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Dimensional stability of adsorbents subjected to thermal cycling

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Abstract The dimensional stability of adsorbent beads subjected to varying temperature conditions must be understood to assess the effect of thermal cycling on both the adsorbent and the structure that contains it. Most of the literature on the coefficient of thermal expansion (CTE) of adsorbents relates to zeolite crystals or clusters of crystals with application to membranes. Such crystals or powder materials have been shown to exhibit both positive and negative volume expansion coefficients depending upon the temperature range. This duality in the CTE with increasing temperature and the large variation in the CTE magnitude for a given zeolite structure suggest that the dimensional stability of zeolite crystals under varying thermal conditions is not likely a good indicator of the thermal stability of agglomerated zeolites. In this study, a method has been developed and applied to measure the CTE of activated alumina and 13X molecular sieve adsorbent beads. A McBain gravimetric microbalance was modified in a simple manner to be used as a dilatometer. The method was validated by measuring the CTE of a 316 stainless steel rod and showing that the measured CTE of this study agreed with the published CTE within 3.3 %. Average CTEs for alumina and 13X adsorbents were determined as 4.88×10^{-6} and 2.96×10^{-6} mm/mm/ °C, respectively for the range of temperature 20–400 °C.

Keywords Coefficient of thermal expansion · Activated alumina · 13X molecular sieve · Thermal stability of adsorbent beads

1 Introduction

Agglomerated adsorbents are commonly employed to purify gases on a large scale utilizing for example pressure swing adsorption (PSA) or temperature swing adsorption (TSA) processes. Adsorbent beds may be configured to conform to various vessel geometries within which the adsorbent is contained and/or constrained by not only the vessel walls but also by internal support structures inside the vessel. When subjected to significant changes in temperature (as in TSA processes), the vessel walls, internal structure and adsorbent expand and contract. The relative movement of these components results in non-negligible forces applied to both the adsorbent and any internal support structures within the vessel, e.g. screens, perforated plates, etc. If designed improperly, the support structure may fail or separate at the vessel interface and allow adsorbent particles to escape at the edges or through damaged parts of the support. Excessive forces may also result in crushing the adsorbent particles. The thermal expansion and contraction of a packed bed depends not only upon the CTE of the packing material but also upon the configuration of the packing and constraints imposed by the containment structure. The manner in which the CTE enters into the structural calculations involving the bed and vessel containing it depends upon the bed and vessel configurations, as well as the representative force balance equations. In order to perform a complete analysis of such a system, the coefficient of thermal expansion (CTE) of the adsorbent is one of many properties required.

Most of the literature associated with the CTE of adsorbents relates to zeolite crystals or clusters of crystals with application to membranes. Such crystals or powder materials have been shown to exhibit both positive and negative volume expansion coefficients depending upon

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the temperature range. Breck (1974) gives the CTE of hydrated NaA zeolite as 6.9 × 10^{-6}/°C for a range in temperature varying from −183 to 25 °C. Attfield and Sleight (1998) report a strong negative CTE ($\alpha = -4.2 \times 10^{-6}$/ °C) for dealuminated Y (DAY) over the temperature range 25–573 K. In their review of zeolite membranes, e.g. using siliceous zeolites such as MFI, Caro et al. (2000) indicate that the CTE for a single zeolite can vary from $+10 \times 10^{-6}$ to -10×10^{-6}/ °C. They further explain that the volume of the unit cell goes through a maximum, expanding with increasing temperature in the low-temperature phase and then contracting with increasing temperature in the high-temperature phase. The primary means of determining the CTE for these powdered zeolites is by using X-Ray diffraction to determine the zeolite unit cell dimensions and thus the change in the unit cell volume. The duality in the CTE with increasing temperature, the large variation in the CTE magnitude for a given zeolite structure and the variation in the CTE across various zeolite structures suggest that the dimensional stability of zeolite crystals under varying thermal conditions is not likely to be a good indicator of the thermal stability of agglomerated zeolites.

It was suspected that agglomerated adsorbents, i.e. adsorbent powder combined with a binder and formed into spherical beads, may not possess the same thermal expansion characteristics as the unit cell of zeolite crystals when subjected to thermal swings. Because the open literature is sparse with respect to the CTE of agglomerated adsorbent materials, a method was devised to measure this property. CTE results have been obtained for X-type zeolite and activated alumina adsorbents typically used in TSA purification processes. To our knowledge, such information has not been previously reported.

