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# The mechanical properties of model-compacted tablets

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**Abstract** In this study, the compressive strength of tablets made with salt, starch and fat was investigated. The strength was found to increase with compaction pressure, up to a maximum value where further increase in the compaction pressure led to no increase in the strength. The maximum strength corresponded to the point where zero porosity was obtained during the compaction process. However, because of the elastic rebound of the tablets after ejection, the maximum strength corresponded to non-zero final tablet porosities which varied between the materials. For this reason, the use of the density occurring during the compaction process appeared to provide a more reliable comparison between the materials. A simple linear mixing rule did not hold in characterising the strength in the salt:starch:fat systems. However, two regimes were observed depending on the salt volume fraction. At low salt volume fractions, the effect of the salt was negligible. After a certain critical salt volume fraction, increasing the salt led to an increase in the strength. Finite element simulations based on X-ray microtomography images of the tablets suggested that in the first regime, the stresses due to the salt particles were localised but in the second regime, stress-bearing networks were formed between the salt particles.

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## Introduction

A compaction process is utilised in the production of some food products such as seasoning cubes. Similarly, compaction processes are also used in other industries, in particular to manufacture pharmaceutical, ceramics and metals products. The compacts are often made with certain final characteristics in mind, for example, to obtain certain mechanical properties such as the stiffness, strength or friability [1–3].

In general, the compaction process involves decreasing the bulk volume of a powder by the application of a high pressure. The volume reduction is due to a combination of particle rearrangement, deformation and/or fracture. The rigidity of the compact is a result of the formation of new permanent bonds between the particles. The bonds are formed when the movement of the particles is constrained or jammed, such as when the maximum packing of the particles is achieved [4–6].

At very low or zero porosity, a maximum in the mechanical properties of the compact is achieved [7, 8]. These properties have been suggested to represent the mechanical properties of its constituent materials [9]. However, studies have shown that the maximum mechanical properties of a tablet are also affected by the geometrical properties of the particles such as the shape and the size [10]. This suggests that the bonds formed between particles of the same constituent are not necessarily the same as the internal bonds within the constituent. The mechanical properties of a compact also depend on processing conditions such as the speed of compaction [11] and the friction caused by the surface of the mould used for performing the compaction [12].

The understanding of the mechanical properties of compacts has been pursued via a number of approaches,

from percolation theory [9, 13], the packing behaviour of particles [14], computer simulations utilising either continuum or discrete element methods [14–18] to empirical or phenomenological approaches [19]. The complexity of the compaction process is increased in multi-component tablets relative to single component tablets. It remains a difficult problem to predict the strength of multicomponent tablets based on the properties of the constituent components. Some recent work in this area includes that by Van Veen and co-workers, who proposed a method for evaluating the strength of tablets containing two components based on the volume fractions of the components [4]. The method, which is based on a model utilising a mixture of isostress and isostrain conditions, requires an additional measurement to characterise the interactions between the two components. Busignies et al. [20], using a statistical method to account for the interactions between components based on weight fractions, also suggested that interactions between components have to be accounted for. Wu et al. [18] showed that extending the Ryshkewitch–Duckworth equation to multi-component tablets allowed a good prediction of the strengths of the latter. Wu et al. assumed zero interaction between different components, and that the total strength was the sum of the strength of individual components weighted with respect to their solid fractions.

The objective of this study was to investigate the mechanical properties of model-compacted food systems consisting of salt, starch and fat. Although studies on compacted salt and starch systems have been investigated in the literature, three-component systems as considered here have not been studied in depth.

## Materials and methods

### Materials

The materials were salt (sodium chloride), corn starch and hydrogenated palm oil fat. The mixes were prepared using a kitchen mixer where the fat was added in a molten state at 65 °C to the salt, the starch or mixtures of the two. A number of formulations comprising salt:fat, starch:fat and salt:starch:fat systems were made using different weight ratios of the components. The formulations will be referred to in terms of weight ratios, e.g. a mixture with 90% w/w salt and 10% w/w fat will be referred to as 90:10.

### Compressive strength tests

An Instron 5500R with a load cell of 10 kN was used to make and break the tablets. A steel mould comprising a detachable bottom disk, a bottom assist (similar to the bottom relaxation assist in [21]) and an upper piston that was in contact with the crosshead of the Instron was used to make the compacts. The filling cavity of the mould was cylindrical with a diameter of 12.5 mm and a length of 18.5 mm. The tablets were ejected from the cavity by pushing the piston through the cavity manually. For the salt:fat mixtures, 2 g of powder mixture was used. For the other formulations with starch, 1 g of powder mixture was used due to the lower density of the starch compared to the salt.

