette smoke showed a drastic change in color. Their appearance changed to brown or dark brown. The degree of darkness increased with time of exposure. Other desiccants had similar color changes. The impurities from (ambient and contaminated) air that were deposited on the external surfaces of desiccant samples are expected to be the cause of these color changes.

Scanning Electron Microscopy (SEM)

Using SEM, we obtained microscopic views of typical surfaces of a virgin silica gel particle, and of 1-month and 4-month ambient and contaminated samples of silica gel particles. This was done to observe the deposition of any contaminants on the outer surfaces of the desiccant particles. With SEM, a very small segment on the outer surface of a sample is magnified.

Figure 3 shows 15 pictures taken using SEM at three different magnifications for virgin, ambient, and contaminated samples of silica gel (lot 1) particles. Note that the typical size of a silica gel particle is about 1.5 mm, and any magnification with SEM shows a close-up of the surface of the particle. The 6000x magnification of a virgin sample shows a number of peaks and valleys on the surface, which is typical of microporous materials. The 1-month and 4-month ambient samples also show a number of peaks and valleys on the surface. It appears that small changes occurred from the virgin samples to the 1-month and 4-month ambient samples. These changes are mostly expected to be results of the deposition of some impurities such as dust in the ambient air and also thermal cycling of the samples. Drastic changes on the surface of 1-month and 4-month contaminated samples occurred. The intensity of valleys and peaks on the two samples disappeared. In the 4-month samples, the peaks and valleys can hardly be seen, most likely as a result of deposition of higher amounts of contamination. It appears that a layer of a liquidlike material has covered the surfaces. Because the contaminated samples were exposed to cigarette smoke, it is reasonable to assume that constituents of cigarette smoke such as nicotine and tar have been deposited on the outer surfaces of the particles.

X-Ray Photoelectron Spectroscopy (XPS)

XPS is one of the surface analysis techniques to determine the elemental composition of the surface monolayer(s) of a solid (Czanderna 1984). Briefly, in the XPS technique, a sample is exposed to an x-ray beam. The x-ray beam penetrates only a few microns into the sample and generates photoelectron and Auger electrons. The energy of these electrons can be detected and related directly to elemental and chemical information about the atom from which they were rejected.

We used XPS to analyze a virgin silica gel sample and a silica gel sample, both from lot 1, removed from the contaminated test cell after 2 months of exposure. The results are presented in Table 1. From the XPS results, the atomic composition of the surface elements was estimated. Note that the atomic composition of unexposed silica gel (which is 99.9% silicon dioxide) is 33.3% silicon and 66.6% oxygen. The atomic composition of surface elements for the virgin sample was found to be 30.2% silicon, 64.5% oxygen, and about 5.3% carbon. The possible source of the carbon was estimated to be from hydrocarbons and carbon dioxide and monoxide adsorbed upon exposure to ambient air.

