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Journal of Chromatography A, 977 (2002) 213–223

JOURNAL OF CHROMATOGRAPHY A

www.elsevier.com/locate/chroma

Visualization of bed compression in an axial compression liquid chromatography column

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Received 17 May 2002; received in revised form 14 August 2002; accepted 14 August 2002

Abstract

The consolidation of a packed bed undergoing axial compression was studied in glass columns using an on-column visualization process. In this visualization process the refractive indices of the mobile phase (carbon tetrachloride) and the stationary phase (YMC C_{18} silica) matched perfectly, hence the otherwise opaque stationary phase became transparent to the eye. Alumina layers, which have a different refractive index, were placed at regular intervals along the column bed. These layers were therefore visible and their movement could be tracked during the axial compression of the bed. Consequently, the Young's modulus could be measured at three radial locations and at four bed depths below the head fitting. The results showed that the bed was heterogeneous in packing density, both radially (with the bed density increasing from the column center toward the wall) and axially (with the density increasing from the column top toward its center). Furthermore, the bed was shown to be non-symmetrical about the column axis. This was thought to be due to the column inlet head fitting making contact with the packing material on one side of the column first, rather than making contact with the entire cross section of the packing simultaneously.

Keywords: Bed compression; Axial compression; Stationary phases, LC; Consolidation

1. Introduction

The separation efficiency of chromatographic columns is directly related to the quality of the packing of their beds. This efficiency increases as the distribution of the packing density of the bed becomes more homogeneous across the column, both in the axial and in the radial directions. The fundamental observation that the packing of these columns is heterogeneous is now well documented [1]. Conclusive and consistent reports were published on this issue by Knox et al. [2], Eon [3], Baur and co-workers [4,5], Farkas and co-workers [6,7], Tallarek and co-workers [8–10] and Fernandez et al. [11]. Therefore, it has become the aim of the manufacturers of chromatographic columns to produce homogeneously packed beds in order to obtain highly efficient columns. However, no such columns exist at this time. The problem is important for analytical columns because there is a close relationship between the column efficiency and the analysis time. It

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0021-9673/02/$ – see front matter
PII: S0021-9673(02)01273-6

is critical for preparative columns which have been historically much less studied, a fact that is somewhat perplexing because it is in this field that improvements in column performance would potentially translate into the largest costs reduction.

The consolidation of packed beds, whether they be columns prepared using slurry packing methods or columns prepared through the application of a dynamic mechanical compression (axial or radial compression columns), relies on the application of mechanical stress to force the particles to come to rest in a configuration that resists further movement. Ideally, this packing configuration should be close packed, all particles making contact with the maximum number of neighboring particles and the flow channels from the inlet of the column to the outlet of the column having the same length and the same volume. In reality there are several reasons why such a close packed configuration is impossible to obtain. In the first instance, the particles of any packing material are not monodisperse but have a size distribution having a finite width. Hence, a regular, geometrically close packing is impossible. Also, the wall region of the column establishes a degree of heterogeneity within the column that extends deep into the column bed. This has now been well documented and two factors have been described that contribute to this heterogeneity [12]. The first is associated with the geometry of the wall region. Because the wall is rigid and smooth, the particles cannot penetrate into the wall and close packing near the wall is not possible. Consequently, the void fraction increases in the immediate vicinity of the wall. Secondly, as compression is applied to the column bed, stress builds up between the particles that are in contact. Friction prevents them from sliding against each other or from rolling. This stress is translated from particle to particle and eventually to the column wall as the particles are forced harder and harder to the wall surface. Friction between the particles and the wall and friction between the particles themselves allows these particles to stick to the wall and to stick together. This leads to an increase in the packing density from the column center to the column wall.

Friction can also account for variations in the axial packing density. Once the particles are fixed in place during the consolidation process, friction between these particles greatly reduces the freedom of movement of these particles. If consolidation was not sufficient to achieve close packing in the first instance, clusters of particles may be grouped together in regions of high and hence also others of low packing density. Then, in order fully to consolidate the packed bed, there must be a mass reordering of the stationary phase so that regions of low packing density can be compressed. Jaeger et al. [13] eloquently described this process as the ''parking lot effect''. They then went on to summarize that, although friction between the particles may, on an individual basis, be quite insignificant, when taken on a whole for the entire column contents it provides an overwhelming resistance to particle movement. In other words ''gentle forces build strong bridges'' [13]. This considerable resistance to the sliding of the bed along the wall was demonstrated by Guiochon and co-workers [14–16].