Various means for measuring the CTE of materials have been described by Rapp (1990). These include mechanical dilatometers, various types of interferometers, laser optical comparator, etc. Instruments incorporating some of these methods are commercially available. Typically, a certain size and/or shape of the sample are required in order to support the sample in the instrument. Moisture adsorption can also affect the measurement of the CTE. None of these instruments are particularly well-suited to the measurement of the CTE for spherical adsorbent beads of 1 to 5 mm diameter. There is then not only a practical need for the CTE of agglomerated adsorbent materials, but a means for measuring the CTE is also required.

The primary intent and scope of this study is to establish realistic values for the CTE of agglomerated adsorbents by developing a simple, practical and reasonably accurate method for measuring CTE, determining whether the CTE is positive or negative and measuring the CTE for two representative adsorbents of commercial significance. This study is neither exhaustive with respect to the type of adsorbents (or other agglomerated materials of interest), nor is it extensive enough for a rigorous statistical analysis. Nevertheless, the methodology has been carefully developed and validated to insure reasonably accurate CTE results for materials of the appropriate size and shape. The method as presented is limited to adsorbents in the form of spherical beads (and perhaps cylindrical extrudates having straight walls and uniform diameter). Irregular shape materials would not be well-suited to the cylindrical tube.

2 Experimental

2.1 Materials

Two different adsorbents are used in this study: activated alumina D201 and molecular sieve13X APG II supplied by UOP (Des Plaines, IL). These adsorbents are in the form of spherical beads. The sphericity of these beads is generally equal to or greater than 0.90. Both the alumina (5 × 8 mesh) and sieve (8 × 12 mesh) adsorbents were screened (11 × 12 mesh) to obtain a narrow size distribution of beads with a diameter between 1.7 and 1.85 mm.

For validation purposes, the CTE of a 1.588 mm (1/16in) diameter 316 stainless steel rod was measured using the same method as applied for the adsorbent. The adsorbent beads and stainless steel rod were contained within precision bore (2.0 ± 0.025 mm internal diameter) borosilicate glass tubes during the experiment.

2.2 Apparatus

A simple dilatometer was configured for this study by temporarily modifying a McBain O_2 adsorption apparatus (Allen 1997; Bolton 1976; McBain and Bakr 1926). Although this instrument is designed as a gravimetric microbalance, it already incorporates key elements of a dilatometer, i.e. means to support and heat samples and an accurate measurement of displacement. The McBain system also includes the ability to operate with the test sample under vacuum conditions, particularly important in this study in order to obtain the CTE of adsorbent beads in a dehydrated state. This last feature may not be a part of standard dilatometer instruments.

When the McBain apparatus is used as a gravimetric microbalance, the change in length of an accurately calibrated quartz helical spring is converted to a change in mass resulting from adsorption of O_2 (or other probe molecules) by an adsorbent suspended in a glass bucket at the end of the spring as illustrated in Fig. 1. The change in spring extension is measured using a cathetometer (with 0.01 mm precision). A cathetometer is a device for