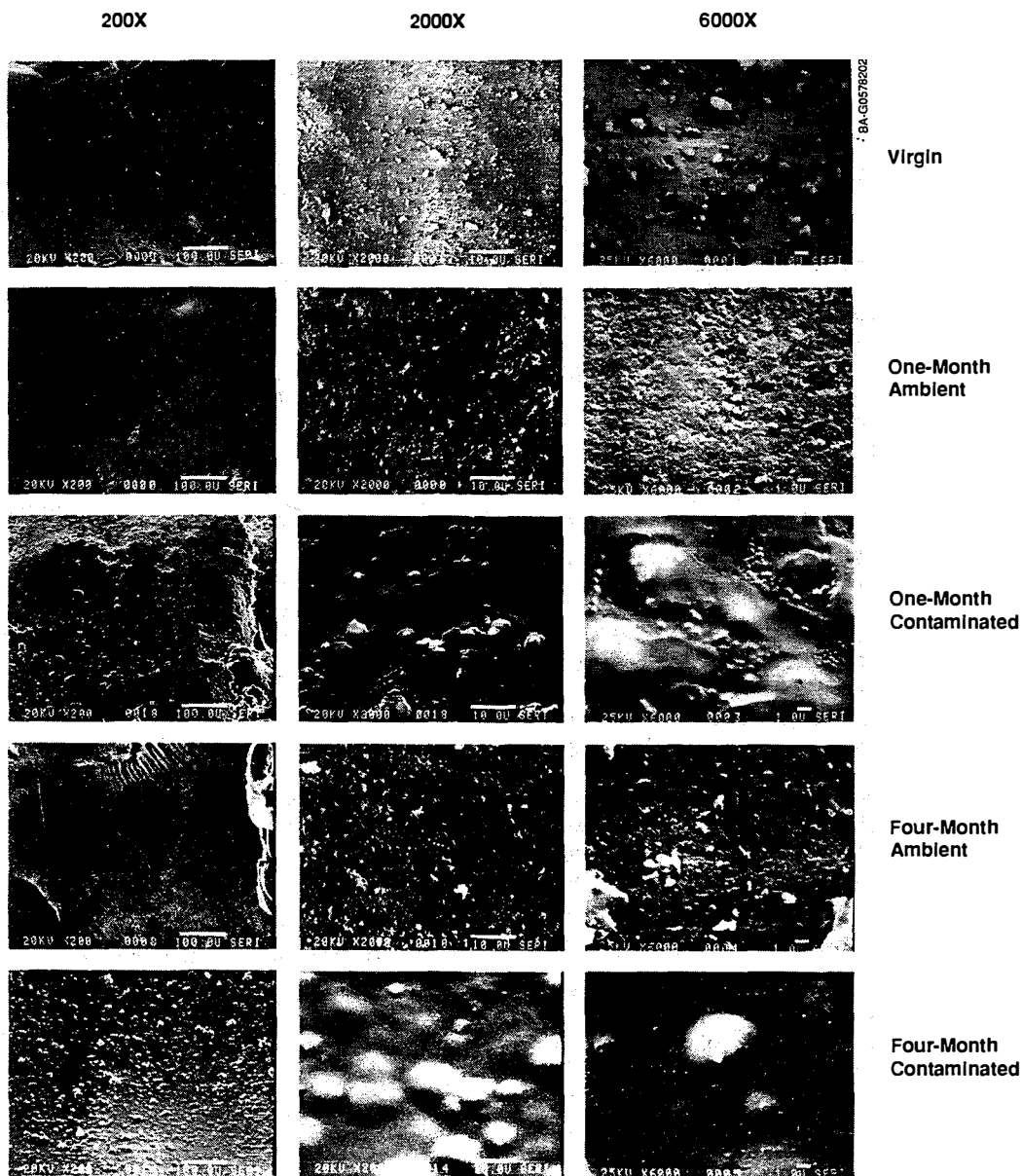


Fig. 3. Scanning electron microscopy of silica gel particles

TABLE 1
Surface Atomic Composition from XPS Analysis

Sample	Element (atomic %)		
	Carbon	Oxygen	Silicon
Virgin silica gel	5.3	64.5	30.2
Contaminated silica gel, particulate	89.2	9.95	0.82
Contaminated silica gel, ground	7.17	72.2	20.6

The results of XPS on the contaminated particles showed that the atomic composition of the surface elements was 0.82% silicon, 9.95% oxygen, and about 89.2% carbon. The comparison between the two XPS results indicated that the contaminated sample has a much higher carbon content. The silicon presence on the surface decreased because a much thicker or denser layer of surface carbon was formed. The source of the carbon could be hydrocarbons and tar present in the contaminated air.

A few particles of the contaminated sample were ground and analyzed with XPS. This was done to determine if the contamination layer had penetrated completely through the particles. The grinding of the particles mixed the internal and external surfaces of each particle. Analysis of the XPS results of

the ground sample showed 20.6% atomic silicon, 72.2% atomic oxygen, and 7.17% atomic carbon on the surface. From the difference between the ground and particulate results, one can conclude that the interior parts of the contaminated particles of the 2-month sample were less contaminated than the outer surfaces. In other words, the contamination resides mostly on the outer surface of the particles and has not significantly penetrated into the internal surfaces.

Gas Chromatography/Mass Spectrometry (GC/MS) Analysis

The GC/MS technique was used to identify the volatile organic contaminant in the air and in the desiccant samples. The gaseous samples were analyzed directly in this method; the solid samples were first extracted with a nanograde (ultrapure) solvent. Then the extracted sample was injected into a gas chromatograph, where separation of various sample components was accomplished by a column. Eluted components were then detected using a mass spectrometer.