To make the tablets, a compaction speed of 1 mm/s was used. Only the upper piston was displaced during the compaction, and the compaction pressure was immediately removed after the maximum compaction was achieved. Compaction forces were varied from 0.2 to 9.5 kN (corresponding to compaction pressures of 1.6–77 MPa).

Once made, the tablets were subjected to uniaxial unconfined compression tests between unlubricated polished steel platens. A crosshead speed of 0.5 mm/s was used, and the tablets were tested within an hour after making. The compression tests were performed in the same direction as the compaction, i.e. parallel to the cylindrical axis.

The final dimensions of the tablets were measured with a vernier calliper and their mass was determined with an analytical balance. The true densities used in calculations were 2.15 and 1.46 g/cm$^3$ for the salt and starch respectively. These were measured using a Helium-pycnometer. The true density of the fat was assumed to be 0.94 g/cm$^3$.

### X-ray microtomography (μCT) and finite element simulations

The same Instron 5500R machine was used to make the tablets. In this case, to facilitate imaging of the microstructure using the μCT technique, tablets of 5 mm diameter with mass 0.2 g were made. A smaller steel mould was used to make these tablets. The formulations investigated were salt:starch:fat systems of 45:45:10 and 60:30:10 which were also studied in subsection "Compressive strength tests" under "Materials and methods".

μCT [22] was used for the non-invasive visualisation of the internal structure of the tablets. The contrast in μCT images is based on the difference in absorption of X-rays by the constituents of the sample. In the salt:starch:fat systems, the contrast was caused by a difference in density and difference in element composition, e.g. between sodium chloride and fat. Images were obtained using a SkyScan 1072 desktop μCT system. X-rays were generated by a microfocus X-ray tube (10 μm focal spot size) with tungsten anode. Power settings of 100 kV and 100 μA were used. The image size was 1024 × 1024 pixels. The tablets were imaged using a plastic cylindrical tablet holder with an inner diameter of 6.0 mm. For tomographic reconstruction, beam hardening correction of 50% and low

ring artefact reduction (5%) were selected. A magnification factor of 45 was selected resulting in a pixel size of 6.1 μm.

For image processing, analysis and visualisation, the Amira software (Mercury Computer Systems) was used. The noise was reduced by using a 3D median filter. Segmentation was done by using thresholding generating a stack of binary images with pixel values corresponding to the different phases in the tablets.

The finite element simulations were performed using the ABAQUS software. A two-dimensional image of the microstructure was first selected. From the image, a rectangular selection was extracted and the selection was divided into a uniform mesh consisting of two-dimensional elements, each element having four integration points. A user-defined subroutine was used to assign the appropriate material properties to the integration points of the mesh using the corresponding binary value of the microstructure image. The different phases were assumed to bond completely to each other.

The simulations were performed assuming uniaxial compression and 2D plane stress conditions. A consequence of the 2D assumption was that the particles were effectively modelled as rods along the thickness direction. The elastic modulus, yield stress and Poisson's ratio of the salt were assumed to be 40 GPa, 2.4 MPa and 0.252 respectively.[1] The starch and fat were assumed incorporated into a single phase (see later). Mixes of the starch and the fat were made and compacted into tablets. Uniaxial compression tests were then performed on these mixes to obtain their stress–strain properties. In the simulations, the starch:fat matrix was assumed to behave in a simple elastic-plastic manner, with the elastic modulus given by the slope of the stress–strain relationship and the yield strain arbitrarily chosen to correspond to the fracture strain.

## Results

### Compressive strength tests

Figure 1 shows the stress–strain profiles of tablets measured from the uniaxial compression tests. The stress–strain data correspond to tablets made to different compaction pressures. Formulations corresponding to salt:fat system at 89:11, starch:fat at 90:10 and salt:starch:fat at 80:10:10 and 10:80:10 are shown. The tablets with high salt content exhibited a gradual increase in the stress at low strains, which made the determination of zero strain difficult. Following this initial 'slack', there was a relatively linear increase in the stress–strain profile up to the breaking point. The slopes of the strain hardening region appear to be independent of the compaction pressure, and only the compressive (or fracture) strengths were dependent on the compaction pressure. The tablets with high starch content, however, had different stiffnesses and compressive strengths depending on the compaction pressure. Since the contact surface area between particles increases with increasing densification [23], the stress–strain profiles suggest that for the tablets with high salt content, the extent of strain hardening was independent of the contact surface area, but the compressive strength increased with the surface area. For tablets with high starch content, both the strain hardening and the compressive strength increased with the surface area.

