# INFLUENCE OF PROCESSING METHODS ON MECHANICAL AND STRUCTURAL CHARACTERISTICS OF VACUUM MICROWAVE DRIED BIOPOLYMER FOAMS

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**Abstract:** Control of physical and mechanical properties of biopolymer (derived from food hydrocolloid) porous solids in terms of stress strain relationship during compression, porosity and pore size would enable their use for a wider range of purposes. Different types of dried cellular biopolymer foams were produced using different food hydrocolloids such as locust bean and alginate gums, gelatin, low and high methoxy pectin, methyl cellulose and starches (corn and tapioca) at various proportions. First different types of wet hydrogels were prepared by varying gel processing methods. Then they dried using microwave energy under vacuum called vacuum microwave drying. Before performing the drying process the initial Young's modulus of the hydrogels was measured. Pore size analysis and distribution percentage were done using mercury pore size analyser after drying. Relationship between the pore size distribution after drying and the initial Young's modulus was developed. Compressive test was performed for dried porous solids and true stress strain relationship curves were developed to classify nature of dried foams obtained from various gel types. Scanning Electron Microscopic study of individual samples was performed to view the internal structure of dried porous biopolymers.

**Keywords:** vacuum microwave drying; Young's modulus; stress–strain curve; pore size; mercury pore size analyser.

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## INTRODUCTION

Biopolymers from food hydrocolloids are natural and biodegradable materials and produce no toxic compounds. They are derived from polysaccharides, yielding fine textured gels at low polymer concentration, or from proteins using higher polymer concentrations (Rassis *et al.*, 1997). Biopolymer foams are getting great significant in biomedical applications such as drug carrier after surgery, vitamins and mineral carrier, microbicidal sponges, scaffold for tissue regeneration and so on (Harris, 1990). Generally biopolymer gels have a low solid content and therefore require extensive drying (Rassis *et al.*, 2002; Reifen *et al.*, 1998; Eichler *et al.*, 1997). It is very clear that successful gel-based foams should exhibit appropriate mechanical strength along with appropriate chemical compatibility, sorption capacity and biodegradability for its intended use.

Dried biopolymer foams have a low density and low mechanical strength based on the cell wall and the entire cellular structure. The most valued properties of cellular foams are their density, pore size distribution, Young's modulus and mechanical strength. Dried polymeric foams usually have relative densities of less than 0.3 kg $m^{-3}$. Different structures of foams lead to a wide range of such properties and a much greater availability. Water plasticization of foams changes their stress–strain behaviour during mechanical compression. Vacuum dried gels, conditioned to water activity 0.33 were collapsed by brittle fracture. Sponges conditioned to water activity 0.57 and 0.75 were appeared to collapse by elastic buckling (Attenburrow *et al.*, 1989).

Varying the preparation procedures can modify the mechanical properties of these sponges (Nussinovitch and Gershon, 1997). For example, internal gas bubbles contained in wet agar gels drastically reduced the mechanical integrity of the dried sponges and affected their porosity. However, the same process in alginate sponges caused only minor mechanical changes (Nussinovitch *et al.*, 1993). Oil included in alginate gels weakened the mechanical strength of the dried sponges, lowered stress and stiffness at failure as reflected by the deformability modulus,

and changed the size distribution and structure of pores of the dried sponges (Nussinovitch and Gershon, 1997).

Gibson and Ashby (1988) described the relationship between the structure of cellular foams and their mechanical properties. The mechanical properties have been evaluated through uniaxial compression tests, which can be performed using a texture analyser. The typical compressive stress–strain curve of the cellular solids characterizes foam type into three category *viz.*, brittle, plastic and elastomeric foams. Each type of curve is characterized by three regions. First is the linear elastic region, followed by the cellular collapse region and the densification region. The first region corresponds to elastic deformation, which is the result of cell wall bending. The second region corresponds to collapse of pores by elastic buckling, brittle crushing or plastic collapse depending on the foam water activity, temperature and type of material used to produce the foams. The densification region corresponds to the collapse of the pore structure throughout the material and subsequent loading of the pore edges and faces against one another. Since many polymeric types of foam have low relative density, they deform to large strains before densification.

