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Packing soft spheres: experimental demonstrations with hydrogels

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Abstract

We describe a number of different experimental set-ups that use hydrogel spheres to demonstrate dense packings of deformable spheres in various geometries. The arrangements are similar to those of bubbles in foams, drops in emulsions, biological cells, etc. The experiments are easy to perform in the class-room or an undergraduate science laboratory. They are described in the context of the history of packing problems to which this convenient system, not yet fully explored, can add significant new findings.

Keywords: soft spheres, hydrogel, packing fraction, dense packings, columnar structures, buckling

(Some figures may appear in colour only in the online journal)

1. Introduction

Using readily and cheaply available hydrogel beads, a wide variety of close-packed arrangements can be experimentally achieved. These range from hard-sphere packings to more compact arrangements brought about by external compression. The experiments that we describe are easy to set up and ideal for use in the classroom or in the undergraduate teaching laboratory.

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We describe five different types of experiments, making contact with current research problems. While packing experiments do not appear to have yet entered the canon of classroom demonstrations, they can make a good introduction to various concepts in mathematics and physics, including geometrical considerations, minimal surface area, surface tension, elasticity, computer simulation, and much more. Due to their visual appeal they also attract the attention of students whose interest tend more towards art than science.

We have identified the following Learning Outcomes: upon completion of the suggested experiments students will be able to

- design and construct a number of different types of packing experiments using hydrogels
- experiment with different forms of data presentation, including photography, video, or mathematical analysis
- recognize the relevance of packing problems for many areas of science

In recent decades, a new area of physics has risen to prominence—*soft matter*. (See [1] for a set of lectures given by the soft matter Nobel Laureate Piere-Gilles de Gennes at various schools). It is of direct industrial relevance (soaps, detergents, ointments, and much more). It is time to give it a proper place in the curriculum and the suggested list of experiments involving soft spheres can contribute to this.

2. Historical background

The study of the dense packing of objects has a rich and fascinating history [2, 3]. Often Kepler's description of sphere stacking in crystalline layers is cited as one of the earliest accounts of a packing problem. The proof of Kepler's claim from 1611 that an arrangement in which each sphere is in contact with twelve neighbours is the densest possible sphere packing (filling around 74% of space) [4], was achieved only in 1998 by Thomas Hales [5].

Deformable spheres have also been the subject of longstanding debate. In 1727 the Reverend Stephen Hales reported observations of the arrangement of peas contained in a pot, when compressed using different weights [6, 7]. This process resulted in dense polyhedral packings, described by him as 'pretty regular Dodecahedrons' [6], i.e. cells with 12 faces. Such packings resemble the arrangements found in many biological cells [8]. More recently a particular shape of cells in the curved epithelial tissue of the fruit fly (drosophila) created great interest [9]. These so-called *scutoids* have since also been found in experiments using soap bubbles that are packed between two concentric cylinders [10].

D'Arcy Wentworth Thompson's book 'On Growth and Form', first published in 1917 and substantially extended by the author in 1942 [11], remains a classic in the area of geometric and physical principles of biology. It dedicates a full chapter to packing problems ('The forms of tissues'), with numerous examples taken from the arrangements of soap bubbles. Also discussed in detail is the 1887 conjecture of Lord Kelvin (then Sir William Thomson) to the effect that the cell type which partitions space with a minimum of surface area is that of the 14-sided tetrakaidecahedron [12, 13]. On the basis of computer simulations, Weaire and Phelan presented an alternative arrangement of bubbles with a slightly lower surface area [14] in 1994. Its elegant form inspired the design of the Water Cube for the 2008 Beijing Olympics.

Packings of hard (undeformable) objects continue to offer surprises. Experiments by Bernal in 1959 established that the densest *disordered* packing of equal spheres has a packing fraction of about 64%, significantly lower than that of the ordered Kepler packing [15]. In 2004 an experimental study of the packing of ellipsoidally shaped M&M candies showed that

their packing density is about 72%, with even 74% possible, depending on the aspect ratio of the ellipsoid [16]. A similar phenomenon exists in two dimensions, where ellipses within a certain range of aspect ratios pack closer than circles of the same area [17].

Nowadays, computer simulations are at the forefront of research into packing problems [18], such as that of spheres packed into cylinders [19], the packing of non-spherical objects, such as sphero-cylinders [20], or so-called superellipsoids which allow for a continuous mathematical representation of objects ranging from spheres to cuboids to platelets [21]. Simulation results can be compared with experimental packing data obtained using x-ray tomography, for example for spheres [22, 23], ellipsoids [24], and foams [25, 26].

The exploration of packing problems invites discoveries across the fields of mathematics, biology and physics. The employment of deformable hydrogel spheres in experiments lies at the intersections of these disciplines, making them an ideal medium for further exploration.

This may be pursued at the level of a primary or secondary school, with the virtues of ease, safety, economy, and a short time-scale. It may also serve to introduce soft matter into the curriculum of university education, applying some more rigour and analysis to the introductory material provided here. The dimension of *chemistry* could also be added.