The components of cigarette smoke have been identified by many investigators (e.g., Meyer 1983). Therefore, initial experiments concentrated on the identification of compounds in the air stream reaching desiccant samples in the contamination test cell. Using airtight syringes, we withdrew gaseous samples from the contamination test cell and analyzed them by GC/MS. However, we could not detect any compound in the samples. This was attributed to sampling procedure, low concentrations of smoke in the test cell, or condensation of the gaseous sample in the syringes.

Characterizing the smoked-filled air before or after exposure to the desiccant samples would require some form of increase in the concentration of pollutants of the samples removed before instrument analysis. This could be accomplished with a liquid nitrogen cold trap, fused silica capillary tubing, a vacuum pump, and a gas chromatograph with either a flame ionization detector or a nitrogen/phosphorus detector. This approach is recommended to be taken in future work.

The desiccant samples considered for GC/MS analysis were a 1-month contaminated sample, a 4-month ambient sample, and a 4-month contaminated sample. All samples were silica gel, lot 1. To analyze the samples, we extracted 0.25 g of each desiccant with 0.5 mL of a methanol and methylene chloride mixture for 10 min in a sonicating water bath. One μL from each extract was injected into the GC/MS for analysis. The components in the mass spectrometer were first divided into ion groups and then detected. The total ion chromatograph for all the extracted samples, as well as for a solvent blank, are shown in Figure 4. The time at which an ion group is detected is called elution time.

The ion chromatographs from the two contaminated desiccants are quite similar and differ only in the degree of contamination. As expected, the desiccant exposed to smoke for 4 months collected more contamination. The major contaminant in the exposed desiccants is nicotine, which eluted at 17.0 min for both contaminated desiccants. Estimating the area under each peak indicated that the 4-month contaminated desiccant collected between two and three times pollutants than the 1-month contaminated desiccant.

Pore Structural Analysis

All the desiccants tested, except lithium chloride, are microporous materials. The internal structure of a porous desiccant may change as a result of contamination or thermal cycling. The internal pore volume, pore surface area, and pore size distribution

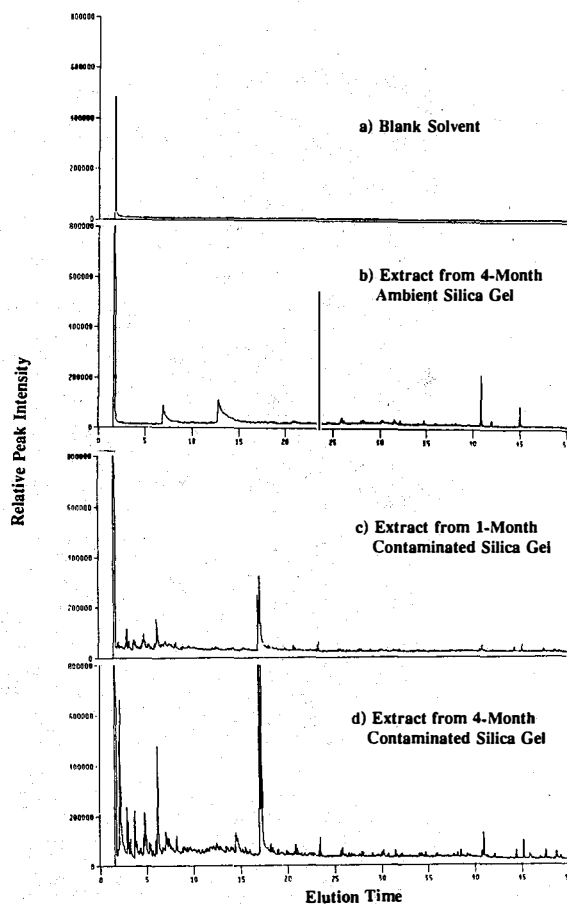


Fig. 4. Total ion chromatograph of several silica gel sample extracts and the blank solvent

are measures of the internal structure of a porous desiccant. The standard method for pore analysis is the nitrogen adsorption/desorption technique used in this study. The following five silica gel samples of lot 1 were sent to a commercial laboratory for pore analysis: virgin silica gel, 1-month ambient, 1-month contaminated, 4-month ambient, and 4-month contaminated.

The following is a summary of the pore analysis work. All samples were degassed at 150°C . Then, nitrogen adsorption/desorption data were obtained at -195.8°C . Forty data points for desorption and 40 data points for adsorption legs of the nitrogen isotherms were obtained. The volume of nitrogen adsorbed or desorbed at various pressure ratios was obtained. The data were used to calculate the pore volume, the Brunauer-Emmett-Teller (BET) pore surface area, the micropore volume, the microsurface area, and the average pore radius. Micropore volume and surface area are indications of how many of the pores are micropores (with radii below 20\AA). Table 2 presents a summary of the pore analysis of five samples of silica gel. The values presented for the virgin samples are within the data values found in the literature for Grade 40 silica gel.