However much friction contributes to packing heterogeneity, without it chromatographic columns would simply fail [14]. Without the bed applying a strong stress normal to the wall, the bed would not stick to the wall and the mobile phase would bypass the bed altogether. But this normal stress causes friction to appear as soon as an axial stress component appears. In some respects a chromatographic bed behaves in much the same manner as a spring. Application of pressure at column inlet causes the bed to compress. Without friction, this compression would be elastic to a large extent. Then, removal of the pressure would allow the bed to return to almost its original length [14]. If it were not for wall friction, bed compression would take place every time the column is subjected to compression stress, that is every time the inlet pressure is changed in order to adjust the flow-rate. A void would appear at the column entrance during use, only to disappear again when the flow was reduced.

So, here we are in conflict. Friction is required to keep the bed in place once packed, but at the same time, it is friction during the packing process that prevents the bed from becoming well packed and homogeneous in the first instance. Obviously then, friction should be minimized during the packing process, but maximized when the column is well packed. A task that has not been mastered yet.

This study provides direct visual evidence of the

bed consolidation process during axial compression in a liquid chromatography column. It was carried out using the visualization process previously described. The phase system was selected so that the refractive indices of the stationary phase and the mobile phase match exactly. Provided the column container is transparent, the bed becomes transparent to the eye. However, if a packing material that has a refractive index different from that of the mobile phase is placed in thin layer regions within the column bed, these regions of the bed become opaque and their movement during the compression process can be followed. This gives novel information on the consequences of the process of consolidation.

2. Experimental

2.1. Chemicals

Reagent-grade carbon tetrachloride was obtained from Sigma (St. Louis, MO, USA). Reagent-grade methanol was from Fisher Scientific (Fairlawn, NJ, USA). The stationary phase used was YMC C_{18} silica (YMC, Kyoto-Fu 613, Japan). The particles of this packing material are spherical, with a particle size distribution given as 15–30 μm and an average particle size of 21 μm. Alumina (50 μm average particle size, Cohesive Technologies, Franklin, MA, USA) was used as the opaque stationary phase, forming the layers within the column.

2.2. Columns and packing material

All chromatographic experiments were performed using a 100×17 mm I.D. borosilicate (Pyrex) glass column supplied by Omni (Cambridge, UK). The column end fittings were prepared in the laboratory, at the University of Western Sydney and were machined from Delrin plastic. These fittings included a fixed length outlet fitting and an adjustable inlet fitting that allowed axial compression of the column while preventing the formation of a void at the column inlet. Stainless steel frits having a diameter of 15.9 mm and a thickness of 1.57 mm were obtained from Bodman (Aston, PA, USA).

Alternating layers of methanol slurries of the silica and alumina packing materials were placed into the column and allowed to settle in the gravitational field. The silica and alumina layers were added separately and each layer was allowed to settle completely prior to the addition of any subsequent layers. The amounts of slurries added were such that a silica layer had a thickness of approximately 1 cm while an alumina layer had a thickness of approximately 0.1 cm. Once the required column length had been obtained, the methanol impregnating the column was replaced with carbon tetrachloride. Once carbon tetrachloride completely filled the column, the head fittings were assembled and inserted into the head of the column.

Axial compression of the column was then achieved using a hydraulic jack located at the outlet end of the chromatographic column. A detailed diagram of the column assembly is given in Fig. 1. The application of mechanical compression was recorded using a balance located below the jack assembly. During bed compression, both the outlet and the inlet end fittings were open, allowing the liquid within the column to flow freely from within the column cylinder. Note that in the experiments performed by Guiochon and co-workers [14–16], the slurry solvent was exiting the column only through the end opposed to the compression piston. Care was taken to ensure that carbon tetrachloride expelled from the column during compression flowed into the proper waste reservoir. Because of the breaking strain of the glass column, cylinder compression was restricted to 50 kg/cm^2. The axial compression must be carried out with extreme care to avoid collapse of the bed.