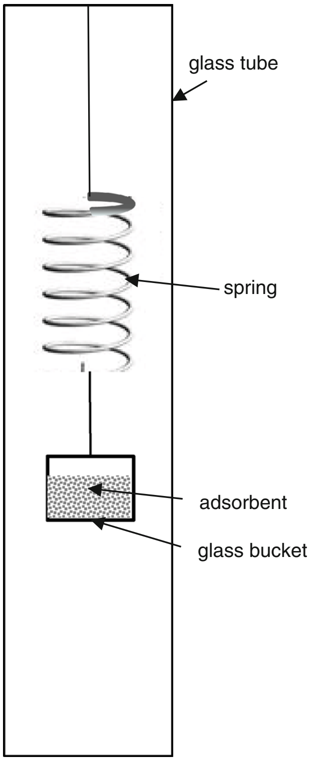

Fig. 1 Traditional McBain configuration

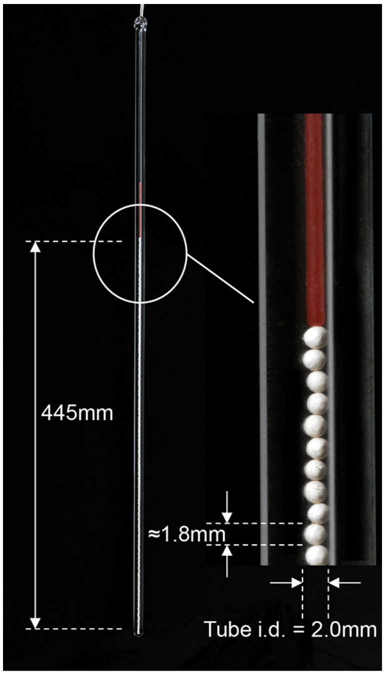

Fig. 2 Column of beads in sample tube

measuring the vertical distance between two points. It consists of a horizontal telescope mounted to a rigid vertical column with an accurate scale attached. The device is leveled and the telescope can move up and down the column to site the position of various reference points in the sample cell or tube. A heating mantle is fit to the outer glass tube attached to the vacuum manifold. The apparatus is capable of operating with multiple tubes attached to the vacuum manifold.

The standard McBain apparatus was modified by removing the calibrated spring and adsorbent weighing pan and replacing with a custom hanger and a precision bore glass tube containing a column of beads as shown in Fig. 2. The 2.0 mm glass sample tube is suspended from a hanger attached to the top of a bar mounted across the top of the outer enclosing tube. In the modified apparatus, the change in height of the column of adsorbent beads contained inside the precision bore glass tube is measured as the temperature of the system is increased. This change in height of the column of beads must be determined relative to the expansion of the glass tube containing the beads. The sample was maintained under vacuum during the measurement.

2.3 Method of measurement

Approximately 250 beads (screened to 11 × 12 mesh and pre-selected for uniform sphericity) of one of the adsorbents are inserted individually into the 2.0 mm precision bore glass tube. The column length and large number of beads insures a more representative and accurate measurement of CTE compared to methods designed to measure a single sample (bead). The column of beads is verified to remain free to move inside the tube with the addition of each new bead. The clearance between the surface of each bead and the inside wall of the tube is sufficient to allow for imperfections in sphericity and thermal expansion of the beads, but small enough so that the beads remain in single file. The bead column remained free to move vertically within the glass tube during most of the thermal expansion experiments. The initial height of the column of beads is approximately 445 mm. A small red borosilicate glass indicator rod is placed on top of the column of beads as shown in Fig. 2 and in the schematic of the test system in Fig. 3. Part of this indicator rod is in the heating zone, while the remaining part is above the heating zone with its top edge visible to the cathetometer. The position of the top of the glass indicator rod (point E) is measured initially at room temperature, at each incremental step in temperature and at its final position at the end of cooling. The sample(s) is activated by heating slowly to 400C under vacuum overnight. This is sufficient to remove essentially all of the moisture from these hydrophilic adsorbents. This insures that the CTE will be determined at a reproducible dehydrated state of the material. The system is allowed to cool to ambient temperature, but the vacuum is maintained. Temperature was measured using a

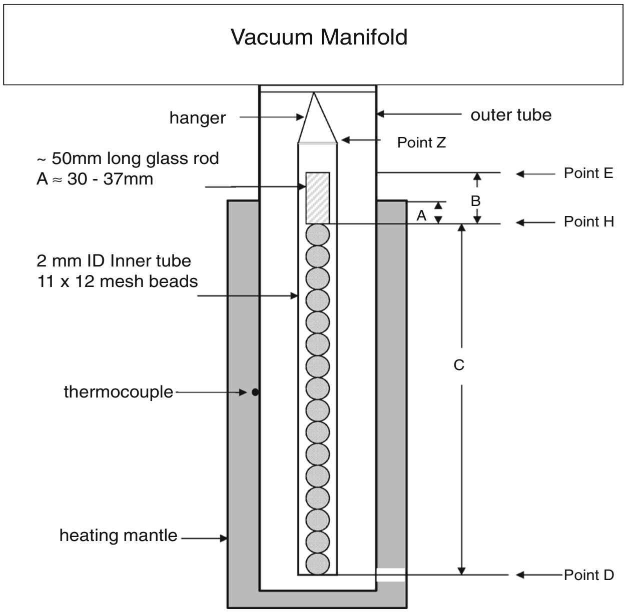

Fig. 3 Schematic of modified McBain system used for CTE measurement—single test section (not to scale)

thermocouple placed between the heating mantle and the outer glass tube.