3. What are hydrogels?

Hydrophilic gels ('hydrogels') are made up of hydrophilic ('water-loving') polymers which are cross-linked in a 3D network which can absorb large amounts of water. They were first reported by Wichterle and Lim [27] and can be formed from natural or synthetic polymers [28]. Hard in the dry state, they are soft and rubbery after water absorption, with well characterized elastic properties [29]. When drying out they return to their initial state.

Hydrogels may also exhibit volume changes in response to certain physical or chemical stimuli, or undergo a liquid–solid (gel) phase transition. Physical stimuli include temperature, electric and magnetic fields, solvent composition, light intensity, and pressure. Chemical stimuli include change of pH value, ionic concentration, and the presence of particular chemical compositions [30, 31]. Here we shall be concerned only with pressure, applied to packings of hydrogel beads in water.

Hydrogels are classified according to their internal structure, which may be amorphous, semi-crystalline, crystalline, or consist of hydrocolloid aggregates. They can be produced in different morphologies: amorphous, thin film, peelable, soft, spherical, stiff, etc [32]. They have many applications, including use in drug delivery, sanitary products, heavy metal ion removal, scaffolds in tissue engineering, contact lenses, pH-sensors, biosensors, spinal cord regeneration, supercapacitors, and oil recovery [27, 30, 31, 33–36].

One of the most common use of hydrogels in agriculture is to help plants by retaining moisture in the soil surrounding them. They are thus readily purchased from garden centres, but also from toy shops (or over the Internet), and we recommend this type of product for present purposes.

4. Materials and equipment for demonstration experiments

All the experiments described in section 5 were performed with standard commercial spherical hydrogels with a diameter of 2.10 ± 0.05 mm when in the dry state.

In addition to the hydrogels the following equipment is required for the individual experiments.

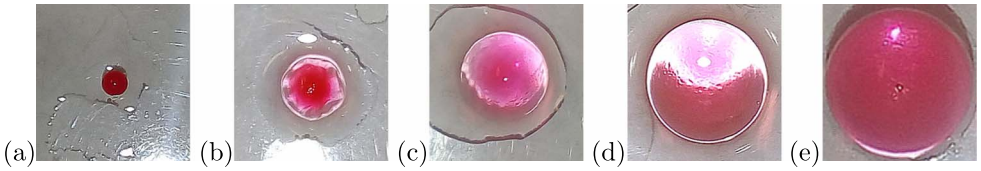

Figure 1. Controlled growth of a hydrogel sphere. The initial dry bead has diameter $d = 2.10 \pm 0.05$ mm. In the final stage (e) the hydrogel bead is fully saturated with water and has reached its maximal size (diameter $d = 11.95 \pm 0.05$ mm).

Experiment 1: Petri dish, water (beaker), pipette, ruler, camera (mobile phone), white sheet of paper to be placed underneath the Petri dish for contrast.

Experiment 2: in addition to the above: flat container with hexagonal boundary: this can be produced by gluing together six (microscopy) glass slides of equal length.

Experiment 3: acrylic or glass cylinders (inner diameter 16 mm in our experiments), stoppers, pipette/syringe needle.

Experiment 4: acrylic cylinder with stopper and piston, two watch-glasses, concentric cylinders, adhesive tape.

Experiment 5: acrylic hollow cylinder cut in half along its long axis, two stoppers.

5. Experiments

5.1. Experiment 1: Controlled swelling of hydrogels

When hydrogel beads are immersed in water their colour steadily fades upon swelling, up to the point where their outline becomes barely visible. At this stage, they are nearly neutrally buoyant.

In most of the experiments described here, we chose instead to add water in small increments to each bead, using a pipette. Figure 1 shows this process for a single hydrogel bead (diameter 2.10 ± 0.05 mm). As water is gradually absorbed the hydrogel begins to swell. Once all added water is absorbed, more drops are added, resulting in further growth of the bead. Eventually, the hydrogel develops a spherical shape [37], see figures 1(c)–(e). We can use this procedure to obtain hydrogel spheres of a specified size.

5.2. Experiment 2: Two-dimensional packings within circular and hexagonal boundaries

Having demonstrated the growth of a single hydrogel bead, we will now monitor how a large number of these arrange themselves in a two-dimensional layer, in a circular Petri dish. We will see that upon coming into contact with each other they self-organise into a mainly triangular pattern with each internal sphere contacting six others.

One hundred dry hydrogel beads were placed in a Petri dish with a flat bottom (diameter 86 mm) and the same small amount of water was added to each sphere using a pipette. Once all the hydrogels had soaked up all this water, this process was repeated.

