



THE INFLUENCE OF THERMAL TREATMENT ON PHYSICAL PROPERTIES OF GUAR GUM

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ABSTRACT

In this study we treat the guar gum by appointed temperature and time in full factorial design this result in different viscosity grads of guar gum according to different temperatures and time, the un expected increase in viscosity at temp.50 to 70 this corresponds to solubilisation of the polymer then there is a gradual decrease in viscosity with increasing temperature and time due to polymer degradation.

When we compare the treated guar gum with non-treated and xanthan gum we found different physical properties, less viscosity and swelling capacities and good compressibility of treated guar.

KEY WORD: Guar gum, treated guar gum, temperature, viscosity, swelling, bulk density, tapped density, compressibility

1. INTRODUCTION

1.1. Guar gum

Guar gum is a polysaccharide derived from the seeds of *Cyamopsistetragonolobus* and many reports in the literature have proved its efficacy for colonic drug delivery. It consists of linear chains of (1→4)-b-D-manopyranosyl units with α-D-galactopyranosyl units attached by (1→6)-glycosidically linked galacto-pyranoses linkages. The main components are polysaccharides composed of D-galactose and D-mannose in molecular ratios of 1:1.4 -1: 2. (Goldstein, et al, 1973).

1.1.1. Physicochemical characterization

Guar gum occurs as an odorless or nearly odorless, white to yellowish-white powder with a bland taste which is practically insoluble in organic solvents. In cold or hot water, guar gum disperses and swells almost immediately to form a highly viscous, thixotropic sol. The optimum rate of hydration occurs at pH 7.5–9.0. Finely milled powders swell more rapidly and are more difficult to disperse. Two to four hours in water at room temperature are required to develop maximum viscosity (4860 cps for 1% w/v dispersion) and the viscosity depends on temperature, time, concentration, pH, rate of agitation, and particle size of the guar gum powder (Rowe, et al 2006).

To understand the solution properties of guar as well as other water-soluble biopolymers, it is often necessary to degrade the native polymer to prepare samples with various molecular weights (MW). These polymer samples can be used to study the MW dependency of the physicochemical properties of the polymer.

Guar undergoes depolymerization through several mechanisms, including shear flow, ultrasonic irradiation (Tayal, et al., 1999), chemical and enzymatic cleavage (Tayal, et al., 1999; Cheng, et al., 2002) to yield large amounts of guar samples with various, consistent, high polydispersity and broad MW dependency. However, utilizing acid hydrolysis, the Huggins coefficient for degraded guar is much smaller than that of the native guar, suggesting a weakening of aggregations in guar by the acid degradation.

It has been reported that guar gum can be degraded in to lower molecular weight fragments using free radical generators like potassium persulphate and hydrogen peroxide (Reddy and Tammishetti, 2004). Though both thermal energy and microwaves could effect depolymerisation, the latter did so at a more rapid rate than the former. Thus prepared lower molecular weight guar is shown to have better film forming properties.

To enhance mechanical stability of guar by chemical cross-linking which is a highly versatile

method to create hydrogels with good mechanical stability, Gliko-Kabir et al. (1999) have reported some hydrogels of GG cross-linked with glutaraldehyde (GA) and phosphate whereas Burruano et al. (2002) have proposed a guar gum-based formulation that is cross-linked with borate for the development of synthetic cervical mucus. On the other hand, some reports showed that esterification of guar gum increases its degradation rate and decreases its thermal stability, with the exception of the maleates of the guar esters (Patel, et al., 1988).

Ko'k MS, et al. (1999) were capable to show that guar gum has a relatively low activation energy for depolymerisation, but high rate constants in the temperature range of interest. Moreover, in a study conducted by Bradley et al. (1989), it has been concluded that thermal degradation in particular results in the production of significant quantities of low molecular weight material with a consequent large increase in polydispersity.

Guar gum has been successfully modified by grafting with acrylamide to form graft copolymer pAAm-g-GG, which showed a better thermal stability than guar gum the microspheres developed here may serve to be good biomaterials for the controlled release of both water-soluble and waterinsoluble bioactive materials (Soppirath and Aminabhavi, 2002). Hydroxyethyl guar gum (HEG) derived from natural guar gum, reaction with ethyleneoxide, was reported to have a good dispersibility in water, and high compatibility with anionic, cationic and nonionic surfactants (Ling-Sa He, et al. 2007), moreover, the authors concluded that with its excellent electrolyte resistance, viscosity stability and good shear stability, HEG can be used in fabric printing and fracture fluids, etc.

HEG is made from guar gum by reaction with ethyleneoxide. As with other synthetic polymers, HEG is a heterogeneous group of molecules. Its chemical, physical and pharmacological properties are primarily dependent on molecular weight, molecular weight distribution, the amount of hydroxyethyl substituents, and the pattern of that substitution. The ability to rapidly and accurately determine the degree of the substitution is a critical factor in proper control of chemical, physical, and biological properties of HEG.