Figure 2 shows the relationship between the compressive strength and compaction pressure in more detail. For the salt:fat and starch:fat systems, only three formulations are shown for simplicity. Initially, the strength increased with compaction pressure. After a certain compaction pressure, the value of which depended on the formulation, a 'plateau' regime was achieved. In this regime, the compressive strength attained a maximum value and remained independent of the compaction pressure. This behaviour has also been observed in other compacted systems in the literature [7, 8].

In Fig. 3, the relationships between the compressive strength and the relative densities are shown. The relative densities were obtained by dividing the bulk density to the true density of the tablets. Two bulk densities were calculated:

1. the first is the relative compacted density which corresponds to the density under maximum pressure during the compaction process;
2. the second is the relative final density which corresponds to the density immediately after the tablets were ejected.

The relative compacted density should not exceed the value of one since this would refer to the condition of zero porosity in the system. However, in the measurements, the relative compacted density exceeded the value of one because of the inherent deformability of the Instron machine and the mould which were not taken into account in the calculations.

Comparing Fig. 2 with Fig. 3, it can be seen that the maximum strength of a tablet is associated to the condition of zero porosity during the compaction process. Furthermore, the relative final density was always smaller than the relative compacted density. This could be due to the elastic recovery of the materials, which may also increase the porosity of the tablet after ejection [24]. The elastic recovery of the starch was much greater than that of the salt, and this was shown both in the two-component as well as the three-component systems.

[1] http://www.hilger-crystals.co.uk/prior/mat_nacl.htm.

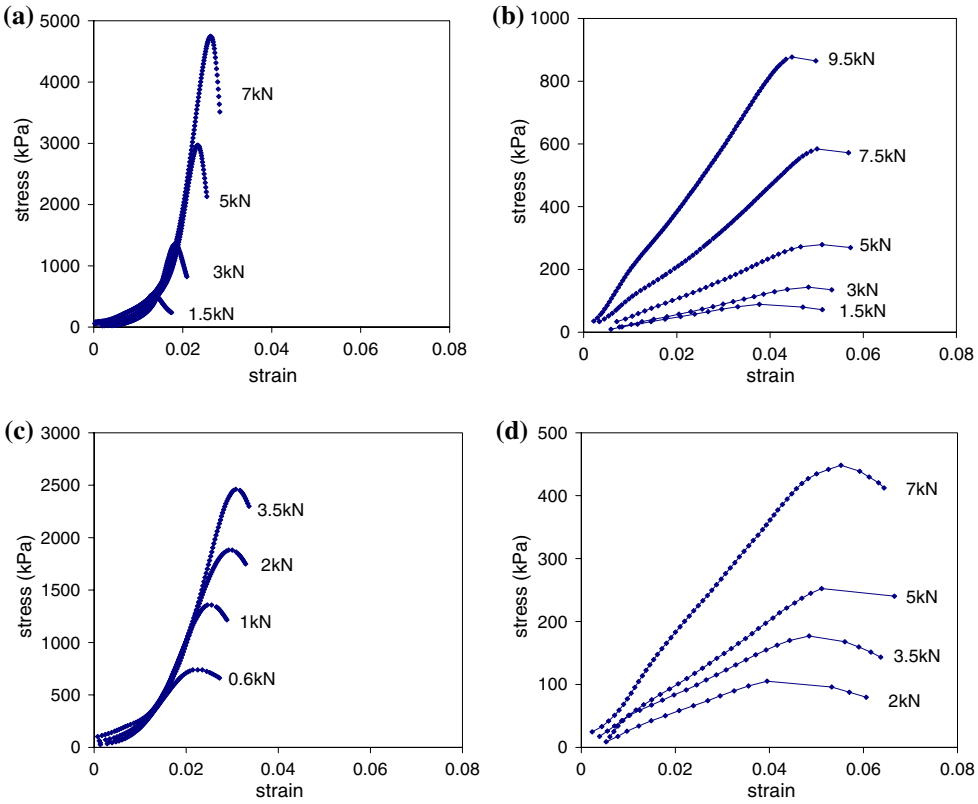


**Fig. 1** Compression stress–strain profiles **a** of 89:11 salt:fat, **b** of 90:10 starch:fat, **c** of 80:10:10 salt:starch:fat, and **d** of 10:80:10 salt:starch:fat tablets compacted to different compaction forces