Figure 2 shows how the swelling spheres arrange themselves in a triangular lattice as they gradually fill up the Petri dish. As the beads continue to grow as a result of further addition of water, they begin to fill the dish and some of them may be pushed upwards to lie on top of the existing layer. (We have specified the number of beads that lie in the bottom and in the top layer for each of the photos shown in figure 2). The ejected beads were removed using a

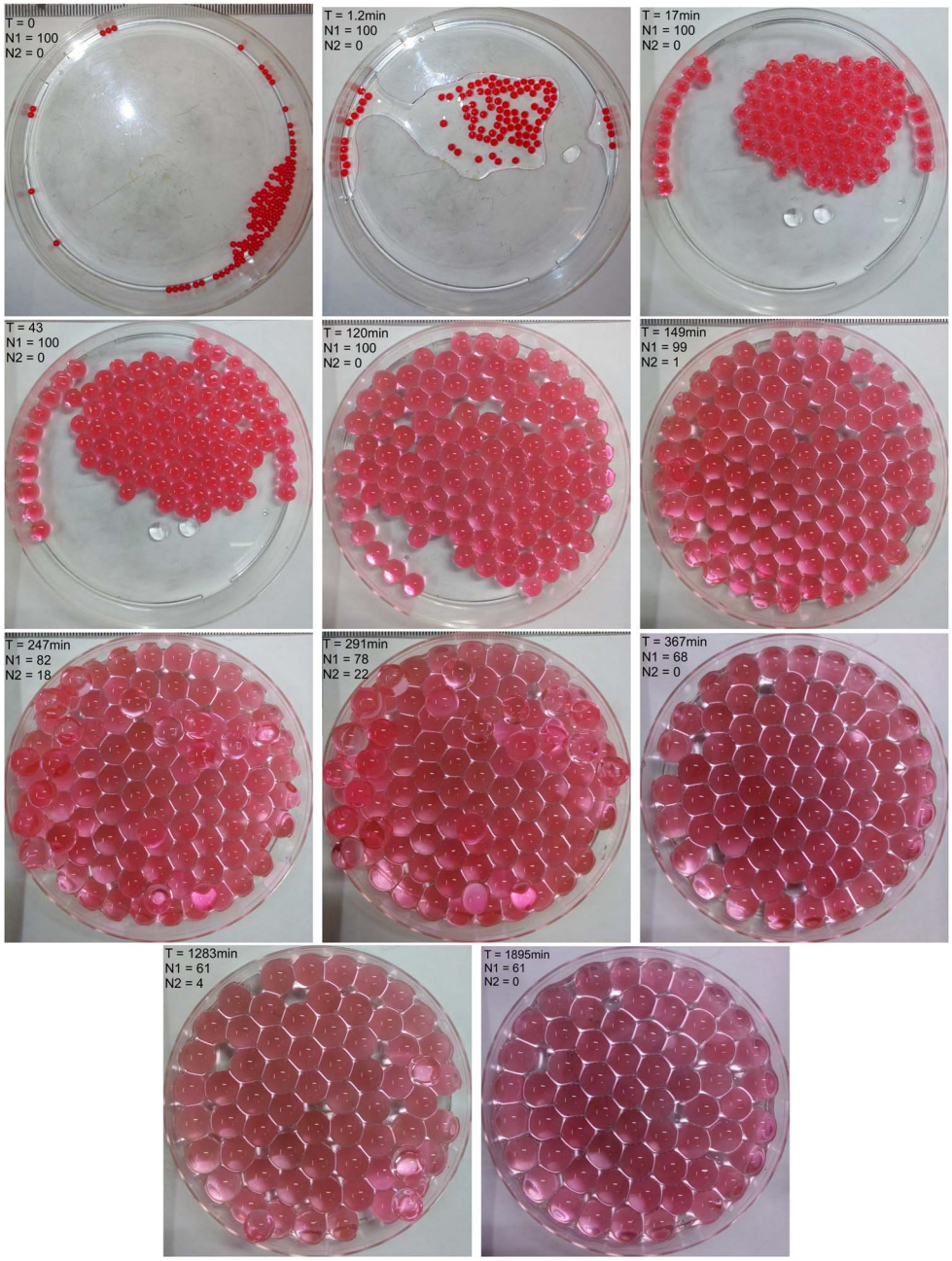

Figure 2. Photographs showing the growth of initially 100 hydrogel beads in a Petri dish. Over the course of the growth some of the beads are ejected out of the formed layer to lie at the top of it. These were removed, but are still visible in some of the snapshots shown. (In each image we also show the time T when the photo was taken, the number of hydrogels in the formed layer, N_1, and the number N_2 of ejected beads lying above it). At the end of the experiment 61 hydrogels remained in the monolayer, all displaying clear deviations from sphericity.