1.2. Xanthan gum

The USP NF 23 describes xanthan gum as a high molecular weight polysaccharide gum. It contains D-glucose and D-mannose as the dominant hexose units, along with D-glucuronic acid, and is prepared as the sodium, potassium, or calcium

salt. Xanthan gum produced by a pure-culture aerobic fermentation of a carbohydrate with *Xanthomonas campestris*. The polysaccharide is then purified by recovery with propan-2-ol, dried, and milled

Xanthan gum is widely used in oral and topical pharmaceutical formulations, cosmetics, and foods as a suspending and stabilizing agent (Rowe, et al 2006). It is also used as a thickening and emulsifying agent. It is nontoxic, compatible with most other pharmaceutical ingredients, and has good stability and viscosity properties over a wide pH and temperature range (Rowe, et al 2006).

Xanthan gum gels show pseudoplastic behavior, the shear thinning being directly proportional to the shear rate. The viscosity returns to normal immediately on release of shear stress. It has also been used to produce directly compressed matrices that display a high degree of swelling due to water uptake, and a small amount of erosion due to polymer relaxation (Rowe, et al 2006).

1.3. Experimental design and formulation optimization

The statistical design of experiments (DOE) is an efficient procedure for planning experiments so that the data obtained can be analyzed to yield valid and objective conclusions. DOE begins with determining the objectives of an experiment and selecting the process factors for the study. In an experiment, one or more process variables (or factors) are changed in order to observe the effect the changes have on one or more response variables. An experimental design is done in advance of doing the experiment and when properly selected, it maximizes the amount of "information" that can be obtained for a given amount of experimental effort. The statistical theory underlying DOE generally begins with the concept of process models of Fisher (1926) and Myers (1990).

1.3.1. Full factorial designs

It is one of the DOE in which every setting of every factor appears with every setting of every other factor in a full factorial design. Full factorial designs not recommended for 5 or more factors since the design under such condition requires a large number of runs and is not very efficient.

1.3.1.1. Three-level full factorial designs

Three-level designs are useful for investigating of the main effects and to some extent the quadratic interaction effects and it is written as a 3^k factorial

design. It means that k factors are considered, each at 3 levels. These are (usually) referred to as low, intermediate and high levels. These levels are numerically expressed as 0, 1, and 2. One could have considered also the digits -1, 0, and +1. The reason that the three-level designs were proposed is to model possible curvature in the response function and to handle the case of nominal factors at 3 levels. A third level for a continuous factor facilitates investigation of a quadratic relationship between the response and each of the factors. The simplest form of the three level full factorial design is The 3^2 design which has two factors, each at three levels (Myers, 1990).

2. SCOPE OF THE WORK

The specific goal of this study is to treat the natural gum (guar gum) aiming at reducing its viscosity and improve its compressibility to be suitable for direct compression.

3. MATERIALS AND METHOD

3.1. Materials

Guar gum (Batch 39B2B2_Expoct 2012) was a pharmaceutical grade product of Nanjing Co, (China). Xanthan gum (Batch XA075462 -EXP jan. 2011) pharmaceutical grade products of Shin Poong pharmaceutical Co. (South Korea), and donated by General Medicine Company

3.2. Instruments and Apparatus

The following instruments and apparatus were utilized in the study: Electronic balance (ABS 120-4, Germany); PH meter (Hanna instrument, Portugal); Magnetic stirrer (Stuart, England); Water bath (Clifton, England); Hot air oven (Memert, Germany); Brook field viscometer (Model HAD v-1 prime, USA)

3.3. Methods

3.3.1. Experimental design for thermal treatment of guar gum

The design adopted for thermal treatment of guar gum was the 3^2 full factorial experimental designs in which two variables (namely, temperature and time) were iterated at 3 levels. These were 60, 80 and 100 °C for temperature with 5, 10, and 20 minutes for time. The layout of the experimental design is fully described in Table 1

3.3.2. Thermal treatment of guar gum

The method reported by Bradley, et al (1989) for thermal treatment of guar gum was followed

where for each run, solution of guar gum was prepared by wetting a known weight of guar with a small amount of absolute ethanol (0.5 ml ethanol per 100 mg guar) to prevent aggregation during the dispersion process. The guar solutions was then dispersed in a mixed phosphate buffer (KH_2PO_4 , Na_2HPO_4) of pH 6.0 containing 0.2M NaCl using a magnetic stirrer for mixing, then heated for a pre-determined temperature and time for each run. The resultant content was then dried in hot air oven.