Table 2 shows that the micropore volume and surface area of are very close to BET (or total) surface area and total pore volume, respectively, for all the samples. This indicates, as expected, that most of the pores in the samples are micropores (with radii less than 20\AA). The BET surface area of the 1-month ambient sample is about 17% lower than the BET surface area of the virgin sample.

TABLE 2
Summary of Pore Structural Analysis of Silica Gel Samples

Silica gel sample	S_t (m ² /g)	S_p (m ² /g)	V_t (m ³ /g)	V_p (m ³ /g)	r_p (Å)
Virgin	652	644	0.366	0.354	11.2
1-month ambient	543	529	0.333	0.315	12.3
1-month contaminated	368	356	0.255	0.239	13.9
4-month ambient	511	497	0.327	0.308	12.8
4-month contaminated	388	379	0.241	0.231	12.4

S_t Total BET pore surface area; S_p Micropore surface area;
 V_t Total pore volume; V_p Micropore volume; r_p Average pore radius.

This indicates the loss of a number of pores, most likely the smaller pores. The pore volume has been slightly reduced (by 9%), which also indicates the loss of small pores. The average pore volume of the 1-month ambient samples has increased by 10%, again an indication that the small pores are lost or not contributing. The small pores are filled or clogged because of contamination or collapsed due to thermal cycling. The loss in small pores can lead to a lower moisture capacity at low humidities. The 4-month sample showed more BET surface area and pore volume losses (22% and 11%). The average pore volume of the 4-month ambient sample increased by 14%.

The 1-month contaminated silica gel sample lost about 44% in BET surface area and 30% in pore volume compared with the virgin sample. Its average pore radius increased 24%. All of these indicate a loss of access to smaller pores in the sample, possibly caused by coverage and blockage with smoke contamination. Interestingly, the BET surface area of the 4-month contaminated sample was about 40% lower than virgin silica gel and 6% higher than the BET surface area of the 1-month ambient sample. The 6% is within the accuracy of the BET results, which is on the order of 5%. The pore volume of the 4-month contaminated sample is about 34% lower than that of the virgin sample. The average pore volume (compared with the virgin sample) increased by 11%. These results indicate that more pores were lost in the 4-month sample than in the 1-month contaminated sample.

The pore analysis of the samples indicated a loss of small pores for ambient and contaminated samples. As a result, the internal surface area and pore volume of these samples are lower than those of the virgin samples. The contaminated silica gel samples have lost more pores or adsorption sites than the ambient samples.

Sorption Capacity Measurements

The most important property of a desiccant for dehumidification and cooling applications is its ability to remove water vapor. This is usually quantified in terms of moisture capacity (kilograms moisture removed/kilograms dry desiccant) as a function of relative humidity at a given temperature, i.e., equilibrium isotherm. The moisture capacity of a desiccant may change upon thermal cycling and exposure to pollutants. We measured the adsorption capacities of virgin, ambient, and contaminated desiccant samples at 30.5°C and at various relative humidities (14%, 29%, 44%, 58%, and 73%). Then, comparisons among the moisture capacities of virgin, thermally cycled, and contaminated samples were made to quantify degradation effects.

To measure their moisture capacity, we removed the desiccant samples from the DCTF and installed them in the SERI heat and mass transfer test facility. The samples were first dried for 60

hours with air (the same conditions under which they were dried before the contamination experiment). The dry weight was measured with a precision balance and compared with the pretest dry weight to establish how much contamination was deposited on each desiccant.

Then, the samples were exposed to humid air at desired conditions (temperature, humidity) for about 20 h. The samples were removed and sealed, and their weight gain was measured with the balance. The moisture gain was obtained by subtracting weight gain from weight of contamination, described in the previous paragraph. The weight of moisture gain divided by the pretest dry weight of the desiccant provided the moisture capacity of the sample. The detailed experimental procedure of sorption capacity measurements can be found in Pesaran and Bingham (1989) and Pesaran and Dresler (1990). Here only selected data on silica gel and activated alumina are presented.

