CHARACTERIZATION AND STANDARDIZATION OF GUM KARAYA

Sarathchandiran I¹ and Suresh Kumar P²*

¹Professor & Principal, Department of Pharmaceutics, Gokula Krishna College of Pharmacy, Sullurpet 524121, Andhra Pradesh, India.
²Research Scholar, Department of Pharmaceutics, PRIST University, Vellam, Thanjavur 613403, Tamil Nadu, India.

ABSTRACT
Floating systems have lower density than the gastric juice; hence residence time and the bioavailability of drugs absorbed in the upper part of the GI tract will be improved. Intragastric floating is useful for administration of drugs with specific absorption site and used for gastric diseases. So Gum Karaya (GK) may be used as a polymer for development of FDDS system due to poor solubility at acidic environment, swellability and low density, moreover it is biocompatible and biodegradable material. GK samples were examined for degree of deacetylation (DD), quantification by FTIR, DSC, XRD, SEM, CNMR, MS and standardized. The DD for GK was determined by potentiometric titration and DDA% was determined at various time intervals from 2 to 20 min. At 14th min 64% of DDA was obtained, at this stage GK exhibited desired properties required for developing FDDS, confirmed by determining density, swelling index, viscosity and solubility. The XRD profile exhibits well resolved and intense peaks, while a broad diffuse scattering and less intense peaks were found at 10.4° and 20.8°, crystallinity percentage was found to be 62.4 and 44.6 respectively indicating partially crystalline polymorph showing antiparallel compact structure. SEM photographs show a lamellar organization and dense structure. The absence of methyl proton resonance from protein between 1.0 and 1.5 in C NMR spectrum indicates the purity. The physicochemical properties were found within limits. Based on the results of characterization and standardization, we concluded that modified GK has all desired properties that are required to develop FDDS.

Key Words: SEM, XRD, C NMR, FTIR, DSC, Gum Karaya.

INTRODUCTION
Gum Karaya is also known as Sterculia gum obtained from Sterculia Roxburgh and other species of Sterculia (Family – Sterculiaceae). Commercial Gum Karaya is commonly obtained from tapping or blazing the mature Sterculiaurens, a large busgy tree growing widely throughout the Indian sub-continent. The average tree can be tapped about five times during its lifetime, with a total yield of up to five kilos per season. The exudates are allowed to solidify on the tree. The native collectors pick this crude gum. These gum ‘tears’ are broken into fragments less than 25mm in diameter, then cleaned, sorted and graded according to colour and purity before selling.

Gum Karaya has been used as an emulsifier, stabilizer and thickening agent for many years. Many traditional applications have been replaced by more cost-effective gums, or by a combination of those hydrocolloids. Gum Karaya, itself a strongly acidic polysaccharide shows good stability in acidic preparations and is used as a food additive. Less than 10% of total production is used in food applications. However, the principal use of Gum Karaya is the pharmaceutical preparations.
Natural Gum Karaya is a complex, branched, partially acetylated polysaccharide with a reported molecular weight of 9 million and 16 million Dalton. An average, new Gum Karaya contains about 10-14% acetyl groups, from which free acetic acid is formed and is split off as an ageing. Increased temperature, humidity and fine particle size increases the rate of acetic acid formation (Food Chemicals Codex, 1996).

After acid hydrolysis, Gum Karaya commonly produces D-galacturonic acid, D-galactose, L-rhamnose and small proportions of D-glucuronic acid. The total uronic acid residue content in the gum can be up to 35-40%. The remaining sugar residues are neutral. About 1% of proteinaceous components are also bound on structure, but the amino acid compositions vary widely with the different species (Anderson et al., 1985). Commercial Gum Karaya contains about 30-43% galacturonic acid, 13-26% galactose and 15-30% rhamnose after acid hydrolysis. Calcium and magnesium are the major cations linked with uronic acid on the gum structure. Gum Karaya has a much higher rhamnose content than other commercial exudates gums. Structurally, it is a heavily acetylated acidic polysaccharide containing α-D-galacturonic acid α-L-rhamnose residues as the main chains with 0-4 of the acid and 0-2 of rhamnose linkages and the acid is linked by 1,2 linkage of β-D-galactose or by 1,3 linkage of β-D-glucuronic acid on side chains, where on half of the rhamnose units carry at 0-4 by 1,4 linkage of β-D-galactose units (Stepien et al., 1995).