2.3. Equipment

Visualization of the bed compression was achieved using two Pentax ZX-M SLR 35 mm cameras fitted, one with a Tamaron 90 mm macro lens, the other with a Makinon 80–200 mm macro zoom lens. Kodak Elitechrome 200 ASA Professional slide film was used throughout. The film was developed by a commercial photographic dealer (Westcolour, Toongabbie, Australia). The photographic images were digitized using a Nikon CoolScan III (Nikon Melville, New York, NY, USA) film scanner. All images were acquired at the maximum resolution of the scanner (2700 dots per inch, dpi).

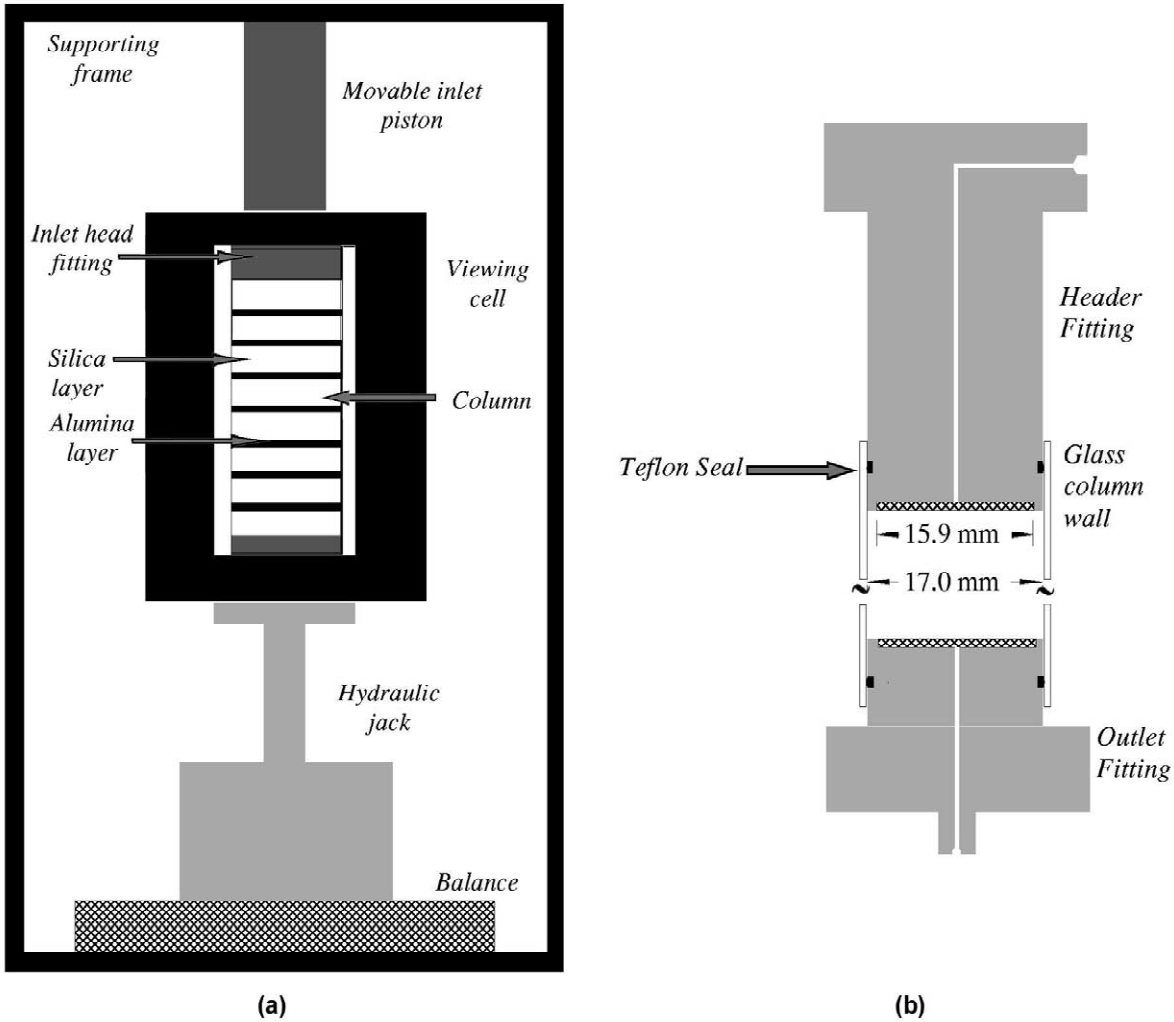

Fig. 1. (a) Diagram of the column contained in the axial compression system. (b) Diagram illustrating the inlet header and the outlet fitting designs.