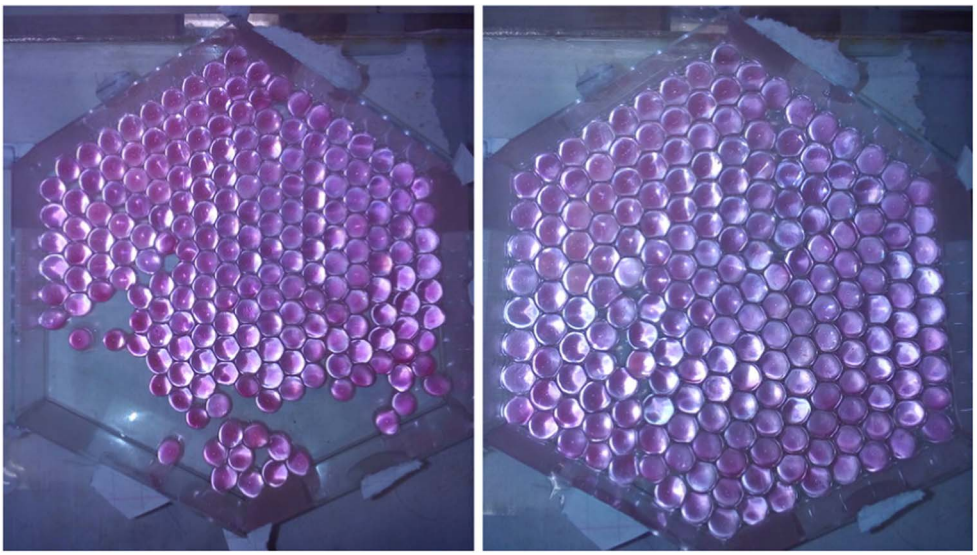

Figure 3. 200 hydrogel beads were placed in a Petri dish of hexagonal circumference. Water was added to the beads in small increments. Upon growth the beads come into contact and organize themselves in a mainly triangular arrangement, with several defects.

syringe to obtain a better view of the packing of the single layer in contact with the bottom of the Petri dish.

The formation of a triangular arrangement is well known from studies of a single layer of soap bubbles of equal size, floating on a bath of soap solution. This observation dates back to the 1947 experiments of the Nobel Laureate Lawrence Bragg and his research student John Nye. They introduced the bubble raft as a model for the crystal structure of metals [3, 38], including defects, which were being studied in the bulk crystalline packings of metal atoms at the time.

We also constructed a hexagonal confinement using glass slides as side walls (side length 75.7 mm). 200 hydrogel beads were placed inside the hexagon and were grown in the same manner as the hydrogels in the Petri dish. As seen in figure 3, the hydrogels readily form a triangular packing. In comparison to the arrangement in the circular confinement of figure 2, fewer vacancies have occurred and the spheres align well along the straight walls.

The above experiments can be used to introduce the concept of a packing fraction (or packing density), ϕ [18]. In two dimensions it is defined as the fraction of area filled by the packed objects [3]. In the case of an infinite plane filled with equal-area circles, the densest packing is a triangular arrangement of the circles. Its packing fraction is given by $\phi = \pi/(2\sqrt{3}) \simeq 0.907$ [3].

Confinement results in less dense packings, with packing fractions that depend on both the number of packed circles and the confinement geometry. Comprehensive catalogues of computer-generated packings of circles confined by a circular or a hexagonal boundary are provided by [39].

When analysing our experimental data we defined the completion of a dense sphere packing as the instant when the first hydrogel bead is ejected from the layer. The corresponding 'critical' sphere diameter is then used to obtain a value for the packing fraction.

For the hydrogels in the circular Petri dish (diameter $D = 86.02$ mm) the critical sphere diameter (obtained for $N = 99$ spheres) was about $d = 7.4$ mm (figure 2). We thus obtain for the packing fraction $\phi = N(d/D)^2 \simeq 0.73$. For the packing of the hydrogels in hexagonal confinement, as shown in figure 3 (side length $a = 75.68 \pm 0.03$ mm), the critical sphere diameter (for $N = 200$) was $d = 8.7$ mm, resulting in $\phi = N(d/a)^2\pi/(6\sqrt{3}) \simeq 0.80$. Reference [39] provides simulation data for packings of equal circles in fixed-sized containers with maximum packing density. For 99 circles within a circular boundary (as in our experiments) the simulated maximum packing density is $\phi = 0.813$. For the case of 200 circles within a hexagonal boundary the simulations result in $\phi = 0.871$.

A contributing factor for this increased packing density of the computationally achieved packings is that the packing algorithm searches for maximum density among many configurations; in our experiments only a slight 'shuffling' of sphere positions occurs over the course of their swelling. The sphere positions will thus not be optimized for achieving a maximum packing density.

5.3. Experiment 3: Columnar sphere packings in a cylinder

In the following we describe three-dimensional ordered arrangements of hydrogel spheres of equal size in a cylinder, also called *columnar* structures or packings [18]. (When using tennis balls, to demonstrate such structures, friction enables their construction also without the presence of confining cylinder walls [40]).

There is an extensive background of computational studies of dense columnar sphere packings, whose details depend on the ratio D/d of cylinder diameter D to sphere diameter d. [18, 19, 41, 42]. For $D/d = 1$ the spheres can only sit on top of each other in a straight chain while in the range $1 < D/d \leqslant 1.866$ the densest arrangement is that of a 'zig-zag' structure, with each sphere in contact with two neighbouring spheres and the cylinder wall. Forty different structures with maximum density have been identified for $D/d \leqslant 2.873$; all structures beyond $D/d = 2.7379$ contain internal spheres which are not in contact with the cylinder wall [19]. Even at $D/d \simeq 4$ the packing fraction is below 0.61, much lower than Kepler's (fcc/hcp) bulk value of 0.74.