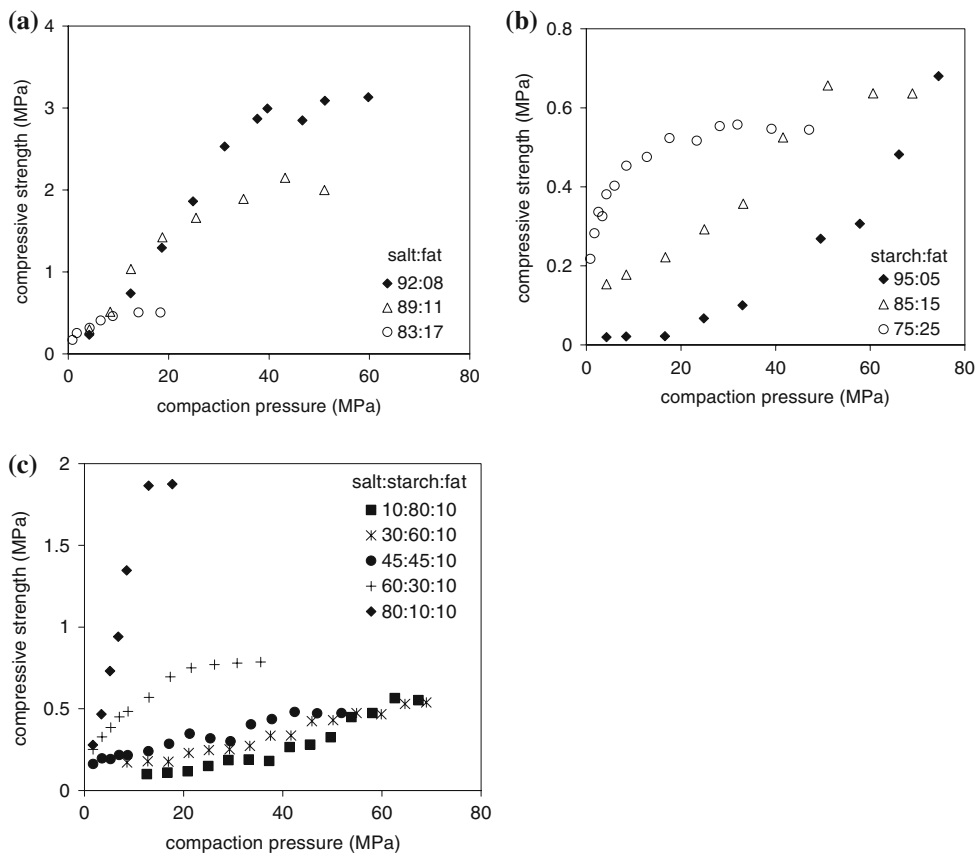


**Fig. 2** Variation of compressive strength with compaction pressure. **a** Salt:fat. **b** Starch:fat. **c** Salt:starch:fat systems

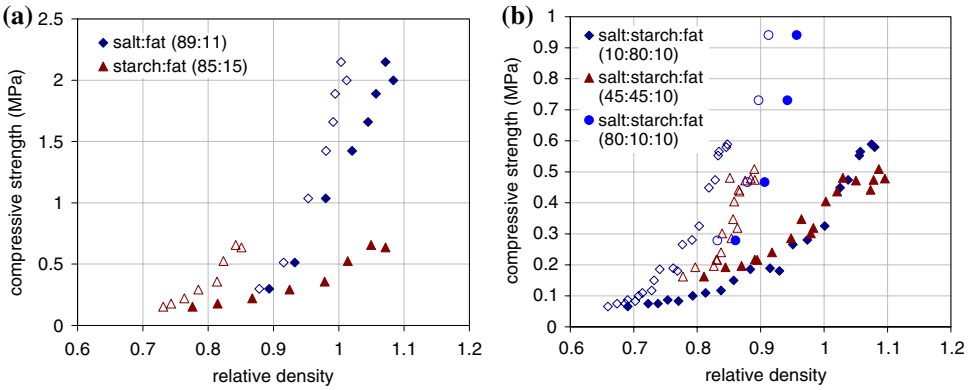


**Fig. 3** Variation of compressive strength with relative compacted (*filled markers*) and final (*unfilled markers*) densities. **a** Two-component system. **b** Three-component systems