Adobe Photoshop 5.0 (Adobe Systems, San Jose, CA, USA) was used to perform image manipulation. Further analysis was done using SigmaScan Pro 4.01 (Jandel Scientific, San Rafael, CA, USA) image analysis software.

2.4. Image collection and analysis

To improve visualization and minimize the cylindrical lens effect, the entire column assembly was placed into a rectangular box-shaped viewing cell filled with dichloromethane (a compound that has a refraction index very close to that of carbon tetrachloride but is much less toxic). This cell assembly was described previously [17,18]. This viewing cell had four windows, one on each face. The column was lighted through two of these windows and the cameras collected the images from the other two. The cameras were at right angles to each other. The local degree of bed compression was measured according to the relative movement of the alumina layers distributed throughout the column length. These measurements were made using the image analysis software at three locations across the col-

umn, using images collected from one camera angle only. These measurements were made at the wall on both sides of the column and in the central region of the column. The second camera served to confirm that the measurements were actually made in the central region of the bed. In order to obtain information from regions other than the central region of the bed, three cameras would be required and even then only half the column could be mapped.

3. Results and discussion

The photographs shown in Fig. 2 depict a consolidated chromatographic column viewed from two directions that are at right angles to each other. The thin, dark grey layers that are suspended within the column cylinder are the alumina layers. They are visible because the mobile phase, carbon tetrachloride ($n=1.54$), and the particles in the layer, alumina ($n=1.68$), have markedly different refractive indices. By contrast, the layers of silica that are contained between the layers of alumina and that make up the majority of the packed bed are not visible because the refractive indices of C_{18} silica and carbon tetrachloride match almost exactly, hence the otherwise opaque C_{18} silica becomes transparent to the eye. In the photograph depicted in Fig. 2, the layers of silica appear as light grey regions. Actually, these regions are transparent and appear as the same color as the light source mounted behind the column. Since the alumina layers are visible in the column cylinder, they serve as reference markers for the radial distribution of the vertical strains achieved during the axial compression of the column. As the bed compresses their movement can be followed.

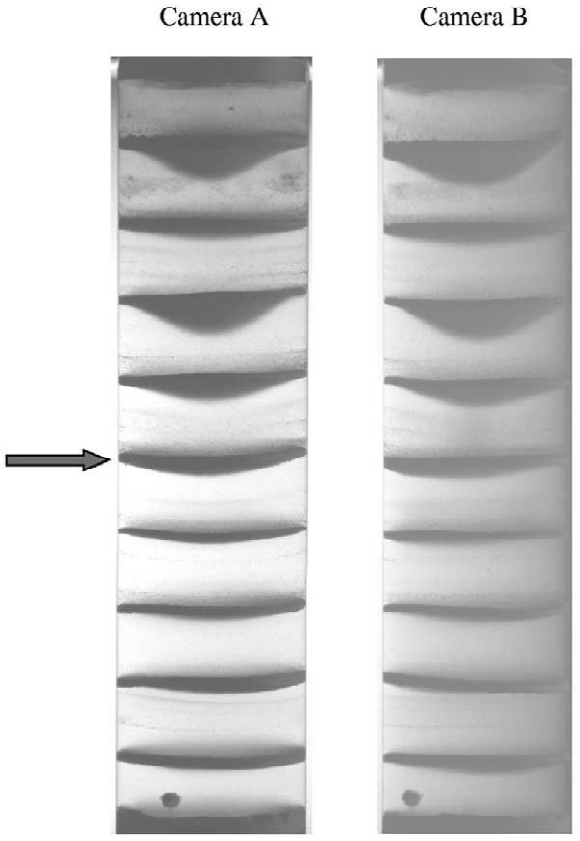

Fig. 2. Photograph of the chromatography column depicting the layers of alumina suspended between the layers of C_{18} silica.