Typical results of capacity measurements of virgin, ambient, and contaminated samples of silica gel (Davison Grade 40) for both

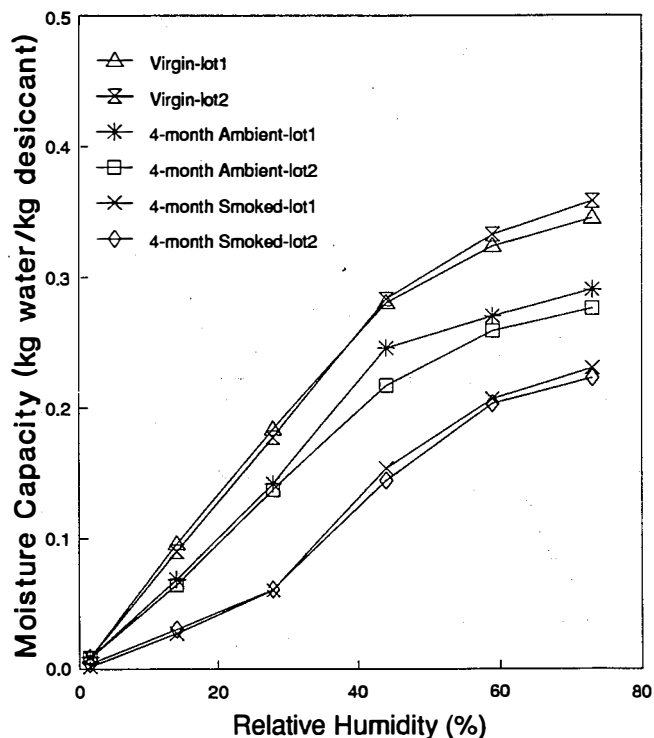


Fig. 5. Comparison of capacities of lot 1 and lot 2 of silica gel for virgin, ambient, and smoked samples

lot 1 and lot 2 at 30.5°C are given in Figure 5. First, we observe that lot 1 and lot 2 capacities are similar; deviations from lot to lot for this grade of silica gel were small (less than 6%) and within the experimental uncertainty. We also observe that the capacity results are independent of the location of the sample tube in the test cells. Figure 6 shows how much capacity of contaminated or ambient sample was reduced relative to the capacity of the virgin sample (percent drop from initial virgin capacity) for silica gel lot 2 as a function of time.

From the sorption measurement results (Figures 5 and 6, and others not shown) the following major observations can be made:

- Because of thermal cycling and exposure to ambient air, the sorption capacity of silica gel decreased with time. The percentage drop from the capacity of virgin samples was between 5% and 30%. Most of the capacity loss occurred in the first month of testing.
- Because of thermal cycling and exposure to cigarette smoke, the sorption capacity of silica gel decreased significantly with time. The percentage drop from the capacity of virgin samples was between 30% and 70% due to the combined effects of thermal cycling and exposure to cigarette smoke. Most of the capacity loss occurred in the first month of testing.
- The loss in capacity caused by cigarette smoke was about 20% to 50%.
- The capacity loss because of thermal cycling and exposure to smoke is more apparent at low relative humidities.

The losses found in the sorption capacity of ambient and contaminated silica gel samples are consistent with the results of other tests, i.e., XPS, GC/MS, and pore analysis.

The results of sorption capacity measurements of activated

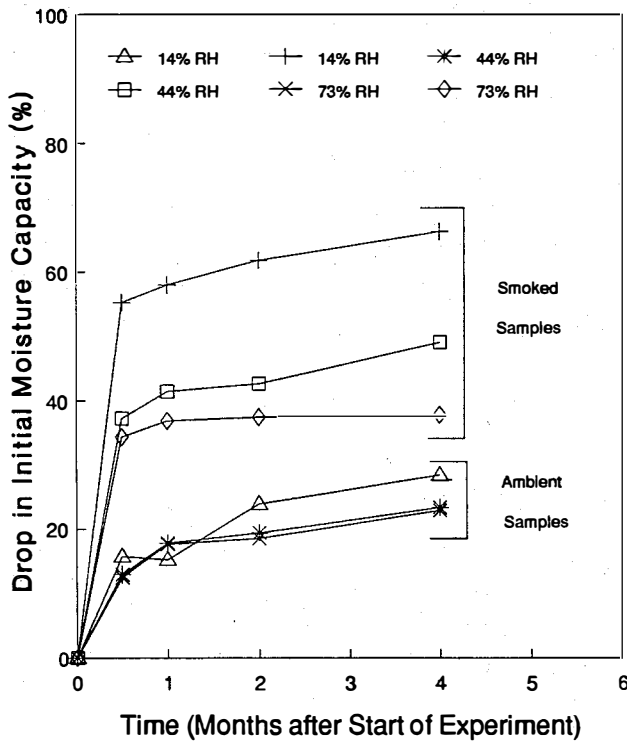


Fig. 6. Capacity loss of lot 2 silica gel vs. time for ambient and contaminated samples (at 30.5°C and various relative humidities)