Table 1: Experimental design lay out for thermal treatment of guar gum

Run	Temperature °C	Time (min)
1	60	5
2	80	10
3	100	20
4	60	10
5	80	20
6	60	20
7	100	10
8	100	5
9	80	5

3.3.3. Viscosity determination of treated guar gum

Measurement of viscosity of resultant treated guar gum for each run was done in rotational viscometer (myer viscometer) where 2% treated guar gum solution prepared by dispersing gum in water and mixed by magnetic stirrer then placed in 250ml beaker, the viscosity is measured by using a suitable speed and spindle combination that gives torque more than 10% at temperature 27°C. Treated guar gum powders obtained from all runs were subjected to viscosity determination

3.3.4. Physical test

Upon thermal treatment of guar gum, samples obtained from all run were further subjected to preformulation testing seeking the possibility to utilize them in dosage form fabrication.

3.3.4.1. Swelling ratio test

The method was described by Jani (2008) and was carried out in a 100-mL stoppered graduated cylinder. The initial bulk volume of 1 g of dried powder was measured, and water was added in sufficient quantity to yield a 100-mL uniform dispersion. The sediment volume of the swollen mass was noted after 24 h storage at room temperature (27°C). The swelling ratio was calculated by determining the ratio of the swollen volume to the initial bulk volume.

3.3.4.2 Bulk and tapped density determination

A method reported by Jani (2008) was followed where preweighed and preserved quantity of dried powder was poured into a graduated cylinder, and the volume recorded. The cylinder was tapped until the powder bed volume reached a minimum value, and the tapped volume was recorded. The bulk and tapped densities were calculated. Hausnerratio and Carr index was then calculated from the bulk and tapped densities.

4. RESULTS AND DISCUSSION

4.1. The influence of thermal treatment on physical properties of guar gum

Upon treatment of guar gum with a reported thermal handling, a series of comparisons has been made between the treated and non treated guar gum in order to explore the possible impacts of thermal treatment on the physical performance of guar gum.

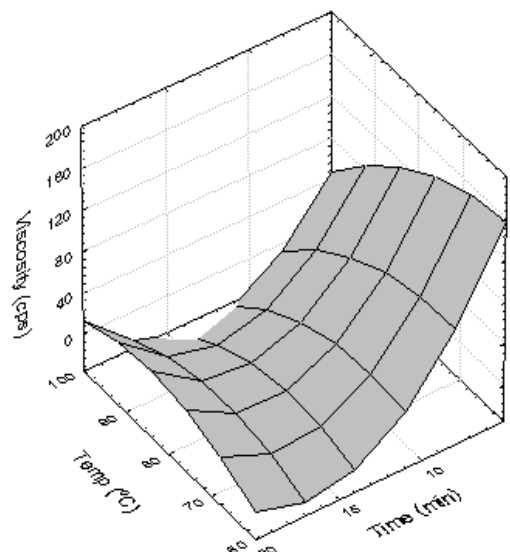
4.1.1 Viscosity

As a result of thermal treatment of guar gum there are two situations that seem to affect the polymer viscosity, the increase in viscosity due to increased solubilisation, and the loss of viscosity due to decreasing of molecular weight caused by thermal degradation (Kok et al., 1999). Temperature causes the water molecules to loose their ordering around the guar molecules, thus affecting the conformation and resulting in reduced-viscosity behavior (Finney and Soper, 1994).

Figure 1 shows a quadratic surface response plot for the impact of temperature and time (as independent factors) on viscosity of thermally treated guar gum. From the plot it appears that both factors affect the viscosity of the guar. There is a marked increase in viscosity at temperatures above 50° C and this corresponds to solubilisation of the polymer. In excess of 80°C, however, the degradation effects offset any further increase in solubilisation (Figure 2). This might be attributed either to the increased energy dissipation movement of the molecule due to elevated temperature or to the decreased inter-molecular interactions, which in turn decrease the interference of the hydrodynamic domain (Chen and Chen, 2001).

Other reason for this behavior could possibly be explained in terms of the reduced effective volume fraction or a less entangled state of guar solution upon the addition of salts where in this concern, Cybulska and Doe (2002) were able to show that upon interaction with water, salts reduce the

availability of water in the solution preventing the expansion of the guar networks, thereby decreasing the viscosity.



$$\text{Viscosity} = 204.7778 + 6.0583 * (\text{temp}) - 57.4476 * (\text{time}) - 0.0621 * (\text{temp}^2) + 0.2214 * (\text{temp} * \text{time}) + 1.3244 * (\text{time}^2)$$

Figure 1: fitted quadratic surface plot showing the relation between temperature, time and viscosity for thermally treated guar gum