In our experiments, dry hydrogel beads were placed in a vertically aligned perspex cylinder, which was sealed at the bottom. The cylinder was then filled with water to swell the beads. From time to time any excess water was temporarily removed using a syringe with a needle to enable taking good photographs of the developing sphere packings. The water was then added again for further sphere growth. Figure 4 shows two examples of columnar structures obtained in this way. The sphere arrangements are labelled using a notation borrowed from the field of phyllotaxis, which describes the arrangements of leaves along a stem [18, 43]. Figure 4(a) shows the zig-zag structure mentioned above. Figure 4(b) shows the structure called (2,2,0) in the phyllotactic notation (see [18], section 2.3); here each sphere (apart from the ones at bottom and top of the cylinder) contacts five other spheres.

While the flat stopper at the bottom of the cylinder is compatible with the (2,2,0) arrangement, it is incompatible with the zig-zag structure, resulting in some local disturbance of the structure. Such an effect can be avoided in computer simulations, by using so-called twisted boundary conditions [19].

Figure 5 shows a sequence of columnar structures for 17 hydrogel spheres which were grown using the procedure described above. We can identify the arrangement in 5(c) as that of the (3,2,1) structure, figure 5(d) shows the (2,2,0) structure (which was already shown in figure 4) and Figure 5(f) shows the (2,1,1) structure.

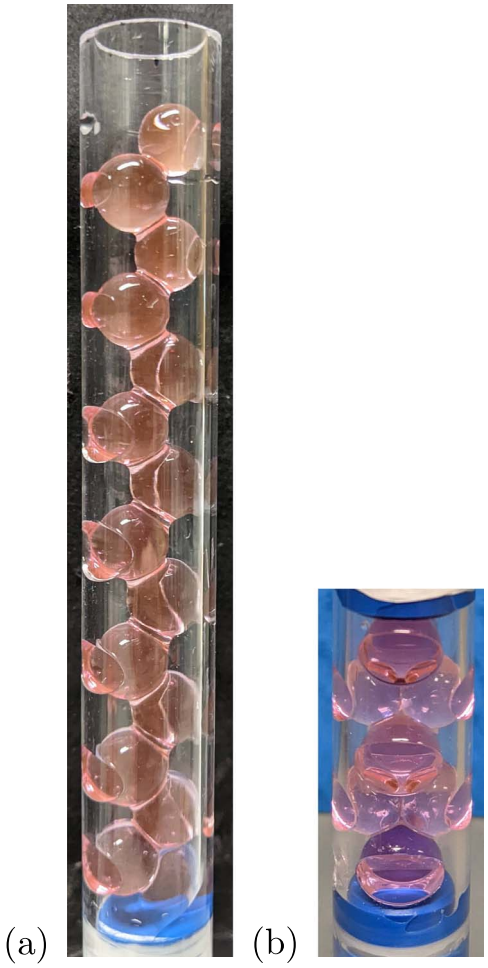

Figure 4. Columnar packings of hydrogel spheres. The type of structure is determined by the ratio of cylinder diameter D to sphere diameter d. (a) zig-zag structure, $D/d \simeq 1.9$. (b) (2,2,0) structure, $D/d \simeq 2$.

It is difficult to determine a precise value for the sphere-diameter d (and thus the ratio D/d of cylinder to sphere diameter) from these photographs; the spheres are slightly deformed and there is some optical distortion. Our rough estimates for the ratio of cylinder to sphere diameter are consistent with published ranges of these values for the different packings [19].

Students are likely to relate the columnar structures to a common sight in shopping centres: spiral arrangements of coloured balloons. Remarkably, the formation of the various structures, in response to the sphere growth due to the addition of liquid, is entirely self-organised; it is reminiscent to observations concerning plant or tissue growth (see e.g. [44] for the use of hydrogels in this context). A related phenomenon is that of the huddling together by flocks of penguins, packing densely to mitigate the cold of winter [45].