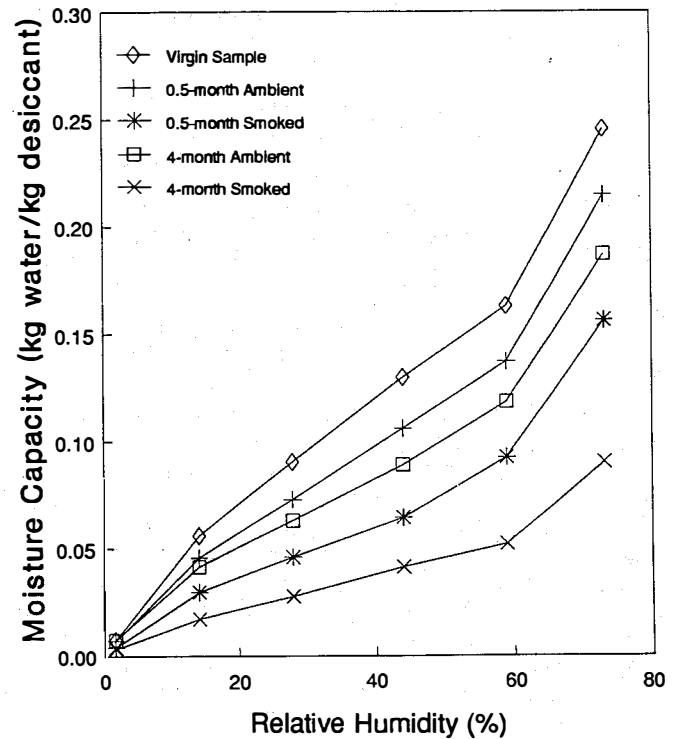


Fig. 7. Capacity of activated alumina for virgin, ambient, and smoked samples at 30.5°C

alumina from Adcoa are presented in Figures 7 and 8. From these results, we observe the following:

- Because of thermal cycling, the sorption capacity of the ambient sample of activated alumina drops with time. The percentage drop from the capacity of virgin samples is between 10% and 30%.
- Because of thermal cycling and exposure to cigarette smoke, the sorption capacity of activated alumina decreases. The percentage drop from the capacity of virgin samples is between 40% and 70% due to the combined effects of thermal cycling and exposure to cigarette smoke.
- The loss in capacity because of cigarette smoke is about 20% to 50%.
- The loss in capacity due to thermal cycling or cigarette smoke increases with the time of exposure. However, most of the capacity loss occurred in the first month of testing. After that, the capacity loss was not significant.

Similar trends were found from the capacity measurements for other desiccants (Pesaran and Dresler 1990). Their detailed presentations are beyond the scope of this paper. Table 3 summarizes the results of the capacity losses for all the desiccants. The degree of capacity loss depends on the type of desiccant. Generally, capacity loss of a desiccant because of thermal cycling and exposure to ambient air is about 5% to 30%. The capacity loss because of the combined effect of thermal cycling and exposure to cigarette smoke is between 20% and 70%. The degree of degradation due only to cigarette smoke was generally between 20% and 50%.

TABLE 3
Comparison of Moisture Capacity Losses of Virgin, Ambient,
and Contaminated Desiccant Samples

Desiccant	Ambient	Contaminated	Difference	Most Loss Occurred
Silica gel	10-30%	30%-70%	20%-50%	In 1 month
Mol. sieves	-- ^a	30%-70% ^a	30%-70% ^a	In 1 month
Activ. alumina	10%-30%	40%-70%	20%-50%	In 1 month
Activ. carbon	5%-20%	15%-50%	10%-40%	In 1 month
LiCl matrix	10%-30%	25%-70%	5%-40%	In 2 months

^aThe capacities of ambient samples were taken as the baseline.