The CTE experiment is initiated from a reference (ambient) temperature of 20 or 25 °C. Height measurements for points E, D, H and Z (shown in Fig. 3) are taken relative to a fixed datum established at the base of the cathetometer. After the initial measurements at ambient temperature, a second set of measurements is taken at a temperature of 50 °C, i.e. the amount of displacement that occurs for temperatures below 50 °C is generally below the precision of the cathetometer. Subsequent sets of measurements were made at 50C increments up to 400 °C.

2.4 Validation

The method was validated by measuring the CTE of a stainless steel rod contained within the precision bore glass tube and comparing the measurement to the well-known CTE for this material. The stainless steel rod offers the advantages of a known CTE, a uniform diameter that will fit within the glass tube and has none of the issues (e.g. sphericity, imperfect stacking, sticking to the walls of the tube, activation to remove moisture, etc.) associated with adsorbent beads. As a result, the methodology and apparatus are clearly validated as a means for determining CTE independent of the above factors for which such non-uniform effects would be difficult to quantify. It is also possible to determine the CTE of the precision bore borosilicate glass tube from the displacement measurements and compare this value to the known CTE for borosilicate glass.

3 Results and discussion

The thermal expansion of a material is governed by the following equation:

$$\alpha = \frac{1}{L}\frac{dL}{dT}, \tag{1}$$

where α is the linear CTE, L is the original length of the material at the reference temperature and the differential term expresses the incremental change in length for an incremental change in temperature (T). This relationship is typically approximated as follows when the CTE does not change that much over the temperature range of interest, i.e. α is constant:

$$\alpha = \frac{1}{L}\frac{\Delta L}{\Delta T}. \tag{2}$$

For the purpose of these experiments, ΔL, L and ΔT will be measured for each of the materials in the test chamber. It is also assumed that the adsorbent beads, stainless steel bar and borosilicate glass materials are isotropic and all have a positive CTE over the temperature range of 20–400 °C. Whether or not the CTE for the adsorbent beads is indeed positive is to be verified.

It is apparent from the configuration of the test sample, sample holder and glass indicator bar illustrated in Fig. 3 that during heating the precision bore glass tube will expand downward and the sample (column of beads) inside this tube will expand upward. The top of the glass sample tube (point Z) remains at the same position throughout any given test, i.e. as verified by measurement. The change in position of the top of the glass indicator bar represents the relative change in expansion of the glass sample tube and the sample it contains. The change in length of the glass indicator bar, part in the heating zone and part outside the heating zone, is exactly offset by the change in length (in the opposite direction) of the equivalent length portion of the precision bore glass sample tube that surrounds the indicator bar. Both materials are borosilicate glass. Generalizing for various materials, Equation (2) becomes:

$$\alpha_i = \frac{\Delta L_i}{L_i \, \Delta T} \tag{3}$$

where the subscript i represents the material type, e.g. adsorbent (beads), glass (g), stainless steel rod (ss). It can be shown then that the expansion of the bead column (ΔL_{beads}) is the sum of the change in position of the top of the glass indicator bar (δ_E) and the expansion of the precision glass tube (ΔL_g) containing the bead column:

$$\Delta L_{beads} = \delta_E + \Delta L_g \tag{4}$$

δ_E is measured from the reference point E (at the reference temperature) and may be negative (glass rod

Table 1 Test materials and coefficients of thermal expansion

Material	L_i (Fig. 3)	CTE (α_i) mm/mm/°C (reference)	CTE (α_i) mm/mm/°C (this study)	CTE (α_{avg}) mm/mm/°C
Glass tube (borosilicate)	C + A	3.25×10^{-6} (Green 2008)	2.82×10^{-6} 2.90×10^{-6} 3.13×10^{-6} 2.99×10^{-6} 2.87×10^{-6} 2.65×10^{-6} 2.68×10^{-6} 2.64×10^{-6}	2.84×10^{-6} (avg. of 8 meas.) $\sigma = 1.74 \times 10^{-7}$
Stainless steel rod	C	18.2×10^{-6a} (Allegheny Ludlum 2006)	1.756×10^{-6} 1.760×10^{-6}	17.58×10^{-6} (avg. of 2 meas.)
Alumina	C	–	5.22×10^{-6} 4.53×10^{-6}	4.88×10^{-6} (avg. of 2 meas.)
13X APG II	C	–	2.88×10^{-6} 2.97×10^{-6} 3.05×10^{-6}	2.96×10^{-6} (avg. of 3 meas.)