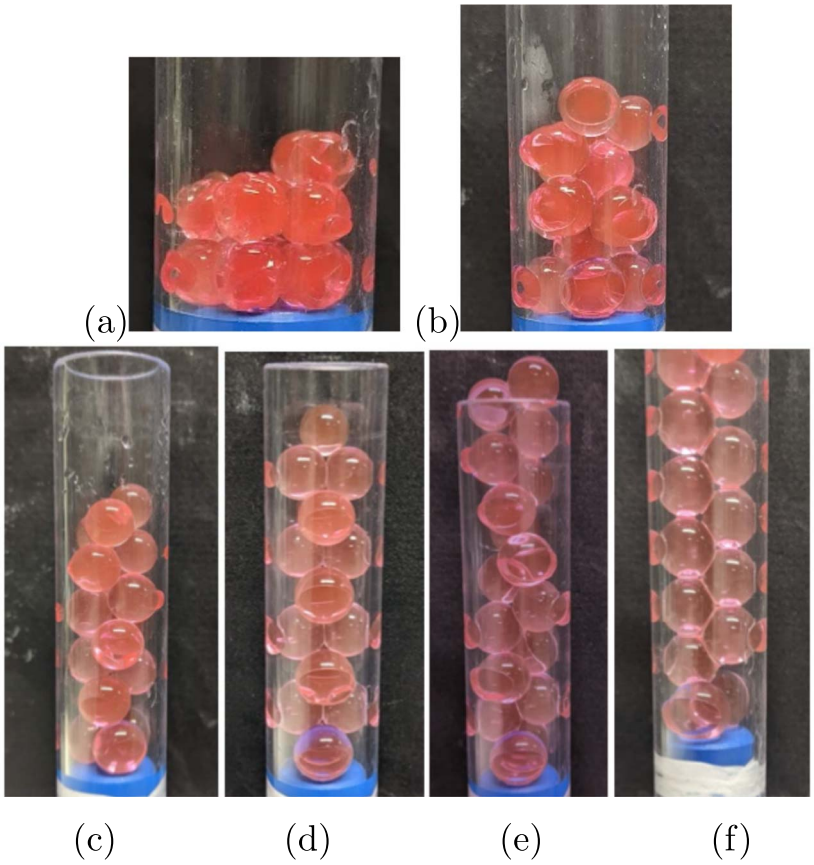

Figure 5. Sequence of photographs showing how swelling hydrogel beads arranged in different columnar packings, depending on the ratio D/d of cylinder to sphere diameter. The cylinder is vertically placed. Clearly identifiable packings are seen in (c) structure (3,2,1) for $D/d \simeq 2.16$, (d) (2,2,0), (e) intermediary structure containing sections of (3,2,1), and (f) (2,1,1) structure with two hydrogel spheres at the base, $D/d \simeq 2.0$.

5.4. Experiment 4: Packings under external compression

We have also applied a compressional force to the columnar crystals described in the previous section. This was achieved by inserting a piston into the open upper end of the cylinder. Pressing down on the piston results in deformation of the hydrogel spheres, eventually leading to the establishment of further contacts or rearrangements. The resulting structural transitions of the sphere arrangements can be compared to those studied in computer simulations of packings of *soft* spheres, in which spheres are allowed to overlap under compression [46–48].

Figure 6 shows four different columnar structure formed by 16 hydrogel spheres. The initial uncompressed arrangement is that of the zig-zag structure of figure 4(a). Downward movement of the piston results in the (2,1,1) structure (see also figure 5(f)) with its additional sphere contacts. Further compression leads to the so-called twisted zig-zag structure, shown in figure 6(c) Finally, at even higher compression, we can see that the upper part of the cylinder

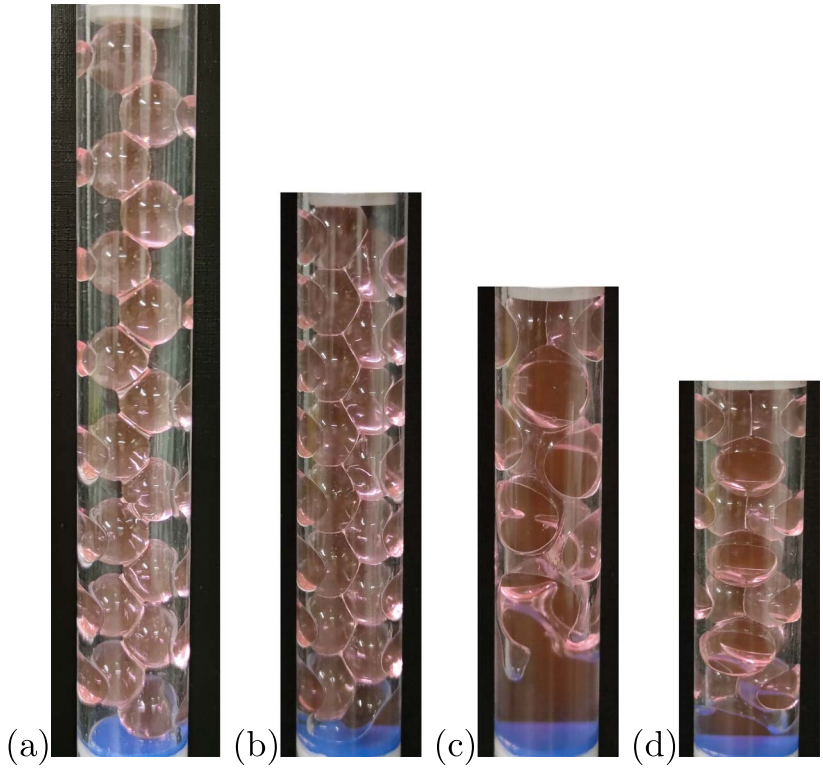

Figure 6. Sequence of structural changes, induced by pushing down a piston into a cylinder filled with 16 hydrogel spheres. (a) Zig-zag structure (column length $L_0 = 135$ mm). (b) (2,1,1) (column length $L = 87$ mm). (c) twisted zig-zag structure ($L = 79$ mm). (d) (2,2,0) (towards the upper section of the cylinder only) ($L = 66$ mm).

displays yet another structure, called (2,2,0) (see Figure 5(d)). The same sequence of structural transitions has been seen in computer simulations [47].