The extent of the differential strain due to bed consolidation in the column is illustrated by the curvature of the alumina layers. If the layers were not curved but flat, there would be no differential strain, i.e., the corresponding sections of the bed would have moved as blocks (or, possibly, they would not have moved at all). Because the experiment demands that layers of alumina be laid down uniformly across regular sections of the column and, then only, that a compression force be applied to the column in order to compress the bed, the entire experimental setup is extremely sensitive to vibrations. Vibrations through an unconsolidated bed cause convection, hence a mass reordering of the layered column. The obvious result is a mix of the alumina and silica particles, and a bed that is opaque in carbon tetrachloride. The column must therefore be packed in the confines of the apparatus described in Fig. 1 in order to minimize vibrations. Even so, it proved quite difficult to lay down a column bed giving the results shown in Fig. 2. Furthermore, the columns must be prepared using methanol as the slurry solvent. Otherwise, the silica particles take several days to settle in the dense carbon tetrachloride, which causes particle size segregation throughout the bed. The requirement to pack the column in a solvent such as methanol further increases the difficulty associated with packing the bed because, during the first step of the preparation of a column, the slurry, first, and, then, the unconsoli-

dated bed are both opaque since the slurry solvent ($n=1.329$) and the packing material have different refractive indices. The layers become visible only when the slurry solvent is replaced by carbon tetrachloride.

The change in solvent from methanol to carbon tetrachloride is another difficult process as the bed instability is especially prone to manifest itself during this change in solvent. Convection and a mass reordering of the stationary phase is commonly observed. This flow of the packing particles may be explained by the strong density gradient associated with the vastly different densities of carbon tetrachloride ($\rho=1.594$) and methanol ($\rho=0.791$). This large density difference is responsible for flow instabilities that are well documented [19,20]. In an unconsolidated bed, this phenomenon may cause the formation of large eddies which could have a destructive effect on an unconsolidated bed, causing its reorganization by mixing. The resulting destruction of the bed is illustrated in Fig. 3, which illustrates the end result following the bed mixing that takes place when the column is not successfully equilibrated with the carbon tetrachloride. In the top section of the column the mobile phase contained in the column consists of suspended particles that have been disturbed in the solvent change. The middle section of the column is a mixture of silica and alumina particles, the layers of which have been disturbed and mass reordering of the stationary phase has occurred. Closer to the bottom of the column, the alumina layers begin to become more obvious, as these layers have largely escaped the mixing process. Although this type of column bed destruction occurred quite frequently, few photographic images of the process were recorded. The image depicted in Fig. 3 has been taken without solvent in the column reservoir and as a consequence the cylindrical lense effect distorts the image. Furthermore, and this is purely speculative as we do not have the photographic evidence to support this statement, we believe that the lower layers of the bed have become more closely packed than when they were originally laid down. We observed this phenomenon on several occasions, but it is perhaps an optical illusion. This problem becomes less serious when the carbon tetrachloride is pumped slowly through the bed (an observation that is consistent with our interpretation).

Fig. 3. Photograph illustrating the bed destruction following a change in solvent from methanol to carbon tetrachloride.

We prepared a total of eight columns with limited success before we were able to achieve the result shown in Fig. 2. Even then, the curvature of some of the alumina layers is slightly larger than we expected. It was impossible to predict the degree of curvature that would arise during the preparation of a bed. Whilst the success rate of preparing these columns was low, this does not mean that the reproducibility of the packing of axial compression columns is poor. The process is fast and convection of the suspension is frozen as soon as all the particles come in contact. In this study we successfully prepared only one column but the results obtained illustrate the bed consolidation process that took place in a column for which there was no convection of the unconsolidated bed. As we discuss later, the

results in this study agree very well with previous results reported in the literature on the compression of conventional axial compression columns.

Measurements of the distances between alumina layers were made by measuring the distance from the exact same radial location on the alumina layer to the column outlet fitting after each compression step. However, because of the opaque nature of the alumina, the measurement of distances between a layer and the outlet fitting cannot be carried out at any pre-defined radial location of the column cross-section—except along the wall regions. All the other measurements that were made within the column cross-section represent values averaged over finite regions, according to the lowest point of the alumina layers. It is therefore extremely important to consider the three-dimensional column aspect rather than a mere one-dimensional perspective. For that reason, we did not make measurements within the bed for layers below the fourth alumina layer. Although possible, these measurements would be biased. When viewed from the second camera angle, these lower layers are skewed toward the front of the column (i.e., toward the camera A). Hence, measurements based on the location of the minima would not reflect the central region of the bed but a region closer to the wall. The arrow in Fig. 2 depicts the fifth layer observed on camera B where the minimum is skewed to the front of the column when viewed through camera A.

