Metallurgical and Materials Data 2, no. 3 (2024): 99-102

Publisher: Association of Metallurgical Engineers of Serbia

Metallurgical and Materials Data

www.metall-mater-data.com

Modified testing protocol for texture analysis of alginate cryogel material

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ARTICLE INFORMATION:

https://doi.org/10.56801/MMD36

Received: 06 October 2024
Accepted: 21 October 2024

Type of paper: Research paper

ABSTRACT

Alginate cryogels are biopolymer materials with wide applications in the food industry, where their textural properties play an important role as a key quality parameter. Textural properties are determined based on data obtained from compression tests. Therefore, the adequate design of the compression testing protocol ensures reliable texture results. The influence of crosshead speed on compression test results was investigated by varying the crosshead speed from 0.5 to 5 mm/min. The results indicate that crosshead speed has a negligible effect on compression results. Notably, after 95% deformation at maximum load, the compressed specimen exhibits recovery to a certain degree of its initial height. This suggests that full plastic deformation does not occur in alginate cryogel material, affirming its status as a highly resilient material. Furthermore, textural properties were calculated using two compression cycles with a 5 mm/min crosshead speed and varying degrees of deformation. The obtained data revealed that the testing protocol for texture measurement provides valid results when using data at 30% deformation, regardless of crosshead speed.

Keywords: Compression test, Mechanical properties, Textural properties.

1. Introduction

Textural characteristics are important quality properties of the food product and key drivers of food acceptability or rejection. In fact, food products should be formulated to have a satisfying taste. Among textural characteristics, crispiness is important attribute which is expected for many food products such as fries, chips, breakfast cereals, breadcrumbs, cookies, and some fresh food (Boudina et al.2023; Fillion et al. 2002). In addition, crispness, crunchiness, and chewiness are textural attributes often associated with the freshness and firmness of natural produce and manufactured foods (Tunick at al. 2013). The difference between crispness and crunchiness is that crispness is relatively more associated with the first snap, while crunchiness may be relatively more associated with chewing, grinding, and successive breaking (Luyten et al. 2004; Ma et al. 2024). Crispness and crunchiness have traditionally been associated with the mechanical force required to compress food until it fractures into small pieces, but these relate to the ease of fracture, or fracture ability brittleness of a structure. Crispness is usually evaluated by biting with incisors and crunchiness is generally determined by chewing with molars. To obtain crispness and crunchiness data, researchers most use mechanical methods. Mechanical techniques resemble mastication and include flex, shear, and compression (Tunick at al. 2013).

Food-grade aerogels are a relatively new class of materials that have gained attention for their potential applications in the food industry. These porous materials are composed of solid particles with interconnected pores filled with the air, resulting in a lightweight, highly porous structure. Aerogels are generally produced by removal of solvent from the crosslinked polymer networks. i.e. gels, by the use of supercritical drying or freeze-drying method. Both methods aim to remove solvent with minimal capillary induced shrinkage of the polymer gels (Beaux et al. 2022). Food-grade aerogels are typically obtained from biopolymers that are biodegradable, biocompatible, and edible. They can be used to enhance the texture of food products, such as chocolate, candy, and baked goods, creating a lighter, crispier, or the more airy texture (Plazzotta et al. 2023). Aerogels could also help to absorb moisture and prevent spoilage, extending the shelf life of perishable products (Berardinis et al. 2004). Additionally, they can be used to encapsulate and control the release of flavors, additives, or active ingredients, providing a more targeted and sustained effect (Selvasekaran et al. 2021; Selmer et al. 2015). Moreover, aerogels can be used as templates for the oil, to reduce fat content in food product, and to provide a creamier, smoother, or crispier texture of confectionary, bakery and dairy products, in comparison to the traditional oils (Ferdaus et al. 2024). Hence, textural properties of food-grade aerogels are

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essential parameters, in order to evaluate their ability to be applicable in food products.

Alginate is a biopolymer that has been extensively employed in the food and biomedical domains, as well as in packaging and textile industry (Maity et al. 2022). Alginate is a polysaccharide containing β-D-mannuronic acid (M block) and α-L-guluronic acid (G block), linked by 1,4- glycosidic bonds. It is extracted from brown algae (Yang et al. 2022). It has been widely used in food for more than 40 years as a thickening agent, stabilizer, and gelling agent (Rahman et al. 2024; Pascuta,et al. 2022). Alginate has ability to form the gels in the presence of divalent cations, especially Ca^{2+} by egg-box mechanism (Yang et al. 2022).

This work is focused on analysis of the textural properties of the alginate cryogel, in order to approximate their potential usage in smart functional foods. The calculation of the texture properties, based on extracted data from compression test, presents a novel, modified method for testing protocol of texture analysis, which contributes to better understanding of such important parameters of food products, and sensory perception.

2. Materials and methods

Investigated alginate cryogels are obtained from alginate hydrogel according to procedure detailed described in previous work (Meseldzija et al. 2024). The compression test was conducted using an Instron Model 1185 Dynamometer equipped with a 10 kN load cell at a temperature of 22 °C and 49% relative humidity. Specimens were 16.0±0.5 mm in diameter, and 22.0±0.5 mm in height. Compression test was performed with various a crosshead speed (0.5, 1, 2 and 5 mm min^{-1}) with same loading and unloading speed, up to 95% of specimen height.