In a variation of the compression experiment, we produced even denser packings. This was achieved by manually pushing swollen hydrogels into the space between two concentric cylinders, or between two watch-glasses which were sealed around the rim so that the beads could not escape when compressed. In the resulting packings, shown in figure 7, the hydrogels can be approximated as polyhedra, reminiscent of the shapes of bubbles in a foam with low liquid volume fraction, cells in biological tissue, or Reverend Hales' compressed peas.

5.5. Experiment 5: Buckling of a line of spheres in a confining potential

Our final experiment is related to recent experimental and theoretical studies concerning the buckling of a line of contacting spheres, under compression [49–52]. We have placed 10 hydrogel beads in a horizontally placed trough made from a cylinder that was cut in half longitudinally. Stoppers were fitted at both ends, as shown in figure 8. Water was then added in equal amounts to all the beads in turn using a pipette; once all the water had been absorbed, a photograph was taken and more water was added, again in equal amounts to all the spheres.

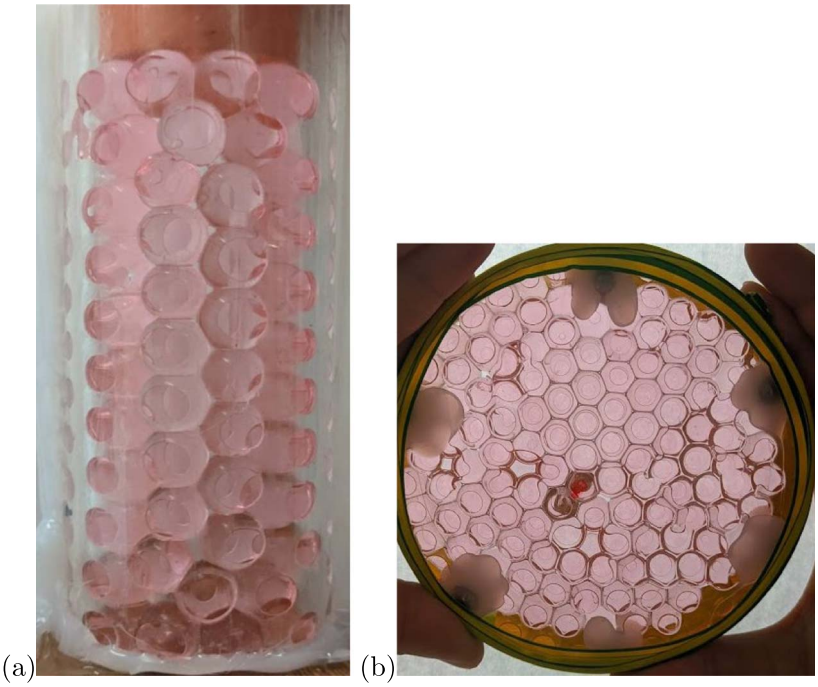

Figure 7. Pushing hydrogel spheres into the spacing between two concentric cylinders (a), or between two watch-glasses (b) results in an increase in packing density. The hydrogel spheres become deformed and take on more polyhedral-like shapes, as found also in foams.

As the spheres swell, they eventually make contacts with their neigbours and the stoppers, resulting in the formation of a line of touching spheres, located at the bottom of the trough to minimize gravitational energy. Upon further growth of the spheres, instigated by adding more water to each of them, the linear chain is no longer stable and gives way to a modulated zig-zag structure in which spheres are displaced sideways from the bottom of the trough; the displacement from a straight line is maximal for the spheres in the centre of the chain. A further increase of sphere size makes the buckling profile more prominent. Its shape, shown in figure 8(d), can be interpreted using a theoretical model recently developed by us [50, 52]. It concerns the arrangements of contacting hard spheres in a transverse harmonic potential, provided by the transverse profile of the trough in the experiment [49, 52].

A charming example of this phenomenon is to be found occasionally in pods of peas. Generally, the row of peas is essentially straight, as was already commented by Kepler [4]. However, this is not always the case, as is evident when looking at a number of carefully opened pods, or simply browsing the Internet, where even x-ray images of unopened pods can be found. The peas can display a zigzag arrangement as shown in figure 9, very much reminiscent of the modulated zig-zag structure displayed by the swollen hydrogels shown in figure 8. The biological example is more complicated, as both peas and the confining pod grow, but not necessarily at the same rate. A related point was made by Kepler when discussing the arrangements of pomegranate seeds [4].