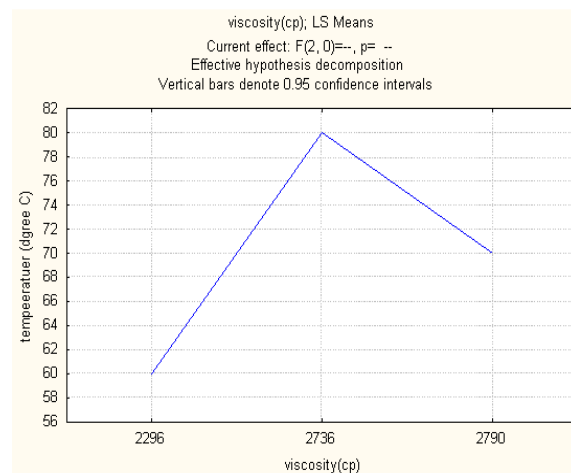


Figure 2: The effect of temperature on viscosity of guar gum

Coefficient terms associated with the regression equation accompanying the fitted surface plot (Figure 1) revealed that both temperature and time has an effect on viscosity of thermally treated guar gum; however, the influence of timing seems to be more sound than that of temperature.

Moreover, there was a large decrease in viscosity after drying of the treated guar gum which might be due to creation of treated guar gum with weak

bond as a result of thermal degradation and salt incorporation as discussed earlier. In fact, the obtained result has necessitated the study of the effect of drying method on the viscosity of thermally treated guar gum as shown in Table 2.

Table 2: Viscosity for treated guar gum after oven drying

Run	Temperature °C	Time (min)	Viscosity (cp)
1	60	5	163
2	80	10	14
3	100	20	12
4	60	10	9
5	80	20	8
6	60	20	12
7	100	10	9
8	100	5	8
9	80	5	159

4.1.2. Swelling capacity

Results of swelling studies are tabulated in Table 2 and it appears that the thermally treated guar gum has less capability to swell compared to non treated (native) guar gum (7:10) and to xanthan gum (7:8). The obtained result is in agree with relevant published works (Kadari, 2001; Dumetrio, 2002; and Omidian, et al., 2010).

The result can be endorsed by the low hydration capacities of the small polymer molecules generated as a result of degradation during thermal treatment. During dissolution of a polymer and polymer swelling, the load originates from the interaction of a polymer and a solvent as well as concentration gradient of ions inside the polymer structure and the solution. The solvent molecules move into the glassy polymer matrix with a well-defined front at a particular velocity and simultaneously, the thickness of the swollen or rubbery region increases with time in the opposite direction (Kadari 2001). Apparently longer end-to end distances are potentially obtained if the polymer chains are longer and have more interaction with the solvent. A hydro gel swells in water or in any aqueous medium because of positive forces (polymer-solvent interaction, osmotic, electrostatic) and negative forces (elastic) acting upon the polymer chains (Omidian, et al., 2010). Unsurprisingly, the swelling of guar gum was found to be affected by the viscosity of the gum (Dumetrio, 2002) as can be traced from Table 2.

It should be noted that swelling behavior reflects the ability of dosage form to uptake aqueous fluid and hydrate. Rate of hydration indicate how fast the dosage form imbibes water whereas the extent

of such hydration measures the diffusional path length for the drug and hence determines the rate of drug release from the dosage forms (Nur and Zhang, 2000).

4.1.3. Compressibility

Compressibility and compatibility of a powder are influenced by the flow properties, and in the micro scale, by the adhesion forces between particles. Moreover, the percent compressibility indirectly provides an excellent representation of uniformity in size and shape, deformability, surface area, cohesion, and moisture content (Carr 1965). Furthermore, crystal morphology and particle size have a direct impact on the filtration of crystal slurry, cake compressibility, bulk stability, bulk density, and flow characteristics.

Results obtained for compressibility testing showed that thermal treating of guar gum has resulted in lowering of compressibility from 25 to 17% (Table 3).

Table 3: Swelling and compressibility testing of treated guar, native guar and xanthan gums

Polymer	Powder swelling	Compressibility index
Guar non treated	10 fold	25%
Guar treated	7 fold	17%
Xanthan	8 fold	27%

Values of compressibility index below 15% usually give rise to good flow characteristics, but readings above 25% indicate poor flowability and between these two values, less than optimum performance might be anticipated (Marshal, 1986). In other words and owing to thermal degradation, thermal treatment of guar gum render the polymer of small molecules with increased homogeneous arrangement and flow. This assumption relies on the fact that particle size distribution is known to directly influence physical properties of powders, such as dissolution rate, powder flow, bulk density, and compressibility (Howard 2003).

5. CONCLUSION

Thermal treatment of guar gum at 70° for 10 minutes is an efficient tool to produce guar gum with desired properties for pharmaceutical processing and industries. The treatment has resulted in production of treated guar gum with

improved flowability, swellability, compressibility. On the other hand, method of drying seems to have a significant influence on viscosity of the resultant treated guar powder and verification of such effect might necessitate more collaborated extended study.

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