The modified testing protocol for texture profile analysis (TPA), contains two compression tests where first compression was done as height controlled and second as load controlled test. The first compression is set to achieve 70%, 50% and 30% deformation of the initial specimen height. The maximum applied load at the height reached has been designated as P1, while P2 is the maximum load for the second compression (set to be 80% of P1). Compressions were done with a crosshead speed of 5 mm min^{-1} for loading, and 10 mm min^{-1} for unloading. The recovery time between the first compression and the second compression was approximately 30 minutes.

3. Results and discussion

Compression test was carried out up to stage where specimen becomes the most flattened (Figure 1). It should be noted that with increasing the crosshead speed the holding time at maximum applied load was increased. The results show that alginate cryogel is very flexible and resilient material. It was noted that even after specimen is being compressed more than 95% of its starting height, at maximum load and extended holding time, the full plastic deformation would not occur. The compressed specimen underwent partial recovery; in this case, nearly 50% of its compressed height was recorded as recovered.

Fig.1. Compression test up to 95% of specimen height

Stress-strain curves obtained from compression tests with various crosshead speed were displayed in Figure 2. Results indicate negligible influence of a crosshead speed on compression results if 70 % of strain is observed.

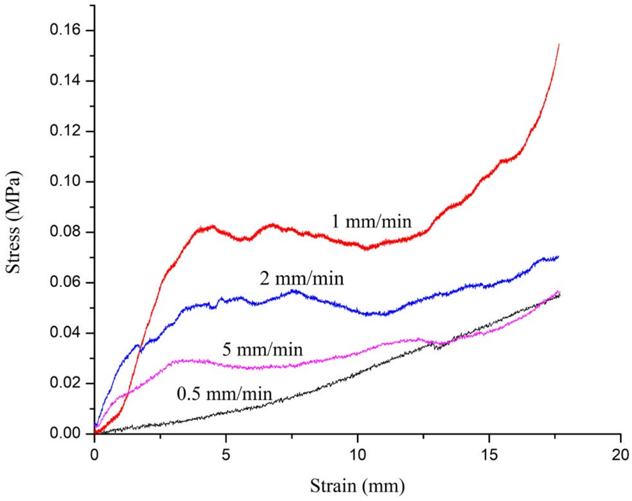

Fig.2. Stress-strain curves obtained from compression test up to 95% of specimens' height, and observed up to 70% of strain

Devices for texture measurement are able to perform test within seconds imitating actual food chewing. Herein, modified testing protocol contains two compression cycles as simulation of chewing but slowed down. In the previous work, it was shown that textural properties extracted from the compression cycles carried out with a crosshead speed of 0.5 mm min^{-1} are in the agreement with literature (Meseldzija et al. 2024). This study aims to investigate influence of the crosshead speed during compression tests on the final results i.e. calculated textural properties. The used crosshead speed for loading was 5 mm min^{-1}, and unloading 10 mm min^{-1}, with specimen deformation of 30 %, 50 % and 70 % (Figure 3).

Fig.3. Compression test up to 30 %, 50 % and 70% of specimen height

The force-versus-time curves for both compressions are shown in Figure 4. As expected, the maximum applied load increases as the degree of deformation increases, with the lowest applied load at 30% of deformation (Figure 4a) and the highest applied load at 70% of deformation(Figure 4c).

The qualitative characteristics of food, often described as textural properties, encompass a broad range of attributes including mechanical, geometrical, and microstructural qualities (related to moisture and fat content). A common classification system (Szczesniak 1962) categorizes mechanical properties into two primary groups: primary parameters (hardness, elasticity, viscosity, cohesiveness, and adhesiveness) and secondary parameters (brittleness, chewiness, and gumminess). If the relations for textural properties calculation are observed, the