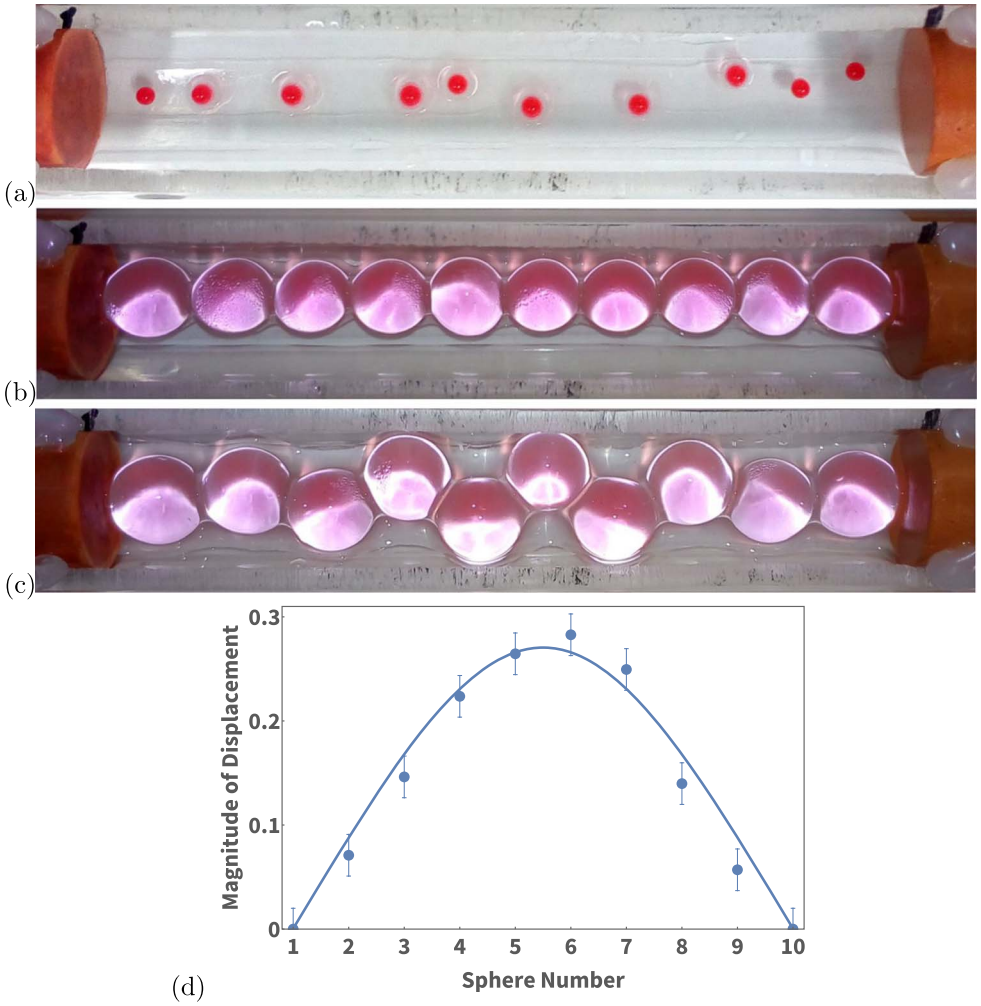

Figure 8. Buckling of a line of contacting hydrogel spheres. (a) Ten hydrogel beads lying at the bottom of a horizontal trough with hard walls at both ends. (b) Upon the addition of liquid they grow, come into contact, and form a straight linear chain. (c) Eventually, upon further swelling of the spheres, the chain buckles to form a smoothly modulated zig-zag structure. The corresponding profile of the transverse sphere displacement is shown in (d), together with a theoretical result (for the same compression), which involves a Jacobi function [57].

6. Summary and suggestions for further experiments

We have demonstrated that hydrogels are well suited to a variety of classroom experiments related to the packing of spheres of equal volume. In contrast to hard spheres, such as ball bearings, hydrogels can absorb water and expand. This invites for intriguing and entertaining observations of structure formation due to growth, not dissimilar to what is observed in biological tissue. The spheres deform into more polyhedral shapes, in response to external compression. Hydrogels therefore provide an ideal medium for exploring close-packed

Figure 9. Photograph of a zig-zag arrangement of peas in a pod. (Photo by Rachel Gorjestani, Pixabay gc10209902_1920. (license is here: https://pixabay.com/service/terms/)).

structures in experiment, to complement the highly sophisticated computational studies of packings performed nowadays on high-performance computers.

The experiments described here should provide stimulation for further explorations. It would for example be of interest to look more closely at the growth of beads in the columnar structures of figure 5 and observe the formation of additional structures.

A closer look at figure 4(a) reveals that the hydrogel spheres nearer to the bottom of the vertically placed cylinder are placed closer together than the ones further up the cylinder. This could be an effect of the combined weight of the spheres. As the hydrogels are soft this might result in a noticeable deformation. Using a longer cylinder should thus result in a *range* of different structures along the cylinder. To mitigate against the gravity-induced vertical compression of the packings one could then add water to possibly trigger a re-organisation of the spheres into a uniform structure throughout the cylinder.

Further variations of the presented experiments could involve the use of cylinders with square or triangular cross-sections (see [53] for foams), or the growth of hydrogels between curved walls, with relevance to curved epithelia tissue material [9, 10].

It would also be of interest to capture some of the experiments on video to allow for a better illustration of the often slow dynamics of the packing process in a speed-up movie. Many of them also provide opportunities for photography, requiring exploration of optimal lighting arrangements. Photographs of related experiments with soap bubbles have already won widespread recognition for art-photographer Kym Cox [54, 55], but the field remains open for further variations.

In summary, we have seen that playing with hydrogels can provide motivation and amusement. One may well add to the growing literature of packing [3] by examining their special properties, and explore the cross-over between science and art [56].

Acknowledgments

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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