[a] 20–500 °C

moves down) or positive (glass rod moves up) from this reference during heating. ΔL_g is the amount the glass tube expands downward, is always positive and is measured from the point D reference established at the reference temperature. For the stainless steel rod, ΔL_{ss} replaces ΔL_{beads} in Equation (4). The cumulative changes in ΔL_g, ΔL_{ss} and ΔL_{beads} are then plotted against temperature. The slope of this characteristic $\left(\frac{\Delta L_i}{\Delta T}\right)$ is inserted into Equation 3 to compute the CTE for that material. The value of L_i should be measured (dimension indicated in Table 1) at the beginning of each experiment for each material type.

3.1 Stainless steel rod and borosilicate glass

In order to verify the validity of this somewhat unconventional use of the McBain apparatus as a dilatometer, the CTE of a 316 stainless steel rod was determined by the method described above and then compared to the well-known CTE for this material. The results of the experiment are shown in Fig. 4. The steel rod expanded 2.9 mm over the temperature change of 20 to 400 °C. The ΔL vs. T characteristic was fit by linear regression ($r^2 > 0.996$) for each of the two tests performed. The CTE was nearly identical for the initial and repeat tests. The average experimental CTE (17.6×10^{-6}mm/mm/ °C) is 3.3 % less than the published CTE value (Allegheny Ludlum 2006) as shown in Table 1. Thus, the modified McBain instrument offers a convenient and accurate method for determining the CTE of various types of materials.

The expansion of the sample tube (ΔL_g) must be determined in all of the eight experiments as required to execute Equation (4). As a result, it is possible to independently estimate the CTE for borosilicate glass in each experiment. As shown in Table 1, the initial length of the glass sample tube (L_g) is taken as that part of the tube that is in the heating zone (A + C). The length of the sample tube outside the heating zone is approximately 165 mm, i.e. from the top of the heating mantel to point Z. α_g was determined from Equation (3) using ΔL_g and L_g determined from each of the eight experiments performed in this study. L_g varied from 474 to 481 mm, while the cumulative value of ΔL_g at 400 °C was an average of 0.50 mm, i.e. the maximum expansion of the glass sample tube. The CTE determined from these eight measurements varied from 2.64×10^{-6} to 3.13×10^{-6} mm/mm/ °C (mean = 2.84×10^{-6} mm/mm/ °C, std. dev. (σ) = 1.74×10^{-7}). This CTE value is about 12.8 % lower than

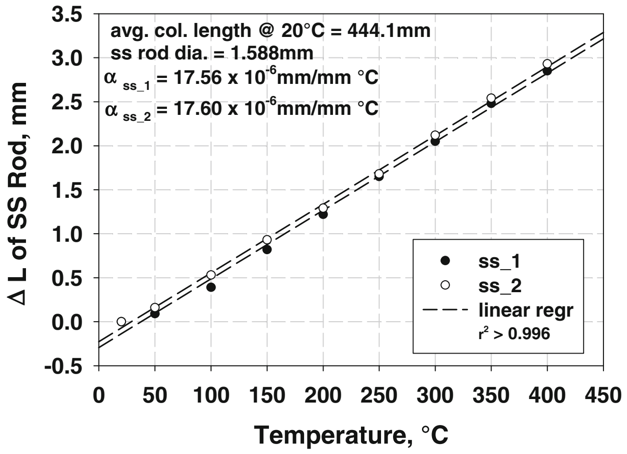

Fig. 4 Thermal expansion characteristic—316ss rod

the published value (Green 2008) as shown in Table 1 for borosilicate glass. One possible explanation for this discrepancy is that the glass tube is not uniformly heated due to the conductance of heat toward the open end of the tube where a length (165 mm) of the tube resides outside the heating zone. The resultant temperature gradient along the tube wall would result in less expansion due to the cooler temperature and is one possible explanation for the underprediction of the CTE. The sample steel rod inside the tube did not appear to be significantly affected by such a heat leak if it indeed exists. Because the sample and tube are suspended in a vacuum, radiation is the primary mechanism of heat transfer from the mantle to the sample. Unlike the glass tube, the sample has a much smaller conductive path to the unheated zone. One possible improvement to the method for future tests would be to perform the tests (after activation in a vacuum) in a dry N_2 (ambient pressure) rather than in a vacuum to promote conductive and convective heat transfer from the mantle to the sample. Based upon the stainless steel rod results and the lower thermal conductivity of the adsorbent beads, the CTE results for the adsorbents are expected to be sufficiently accurate for the purpose of this study.