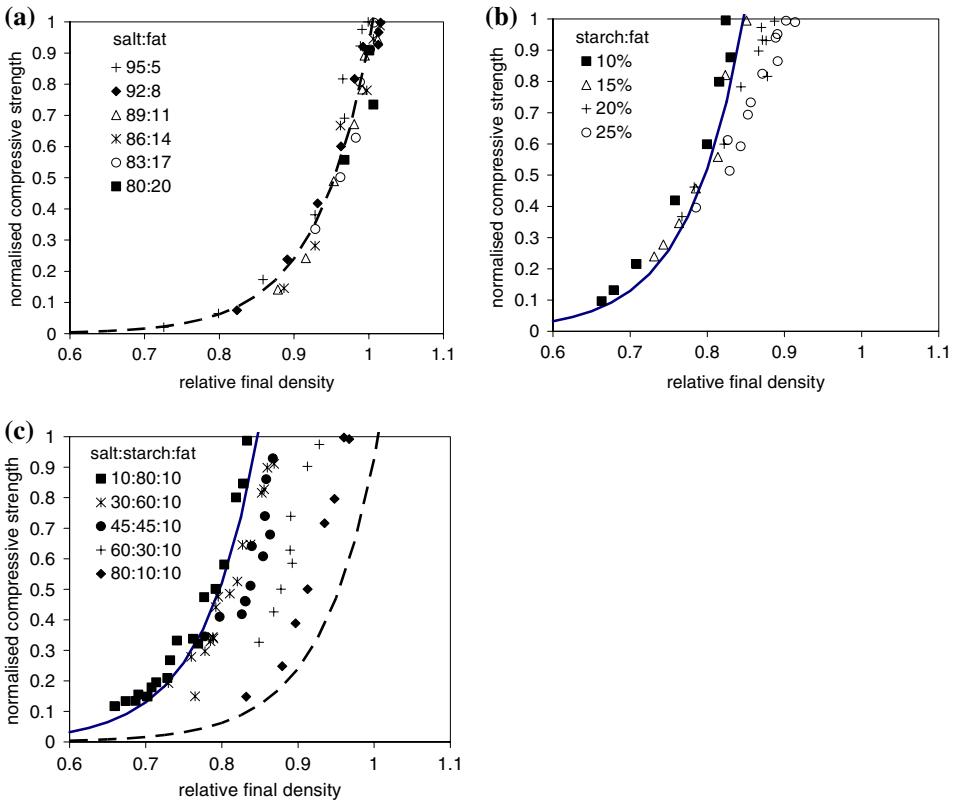


**Fig. 4** Relationships between compressive strength normalised against the maximum strength for each formulation and the relative final density. **a** Salt:fat systems. **b** Starch:fat systems. **c** Salt:starch:systems. *Full* and *dotted lines* in (**c**) are taken from (**b**) and (**a**) respectively

Normalising the strengths of the tablets with the corresponding maximum strength for each formulation, Fig. 4a–c shows how these normalised strengths relate to the relative final densities of the tablets. For the salt:fat systems, the data from the different weight fractions of fat were reasonably superimposed on each other. For the starch:fat systems, the data for the different fat weight fractions did not superimpose on each other, and the maximum strengths were achieved at smaller relative final densities with decreasing fat weight fraction. For the salt:starch:fat systems, the data showed a large shift from a predominantly salt:fat behaviour for compositions with high salt content to that of a predominantly starch:fat behaviour for compositions with high starch content.

## μCT and simulations

In Fig. 5, the relationship between the maximum compressive strength and the volume fraction of salt in the salt:starch:fat systems is shown. The data did not fall on a linear line according to a simple linear rule of mixtures. Instead, the data appeared to be divided into two regimes:

1. For salt weight fractions of 45% and below, the compressive strength was relatively low and changed little with the salt content;
2. However, at salt weight fractions of 45% and above, an increase in the salt content led to a hardening regime,

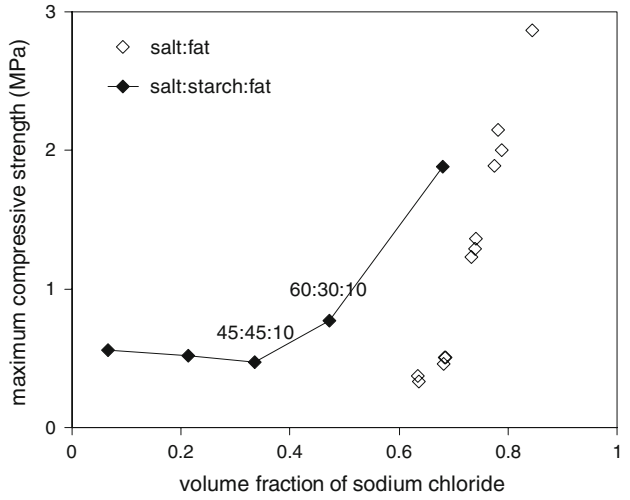


**Fig. 5** Maximum compressive strength of salt:fat and salt:starch:fat systems as a relationship with the volume fraction of salt

i.e. an increase in the compressive strength. The salt volume fraction at which the transition occurred was ~0.4.