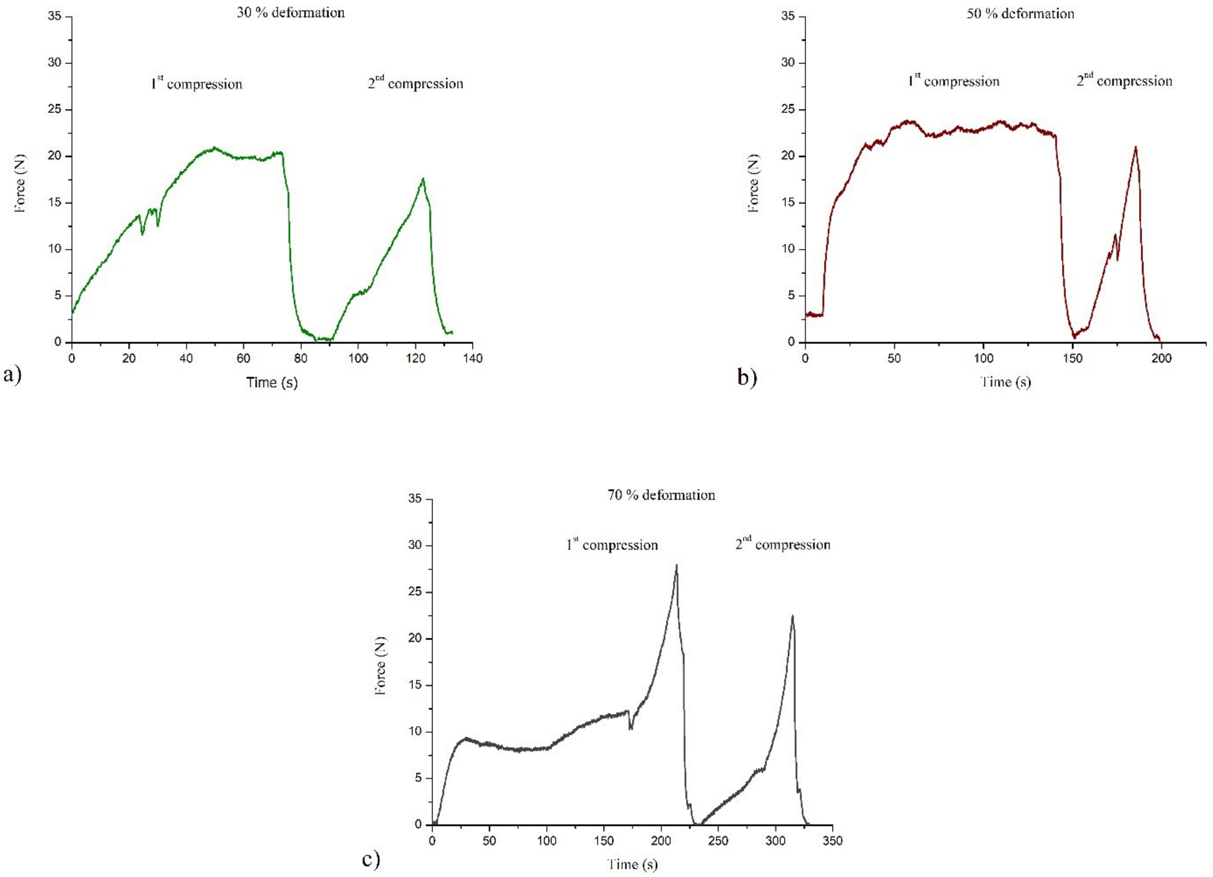

Fig.4. Two compression cycles at a) 30 %, b) 50 % and c) 70 % deformation of the specimen height

textural parameters may be classified as: (i) “fundamental mechanical” parameters - the one directly related to maximum force (hardness/strength, and fracturability), (ii) “basic textural” parameters calculated from the force-versus-time curves (elasticity, cohesiveness, springiness, resilience), and (iii) “complex textural” parameters calculated using relations which involves combining “fundamental mechanical” and/or “basic textural” parameters.

The mechanical and textural properties that were observed in this study are shown in Table 1. Force represents maximum applied force, while strength is maximum applied force per specimens’ area (Meseldzija et al. 2024). Crispiness is considered as a strain energy which is calculated as the area under the first part of the stress-strain curve (10% of strain, Figure 2) (Aprilia et al. 2019). Resilience is material resistance to plastic deformation under applied load. It is calculated when value of the area under the unloading curve is divided by value of the area under the loading curve during 1st compression (Meseldzija et al. 2024). Chewiness is materials resistance to compression during chewing, and it is calculated as multiplication of force, springiness, and cohesiveness values (Meseldzija et al. 2024; Paredes,et al. 2022). The results obtained were compared with the literature, where the data extracted at 30% of deformation are similar to those already published, despite the differences in sample size and shape. On the other hand, data extracted at 50 % and 70 % of deformation are comparable to literature data when the influence of plastic deformation is considered.

Table 1. Mechanical and textural properties of the alginate cryogel materials.

Degree of the deformation (%)	Force (N)	Strength (KPa)	Crispiness (N)	Resilience	Chewiness (N)
30*	21.308	64.72	/	0.218	14.899
30	21.03	98.40	0.137	0.23	12.02
50	23.88	93.89	0.302	0.37	6.36
70	27.97	107.56	0.338	0.13	6.72

*Literature data (Meseldzija et al. 2024).

Conclusions

The study of the mechanical properties of alginate cryogels for application in food preparation has attracted much attention in recent years, since the data obtained from compression tests are used to calculate parameters related to texture. Appropriate texture measurement protocols are crucial for collecting valid data. In present study the modified protocol was introduced. It was shown that crosshead speed does not have significant influence on the collected data when 80 % of strain is observed. Data were collected at 30 %, 50 % and 70 % of material deformation. Textural properties (chewiness, crispiness, and resilience) are calculated at each deformation stage, and compared to literature. It can be concluded that texture measurement protocol established up to 30 % material deformation in the first compression provides the most matching results with literature. On the other hand, the influence of plastic deformation must be taken into account if the measurement protocol includes more than 30% deformation.

Acknowledgments

This study was supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Contract No. 451-03-66/2024-03/200017).

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