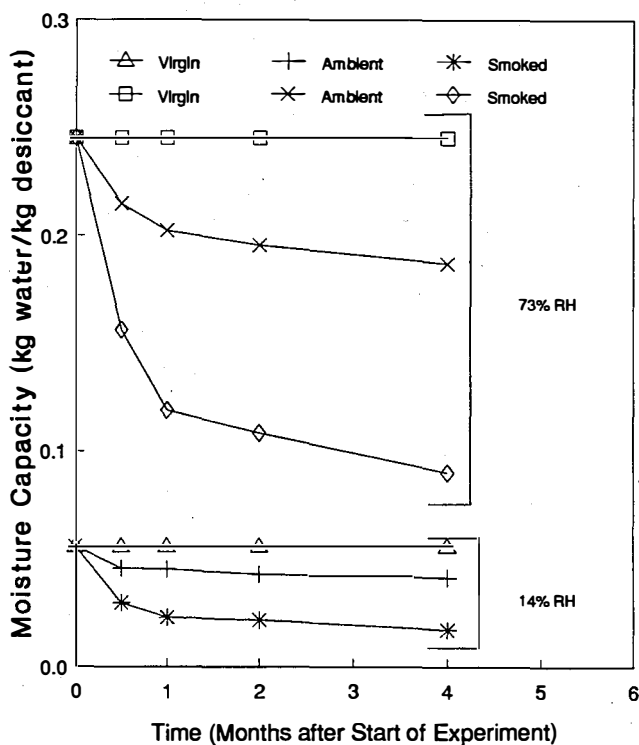


Fig. 8. Capacity of activated alumina samples vs. time at 30.5°C and relative humidities of 14% and 73%

CONCLUDING REMARKS

A desiccant contamination test facility was fabricated to investigate the effects of thermal cycling and contamination (cigarette smoke) on desiccant materials suitable for desiccant cooling applications. One hundred desiccant samples from six different materials were exposed to humid ambient air. Another set of similar desiccant samples was exposed to humid ambient air charged with cigarette smoke. Both sets of samples were thermally cycled between adsorption and regeneration temperatures. After exposure for several months, the moisture capacities of all the

samples were measured as a function of time using a gravimetric technique. In addition, a few of the samples were analyzed using x-ray photoelectron spectroscopy (XPS), gas chromatography/mass spectrometry (GC/MS), and pore size measurements.

The sorption capacities of the desiccant samples tested in the ambient test cell and in the contaminated test cell were lower than the sorption capacities of virgin samples. The degree of loss in capacity in both cases depends on the type of desiccant. The degree of degradation due only to cigarette smoke was generally between 20% and 50%. The moisture capacity loss from the combined effects of thermal cycling and cigarette smoke (the worst indoor pollutant) of all desiccants was significant (20% to 70%). For most of the desiccants analyzed, the capacity loss occurred during the first month of testing. After that initial fast drop in the first and second months, the rate of capacity loss with time was slower. The capacity loss for the microporous desiccants was more apparent at lower humidities. The data from XPS, GC/MS, and pore structural analyses were consistent with the capacity losses observed in the sorption capacity measurements.

It should be noted that say a 40% drop in sorption capacity of a desiccant does not translate to a 40% decrease in the performance of a desiccant cooling system. In another study (Pesaran and Penney 1990), the obtained data on silica gel from this study were used to predict the performance losses of a ventilation cycle desiccant cooling system. Assuming that the desiccant degrades according to the data on a 4-month contaminated silica gel sample (30% to 70% drop in moisture capacity), it was found that the loss in the performance could be between 10% to 35%.

This work has shown that desiccant degradation may occur in a desiccant cooling system. The actual loss in performance depends on the type of degradation. A design engineer has to account for losses that can occur with virgin desiccant due to thermal cycling and contamination (about 20% to 30%). Adjusting rotational speed of a dehumidifier is an easy solution to reduce performance losses caused by some types of degradations (Pesaran and Penney 1990). However, the rotational speed cannot be arbitrarily adjusted because it may adversely affect the performance. In addition to adjusting rotational speed, other strategies to alleviate desiccant degradation are filtering, replacing, cleaning, and deep regeneration (occasional high-temperature burn-off). Further investigation is required before final conclusions on these strategies can be drawn.

The experimental and system simulation work on contamination and degradation of desiccants needs to be continued to obtain reliable desiccant dehumidifiers with long service life.

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