Also plotted in Fig. 5 is the relationship between the compressive strength and the salt volume fraction for the salt:fat tablets. For these tablets, there was a large increase in the compressive strength at a salt volume fraction of between 0.6 and 0.8. The volume fraction at which particles of a component begin to establish a compact is known as the critical volume fraction [4]. The critical volume fraction depends on the packing efficiency as well as interactions between particles [6]. Comparing between the salt:fat and salt:starch:fat systems, it can be seen that the starch reduced the critical salt volume fraction to achieve strengthening of the tablets.

Figure 6 shows the μCT images of salt:starch:fat tablets of 45:45:10 and 60:30:10 respectively. The dark regions correspond to the salt particles, whereas the starch and the fat components were not differentiated between each other. It can be seen that in the 45:45:10 tablet, the salt particles were much sparsely distributed than the 60:30:10 sample.

The stress distribution in the tablets as predicted by the finite element simulations are shown in Fig. 7 for the 60:30:10 tablet and Fig. 8 for the 45:45:10 tablet. It can be observed that in the 60:30:10 tablet, the stress was largely transferred via networks through the salt particles. In the 45:45:10 tablet, the stress concentrations caused by the salt particles were isolated and as the strain was increased, the stress was distributed rather evenly in the starch:fat matrix. These simulations suggest that the salt particles played an important role as stress-bearers in the 60:30:10 tablets compared to the 45:45:10 tablets and that interaction between the salt particles led to the strengthening of the tablets.

## Discussion

The comparison of tablet properties is normally performed based on the relative final density or porosity. For example, a relative density of 0.9 is commonly used to compare the strength of tablets in pharmaceutics [25]. Furthermore, the normalised strength data are also commonly fitted to the Ryshkewitch–Duckworth equation [26],

$$\frac{\sigma}{\sigma_0} = \exp(-\lambda\varepsilon) \quad (1)$$

where $\sigma$ is the strength of the tablet at porosity $\varepsilon$ ($\varepsilon = 1 -$ relative density), $\sigma_0$ is the strength at zero porosity and $\lambda$ is a constant.

However, as can be seen in the current data, the maximum strength was attained despite the tablets having a finite final porosity. Further increasing the compaction pressure of these tablets resulted in no further increase in the strength or the final density. It appears therefore that comparing the strength of tablets based on the final density or porosity can be misleading because of the differences in the degree of elastic recovery of tablets of different formulations. If the data were extrapolated to zero porosity, i.e. relative final density of 1, then the value of $\sigma_0$ predicted would be much higher than the maximum strengths measured in the experiments.

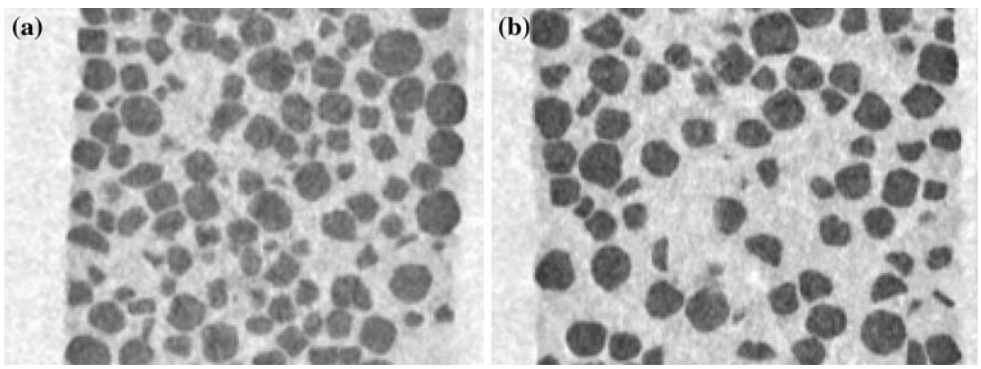


**Fig. 6** μCT images of vertical cross-sections of tablets for salt:starch:fat systems with weight fractions. **a** 60:30:10. **b** 45:45:10. The width of the images is 6.2 mm (972 pixels) for (**a**) and 5.8 mm (705 pixels) for (**b**)