At this point it is worth discussing the significance of the three-dimensional (3-D) column perspective in regards to the information on the movement of the alumina layers. As we have employed only two cameras at right angles, we cannot see what happens in the region behind an alumina layer with respect to the relevant camera, because the alumina layer is opaque, preventing the camera from recording any useful information. In order to gain information on a 3-D perspective, four cameras would be required. What we did observe is a two-dimensional (2-D) ''snap shot'' of the column bed. If the packing density of the bed is heterogeneous, this ''snap shot'' may change as the column is rotated around its axis. Consequently, the ability to reproduce the results in a reliable manner is somewhat limited and relies on aspects such as the precise alignment of the column inlet head fitting (see later) and the process of axially compressing the column. Because the alignment of the inlet head fitting is the most significant aspect with regards to the uniform compression of the bed but this alignment cannot be properly reproduced with the current experimental system, testing other columns in order to gain an understanding of the reproducibility of the packing process is prohibitive. Instead, we must rely on the fact that the general trends in the observed results strongly support previous findings reported in the literature [1–8,14–21].

In the present study, the bed was subjected to a total compression force of 1107 N, which translates into a pressure of 488 N/cm^2 (or 49.3 kg/cm^2). Despite the fact that the column was made of glass, the compression forces achieved in this experiment reflect those which are considered to be appropriate in the compression of axial compression columns prepared in stainless steel tubes [1,14–16]. Compression forces that are higher than those achieved in this work are generally considered to cause particle breakage [21]. Note also that stress do not convey across particulate solids as pressure does inside liquids [13]. The maximum value of the normal stress applied by the bed on the column wall is markedly lower than the average compression stress applied by the compression piston [22].

It is well known that, like all solid materials, chromatographic beds are elastic in nature. It is also well known that solid materials, when subjected to a compression stress, undergo an initial compression that is elastic (i.e., the relationship between stress and strain is directly proportional). Beyond a certain degree of compression, this relationship becomes nonlinear, compression reaches a stage in which the bed will not return to its original length upon release of the compression stress, and the bed has become inelastic. In the absence of wall friction, the bed would compress in proportion to the inlet pressure of the mobile phase, leaving a void volume at the column inlet. Since the bed would return to its initial length upon release of the inlet pressure, it would be hard to identify what happened and why poorly shaped peaks are recorded. To avoid the back-mixing of the sample that would take place in this void and that would explain the poor peak profiles, the chromatographer would seek to compress the bed to such a degree that the bed length would no longer vary during use and not expand when the flow is

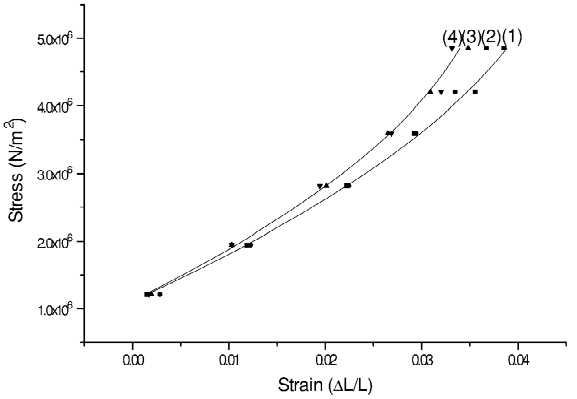

Fig. 4. Graphs of strain versus stress for the central section of the chromatography column illustrated in Fig. 2. (The numbers 1 to 4 are the ranks of the layers used for the strain measurements).

stopped. Unfortunately, compression to this extent would be hard to achieve and would, most likely, result in a substantial degree of particle breakage [21].