3.2 13X sieve

Molecular sieve 13X APG II beads were activated under vacuum and then tested as described above ($L_i = 444.5$ mm). The large number of beads in the tube provides an average CTE for the adsorbent in contrast to that which would be obtained from single bead measurement. The maximum expansion of the bead column at 400 °C varied from 0.47 to 0.57 mm. The results of four experiments (all for the same column of beads) are shown in Fig. 5. Each set of data was fit by linear regression, although only a single regression line is shown (13X_1, $r^2 = 0.995$) for clarity. Experiments 13X_1, _3 and _4 are in good agreement with an average CTE of 2.96×10^{-6} mm/mm/ °C ($r^2 \geq 0.99$). The higher value of the CTE from experiment 13X_2 is the result of an apparent jump in ΔL_{beads} in the temperature interval 200–250 °C. This apparent discontinuity is believed to be the result of sticking or holdup of the beads in the upper portion of column for temperatures in the range of 100–200 °C, i.e. the column of beads separated and formed a small gap (0.10 mm or less) and then the upper part of the column released at 250 °C, resulting in a larger than expected δ_E at this temperature. Although such a small gap in the column of beads was not noticed during the experiment (the column of beads is obscured by the heating mantle), the data up to 100 °C and above 200 °C for 13X_2 are nearly parallel to the regression line for 13X_1, i.e. indicating most of the data (13X_2) suggests a lower value for the CTE than that obtained from fitting the entire set. For these reasons, the average CTE for experiments 13X_1, _3 and _4 is reported in Table 1 and is believed to best represent the expansion characteristic for 13X APG II.

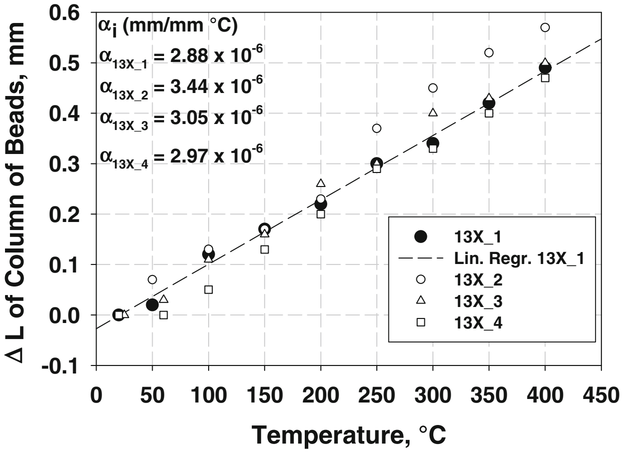

Fig. 5 Thermal expansion characteristics—13X APG II molecular sieve

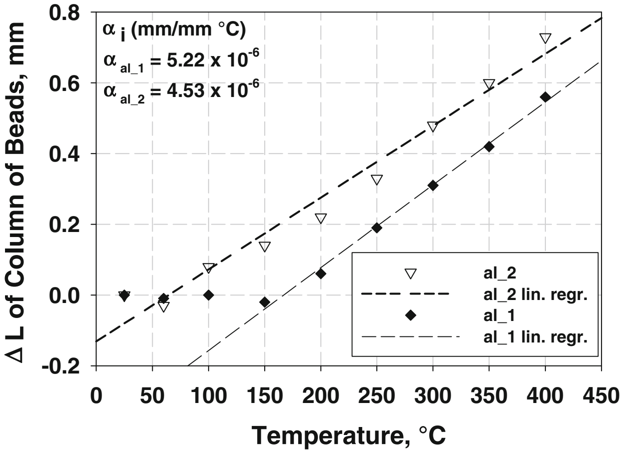

Fig. 6 Thermal expansion characteristics—D201 activated alumina

3.3 Alumina

A glass sample tube containing approximately 250 alumina beads was initially activated and then tested according to the procedures described above ($L_i = 448.7$ mm). The results are shown in Fig. 6. In the first test after activation, there was essentially no expansion of the bead column for temperatures up to 150 °C, i.e. the entire column of beads simply followed the glass expansion. Thereafter the column expanded linearly as shown. The data were fit for the temperature range 150–400 °C ($r^2 = 0.996$). Measurements were also taken during the cool down at 275, 150 and 25 °C and the contraction of the bead column followed the linear characteristic all the way to ambient temperature.