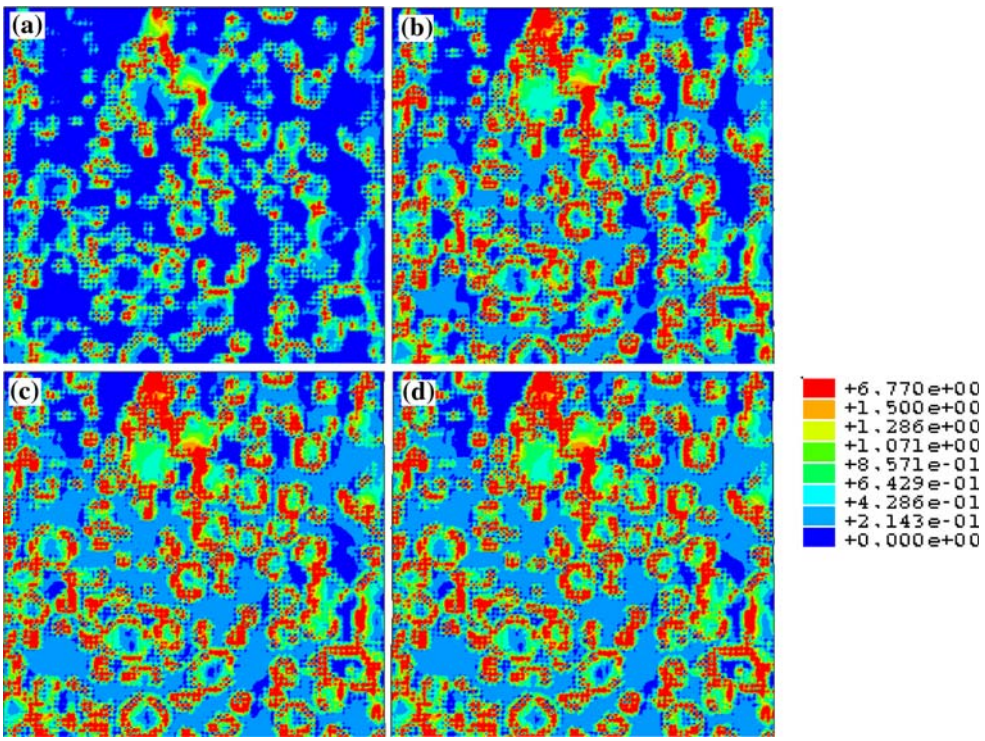


**Fig. 7** Distribution of effective Mises stresses for 60:30:10 sample at different levels of compression strain. **a** 0.003. **b** 0.009. **c** 0.015. **d** 0.02