The graphs in Fig. 4 show the stress–strain relationships in different locations of the chromatographic column shown in Fig. 2. The numbering 1 to 4 in this figure refers to the rank of the alumina layer, from the movable column inlet head fitting down. Only the top four layers of this column were considered, for the reasons discussed earlier. Only the stress–strain relationship for the central section of the column is shown in Fig. 4 but the stress–strain relationships in the wall region are visually similar. In Fig. 4, we see that deviations from linear behavior begin as the stress is increased beyond ca. 2 MN/m^2. The upward curvature indicates the compression stress increases faster than the corresponding strain, which agrees with other results [14–16,22]. Despite the departure from linear behavior, the elastic limit of the column packing was not reached and no major bed collapse was observed. The average or apparent Young's modulus was determined from the linear region of these relationships. The values are given in Table 1. In almost all cases, the Young's modulus increased from the top of the column down to layer 4, a bed depth equivalent to ca. 45% of the column length. The Young's modulus was also at a minimum in the central section of the column (along the column axis), increasing toward the wall region. The averaged value of Young's modulus in the chromatographic column was 96 MN/m^2, a value that is within the range of those previously determined by Guiochon and co-workers [14–16,23], with typical values for spherical particles between 40 and 500 MN/m^2, depending upon the previous history (first or subsequent compressions), the packing solvent, and the type of packing material used (particle shape, size, and size distribution). Given the large particle diameter and the size distribution of our stationary phase, the values obtained in this study are consistent with these earlier results. For a general comparison with known materials, the Young's modulus for bone (compression) is 9 GN/m^2 and that for concrete it is 23 GN/m^2. Note that the detailed interpretation of these numerical results is difficult because the modulus reported are averages over significant volumes of the column bed. Also, measurements were not made in the lower half of the column.

Clearly the column bed shown in Fig. 2 is neither radially nor axially homogenous. The increase in the modulus of elasticity along the bed length, as we move further from the compression piston, indicates that the central region (and perhaps the lower region—although this could not be accurately determined) undergoes a greater extent of strain, and possibly more consolidation and compaction than the

Table 1
Young's modulus for the chromatography column that underwent compression as illustrated in Fig. 2

LHS at wall region		Center of column		RHS at wall region	
Bed depth (column length, %)	Young's modulus γ (MN/m^2)	Bed depth (column length, %)	Young's modulus γ (MN/m^2)	Bed depth (column length, %)	Young's modulus γ (MN/m^2)
8	92	13	85	8	90
20	100	22	89	20	97
30	102	35	96	30	92
43	113	45	95	43	103

top section of the column. Furthermore, the region nearing the wall also has a higher degree of compaction suggested by its higher value of the Young's modulus. The central region of the column (along its axis) is only moderately packed in comparison to the region near the wall. Previous results on the wall effect [12] and on the apparent diffusion coefficient of a droplet in the center of the column [24] support these results.

The consequences of the radial heterogeneity of a column bed on the radial flow profile of the mobile phase and the band broadening of solutes are nefarious to catastrophic. In order clearly to observe this effect, we prepared a separate axial compression column with no alumina layers, inserted a needle below the inlet frit, and injected a solute marker (I_2) at a radial location of 0.6*R* (where *R* is the column radius). By injecting the solute well below the inlet head fitting, we avoided any spurious flow associated with the solute passing through the frit. The photographs shown in Fig. 5 illustrate the progressive change in profile of the solute band as it migrates along the column. Initially, the solute entered the column as an almost perfectly spherical band. When the solute was close to the column exit, the part of the sample plug that was closer to the center of the column had traveled much farther than the part that was nearer the wall. This demonstrates that the velocity of the sample was higher near the center of the column than near its wall. Such an effect is illustrative of a column that has a higher packing density in the wall region and a lower packing density in the core region. That is, the bed is heterogeneously packed in the radial direction, which is consistent with our findings regarding the radial variation of the Young's modulus. We also injected samples at 0.3*R* and obtained similar results although with less significant differences in the flow velocity between the regions near the wall and near the column center (results not shown).

Aside from the variation of the packing density in various sections of the column, one other feature has been discovered during this study. That is, the column is not axially symmetrical as could have been expected from a bed packed in a cylindrical column. The Young's modulus on the left-hand side