As a result, the initial reference at the top of the column was displaced downward by 0.21 mm ($T = 25$ °C). Typically, this difference was less than 0.03 mm in other tests. Although the beads moved freely in the column after initial loading, local sticking or holdup occurred as a result of the activation. The beads in the column were then free to move again above 150 °C in the first expansion test.

A repeat of the first test revealed the expected behavior as shown in Fig. 6. The linear characteristic extends over the entire temperature range ($r^2 = 0.973$). The offset in the position of the top of the column of beads at the beginning and end of the test was 0.02 mm. The CTE for each test is given in Fig. 6, and the average for this alumina is 4.88×10^{-6} mm/mm/ °C as shown in Table 1.

3.4 Measurement accuracy

The limited number of tests conducted with beads is not sufficient to evaluate the accuracy of the results in a rigorous statistical manner. Nevertheless, the accuracy can be estimated from several features of the measurements. These features include the precision of the cathetometer, the degree of linearity, the steel rod and glass tube validity results and the repeatability of the 13X bead results.

The precision of the cathetometer (0.01 mm) compared to the maximum expansion of the bead column over the temperature range of the tests provides one indication of the measurement error. In the tests performed with beads in this study, the change in column length at 400 °F varied between 0.38 and 0.57 mm, depending upon the adsorbent. Thus, the measurement uncertainty in determining this overall expansion is 1.8–2.6 %. Clearly, a longer column of beads and/or a greater temperature range would reduce this error further. The measurements with beads also proved to be reversible as the bead column returned to its initial length within 0.03 mm or less after heating and subsequent cooling, i.e. the initial column height of beads ($\approx$445 mm) remained unchanged to within 0.03 mm at the end of each heating/cooling cycle.

The variation in the results for 13X in Fig. 5 (experiments 13X_1, _3 and _4) at each temperature shows good repeatability over the entire range of the measurements. Similarly, the variation in linear fit to each set of these data (representing the CTE) is an indicator of the repeatability (experimental errors inherent in the method). The replicate steel rod data are almost identical to the initial data. Because the expansion of the glass tube containing the beads must be measured in each test as part of the procedure, there are eight repeat measurements for which the CTE of the glass tube may be extracted. The mean CTE for these measurements (2.84×10^{-6}) is included in Table 1 of the manuscript. The standard deviation (σ) for these data is 1.74×10^{-7}. This value of σ is 6.1 % of the mean CTE. Considering the above factors, it appears that the uncertainty in the data resulting from this study is no worse than 10–20 %, i.e. assuming one can clearly determine the occasional sticking bead condition and disregard the corresponding data.

Several factors that may influence the accuracy include the sticking or hold up of beads in the tube, sphericity of the beads and non-uniform temperature of the glass tube containing the beads. Strategies for minimizing or mitigating these effects have been briefly discussed in the text above.

4 Conclusions

The dimensional stability (as indicated by the CTE) of adsorbent beads in varying temperature conditions is required to assess the effect of thermal cycling on both the adsorbent and the structure that contains it. In this study, a method has been developed and applied to measure the CTE of activated alumina and 13X molecular sieve adsorbent beads. A McBain gravimetric microbalance was reversibly modified in a simple manner to be used as a dilatometer. The method was validated by measuring the CTE of a 316 stainless steel rod and showing that the measured CTE of this study agreed with the published CTE within 3.3 %. Average CTEs for alumina and 13X adsorbents were determined as 4.88×10^{-6} and 2.96×10^{-6} mm/mm/ °C, respectively for the range of temperature 20–400 °C.

The CTE determined for borosilicate glass from expansion measurements of the sample tubes used in the experiments differed from published values of the CTE for this material by 12.8 %. This difference suggests possible heat leak through the end of the glass tube located outside the heating zone with resultant temperature gradient along the length of the tube. Corrective actions include shortening the glass tube and/or performing the tests under a dry N_2 atmosphere. Although the selection of beads and loading the beads into the tube was performed with great care, sticking or holdup of the beads was observed in two experiments. It is recommended that multiple repeats of the test be performed to insure the accuracy of the final results.

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