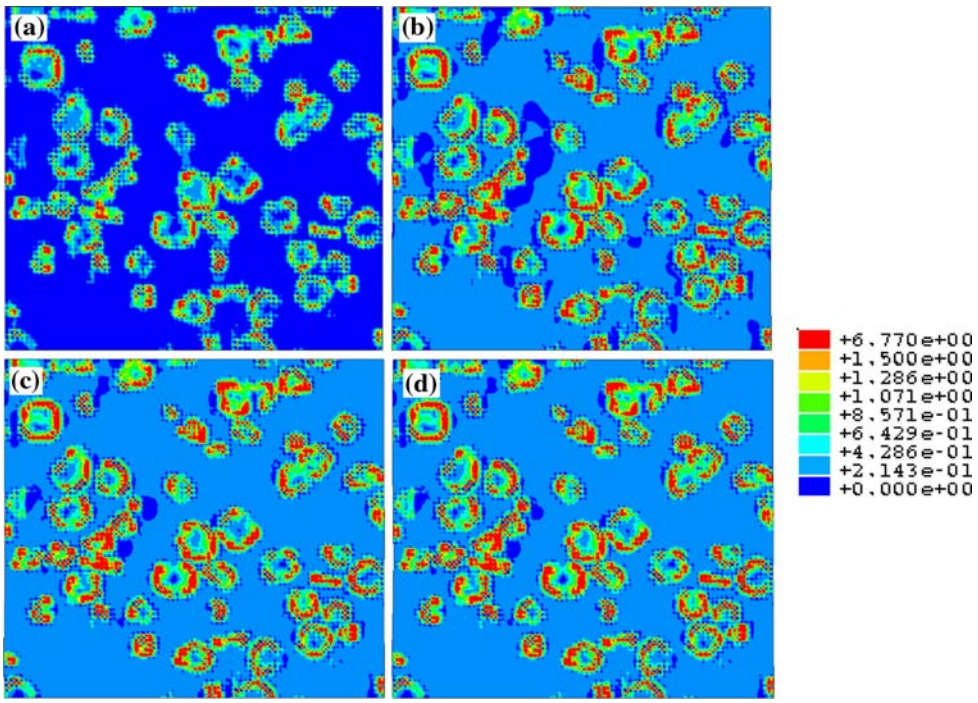


**Fig. 8** Distribution of effective Mises stresses for 45:45:10 sample at different levels of compression strain. **a** 0.005. **b** 0.012. **c** 0.019. **d** 0.022

A more reliable comparison may be obtained by using the compacted density which is not affected by the elastic rebound of the tablet. These data are shown in Fig. 9 for the starch:fat and the salt:fat formulations. The difference between the relationships based on final or compacted density may be seen in the value of $\lambda$. This constant has been used to represent the 'bonding capacity', with high values of $\lambda$ indicating that tablet strength increases rapidly with decreasing porosity which points to stronger bonding of the particles [24]. If the normalised strength data based on the relative final density were used to characterise the salt:fat and starch:fat systems (shown as dotted and full lines in Fig. 4a and b respectively), values of $\lambda$ of $\sim$13.5 and 13.9 were obtained for the salt:fat and starch:fat

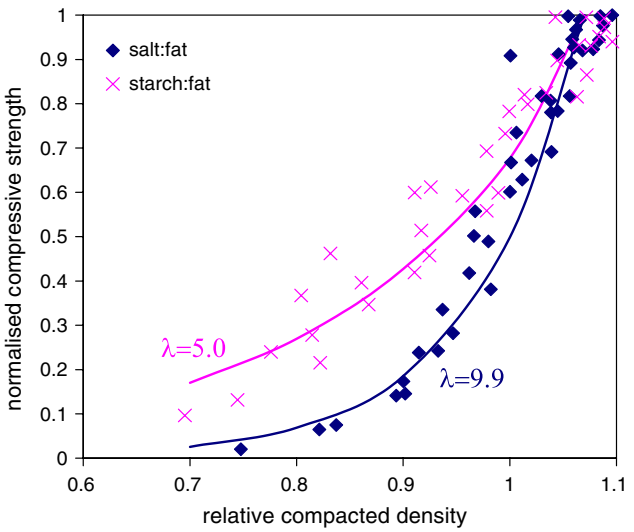


**Fig. 9** Relationships between compressive strength normalised against the maximum strength for each formulation and the relative compacted density for salt:fat and starch:fat systems

systems respectively. However, if the relative compacted density were used, values of $\lambda$ of $\sim$9.9 and 5.0 are obtained, showing a clear difference between the salt and the starch 'bonding capacity'.

It has been said that the mechanical properties of binary mixtures cannot be described using a simple linear mixing rule [20] and this was observed here. On the other hand, Wu et al. [18] had found that strength of compacted powder mixtures could be successfully determined from the strength of constituent components weighted by their volume fractions, although in their case tensile strength was measured which could have a different behaviour compared to the compressive strength. Both Busignies et al. [20] and Van Veen et al. [4] suggested that in addition to data for individual components, additional tests are also required to characterise the adhesion between different components. In the current systems, it appears that the critical volume fraction of a single component can also be changed in the presence of another component, and these effects need to be looked into further if the strength of a multi-component tablet is to be predicted from the strengths of its individual components.

## Conclusions

The compressive strength of tablets containing salt, starch and fat has been investigated. It was found that the maximum strengths of the tablets corresponded to the point where zero porosity was obtained during the compaction process. Due to the elastic rebound after ejection, different materials gave different final tablet porosities, which meant that the maximum strength achievable for any tablet did not correspond to the final tablet porosity of zero, but to some finite porosity. Thus, comparing between tablets of the same final porosity may give misleading results and it may be more reliable to use the minimum porosity occurring during the compaction process instead. The salt particles contributed to the strengthening of the tablets but the simple linear mixing rule was not applicable in characterising the strength of tablets with different weight fractions of the components. The presence of the starch was found to decrease the critical volume fraction necessary for the strengthening by the salt particles. The data suggest that interactions between different components in a tablet are not restricted to simple adhesion between the components but also affect the transfer of stress through packing effects.

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