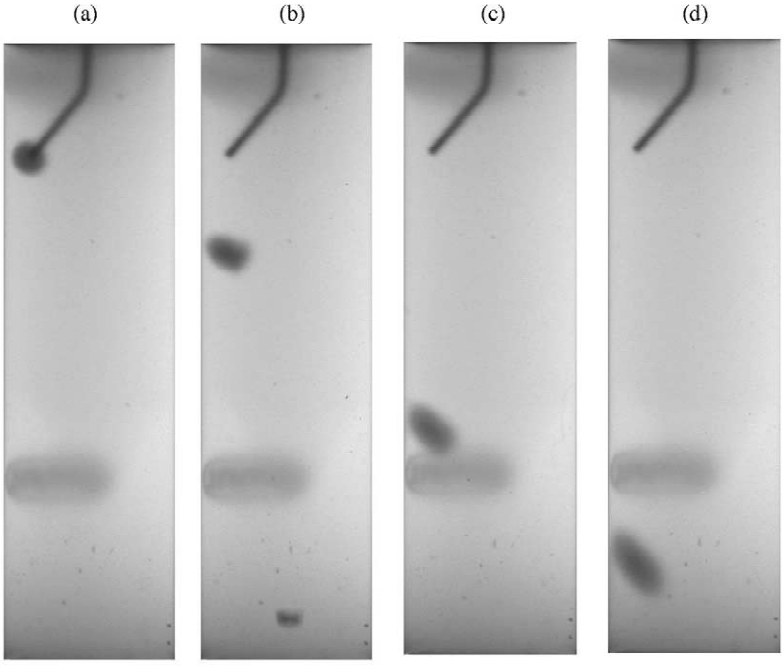

Fig. 5. Photographs illustrating the migration of an iodine solute injected at a radial location of 0.6*R* into an axial compression column. (a) Initial solute, (b) solute after 1 min, (c) solute after 3 min and (d) solute after 4.5 min.

of the column (Fig. 2) is greater than the Young's modulus on the right-hand side. This lack of symmetry, which is puzzling at first, can be explained by the fact that the inlet head-fitting was not perfectly parallel to the bed head in the initial stages of the consolidation. Hence the left-hand side of the column underwent a greater degree of compaction as the head fitting probably made contact with this part of the surface first. As a consequence, one important finding from this study is that when stress is applied to the compression piston of an axial compression column, the piston must be perfectly parallel to the bed head. If the stationary phase is allowed to settle under a gravitational field prior to application of the piston compression, extreme care must be taken to ensure that the stationary phase settles perpendicular to the wall. A better approach may be to begin compression of the bed immediately after the slurry is poured into the column tubing, thereby not allowing the particles to settle under their own mass in any way whatsoever. As such, the packed bed will fill the space within the tubing more uniformly. It is precisely the inability of reproducibly aligning (let alone perfectly doing it) the head fitting with the surface of the packing material, together with the fact that we record a 2-D ''snap shot'' of the column, that makes it essentially impossible to obtain information that characterizes the reproducibility of the column compression process. Every time a new column is made and tested, we obtain a different result, depending on where the images were recorded in relation to the way the head fitting was applied to the column inlet. It is clear, however, that the columns prepared in this study were all heterogeneous. Their general behavior was consistently so and columns prepared in other studies have also shown similar heterogeneity trends [1,6–8,14–21].

4. Conclusion

The very nature of the consolidation process in an axial compression column leads to a packed bed that has an inherently heterogeneous packing density. In the axial direction, the strain of the bed increases with increasing distance from the surface of the compression piston toward the column middle. In the radial direction, it increases from the column center to its wall. It is impossible to avoid the use of the end regions of the packing in the operation of a chromatographic column and it does not seem possible to cut them off. Similarly, using only the core region of the column, although technically possible [25], would result in significant economic losses. Furthermore, the packing density is radially heterogeneous, with the regions near the wall in general being more dense than the central section of the column. This type of variation in packing density is more detrimental to the column performance than axial variations in the packing density. Flat profiles will turn parabolic leading to a drastic reduction in column performance. The extent of the variation in the radial packing density will be dependent on the column diameter. Accordingly, a packed column seems to be destined to perform at a performance level that is well below the one that could be expected on a theoretical basis for a uniformly packed bed, unless better packing methods can be developed.

Another significant factor that was determined in this study was the importance of aligning the head fitting uniformly across the cross sectional area of the packing material. In the event that this fitting is not perfectly aligned, bed compression will take place such that the regions that are made contact with first are consolidated to a greater degree, hence contributing to the heterogeneity of the packed bed. A better and more consistent method of bed compression would be to begin compression of the packing material before the particles settle under gravity.

Acknowledgements

This work was partly funded through a University of Western Sydney (Hawkesbury) Internal Research Grant (32027210). One of the authors (V.W.) gratefully acknowledges the receipt of an Australian Postgraduate Research Award.

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