Biopolymer based foams are most commonly subjected to freeze-drying or vacuum drying for processing. The resultant dried cellular materials are an interconnected network of solid struts that form a cellular solid structure. The foams produced by freeze-drying method have reduction in porosity, and dense or non-porous structures by vacuum drying are common problems faced by many researchers (Nussinovitch and Gershon, 1997; Nussinovitch *et al.*, 1993; Attenburrow *et al.*, 1989). In replacement of above drying methods an investigation was undertaken to determine if the application of microwave under vacuum would result in useful microstructure and mechanical strength for biomaterial (Jaya and Durance, 2005; Durance *et al.*, 2005).

In vacuum microwave drying method there is no need to take additional steps to create pores in the foams like in other methods *viz.*, salt leaching, gas foaming, phase separation and so on. In this drying, the pores are formed by a pressure difference established between the inside and outside of the tissue due to steam generation (Durance and Wang, 2002; Scaman and Durance, 2005). Another advantage of using vacuum microwave method is that there will not be use of any organic solvents for sponge production. Method of sponge preparation using organic solvents requires additional processing step to remove the solvents, because presence of solvent in the sponge affect the cell proliferation and added drugs and nutrients. Actually in vacuum microwave drying even absorption of electromagnetic energy can be achieved by physical movement of the material through the microwave field. The microwave energy is absorbed directly into the material. If this process takes place in a vacuum, quick drying will occur, along with material generating pores. Thereafter the porous form of the product can be stabilized by further drying to increase tissue rigidity. During the time of dehydration due to the effect of thermal energy additional cross linking of the hydrogel material will be taking place. In this work different biopolymer foams were produced as a model from different food hydrocolloids namely gelatin, sodium alginate, low and high methoxy pectin, methyl cellulose, tapioca and corn starches. The major objectives of this research are to produce biopolymer foams from different food hydrocolloids using vacuum microwave drying technique and to evaluate the mechanical and structural characteristics of biopolymer foams and their effect on the processing methods.

## METHODS AND MATERIALS

### Raw Material

Seven different types of biopolymer gels were prepared to study their mechanical and structural characteristics of dried foams using vacuum microwave energy. Food grade gelatin, pectin (low and high methoxy), methyl cellulose, glycerol, corn and tapioca starches were used to prepare the gels. The gel combinations were selected based on their binding nature with each other, compatibility with vacuum microwave drying and gelling characteristics (Durance *et al.*, 2005). All biopolymer gels were prepared as a model gel to study the effect of vacuum microwave drying on their physical and mechanical properties.

### General Gel Preparation Method

Known amounts of biopolymers were mixed with water and glycerol. Glycerol was added as a surfactant. All were mixed well using a hand blender to get a homogeneous solution. Approximately equal amounts of the homogeneous solution were poured into small muffin cups (bottom inner diameter 43 mm, upper inner diameter 56 mm and height is 25 mm). These cups were placed in a freezer at $-80^{\circ}$C (Forma Bio Freezer, Forma Scientific) to quick freeze and mould the solution. The frozen molds were separated from the cups and cross linked with appropriate cross linking agents. This made the solution to become a gel with stable and soft solid structure. During the cross-linking time, thawing of the frozen moulds and cross-linking takes place simultaneously.

### Different Types of Model Gels

*Gel type 1a (AMS1)*

AMS1 was prepared by mixing sodium alginate (3%), methyl cellulose (1%), tapioca starch (2%) and glycerol (0.5%), with 95% water, which is calculated excluding the amount of glycerol. This was cross linked with calcium chloride by immersing the frozen molds into 1% calcium chloride solution for 16–18 h.

*Gel type 1b (AMS2)*

This was prepared similar to the type 1. The only variation was incorporation of sodium bicarbonate and citric acid. One percent of sodium bicarbonate was mixed along with other ingredients. During cross linking, 1% citric acid was added along with 1% calcium chloride. The percentage of salt and acid was not included as total solid to calculate the water percentage. During the gelling process, gelling and acid foaming took place simultaneously.

*Gel type 2a (GSP1)*

Gelatin 10%, low methoxy pectin 5%, tapioca starch 10% and glycerol 1% were mixed with 75% of water. The amount of glycerol is not considered to calculate the water percentage. To get homogeneous mix, heating the mix using boiling water bath and mixing with hand blender was