Regulatory status Gum Karaya is classified as a generally recognized as safe (GRAS) within the USA. It is also classified as ‘ADI not specified’ by the Joint WHOFAO Expert Committee on Food Additives (JECFA) and has the number E416 in the list of additives approved by the Scientific Committee for Food (SCF) of the EEC. Hence, the aim of the present study was to investigate the influence of the analytical methods on Gum Karaya degree of deacetylation (DD) value (Anderson et al., 1985).

**MATERIALS AND METHODS**

**Materials**

Gum Karaya samples were collected from Giriyan Co-operative Corporation, Government of Andhra Pradesh, India. Samples were used in the experimental analysis. Gum samples collected were stored in airtight polypropylene jars in desiccated condition.

**Preparation of deacetylated Gum**

Microwave method has been used for deacetylation of Gum Karaya can be highly facilitated by steeping in strong sodium hydroxide at room temperature before heating (Vinod et al., 2008). This steeping method has been adapted to samples for one day before conversion by microwave radiation method. A mixture of Gum Karaya and 45% NaOH was placed in a conical flask, covered tightly with cotton, and then subjected to microwave radiation. The mixture was then cooled with cold water and after filtration Gum Karaya was washed to neutral pH and freeze dried using VIRTIS Freeze mobile 5EL with sentry microprocessor control freeze dryer. Steps involved in demineralization process are given in following expression (AOAC, 1984).

**Determination of degree of deacetylation**

The deacetylation degree of Gum Karaya was determined by the potentiometric titration method using Calomel and silver/silver-chloride electrodes (Xuan Jiang et al., 2003). 0.5g of Gum Karaya was dissolved in 25mL of 0.1M standard aqueous HCl solution. The solution was then made up to 100mL with distilled water and calculated amount of KCl was added to adjust the ionic strength to 0.1. The titrant was a solution of 0.05M NaOH, pH was measured using pH meter under continuous stirring. The titrant was added until the pH values reached 1, the standard NaOH was then added stepwise and the pH values of solution were recorded and a curve with two inflection points were obtained. The difference between the volumes of these two inflection points corresponded to the acid consumption for the sialification of amine groups of Gum Karaya and permitted the determination of degree of acetylation, through the following Equation,

$$\text{DDA\%} = \frac{12.1(V_2-V_1)}{W} \times \frac{M_b}{\text{M}}$$

Where \(V_1\) and \(V_2\) are the base volumes referred to first and second inflection points respectively in mL, \(M_b\) is the base molarity in g/mol, and \(W\) is the original weight of the polymer in g.

**Characterization**

**Fourier Transform Infrared Spectroscopy (FTIR)**

Infrared spectra was measured by KBr-supported sample of Gum Karaya over the frequency range 4400-400 cm\(^{-1}\) at resolution of 4 cm\(^{-1}\) using a model 2000 Perkins-Elmer spectrometer performed at LailaImpex research centre, Vijayawada. The sample was thoroughly mixed with KBr, the dried mixture was then pressed to result in a homogeneous sample/KBr disc (Harpreet et al., 2006). The FTIR spectra was recorded and interpreted to find out the functional groups presents which were compared with reported reference standard.

**Differential scanning calorimetry (DSC)**
DSC was performed using a 10mg sample from ambient to 350°C at a heating rate of 10°C/min in a dynamic (50 mL/min) synthetic air atmosphere using DSC-50 Shimadzu automatic analyzer performed at Diya Labs, Mumbai. The optimum temperature to melt the Gum Karaya sample was found to be 137.4°C at -4.16(W/g) (Shaji et al., 2010).

**X-ray powder diffraactometry (XRD)**

The XRD measurement on powder sample was carried out to detect the crystallinity of the extracted sample of Gum Karaya. XRD study was performed by using a model D500 Siemens diffractometer at Diya Labs, Mumbai. The diffractometer was operated between 20 angles of 5° and 45°. Ni-filtered Cu Ka radiation was used as the X-ray source. The relative crystallinity of the polymer was calculated by dividing the area of the crystalline peaks by the total area under the curve (Shaji et al., 2010).

**Scanning electron microscopy (SEM)**

The surface morphology of Gum Karaya was observed using SEM. The dried sample of Gum Karaya was ground and then coated with gold under vacuum using a sputter coater. The scanning electron microscopy (SEM) was conducted using a JEOL JSM-630 J scanning electron microscope operated at 5.0 kV (Shaji et al., 2010).

**Nuclear magnetic resonance $^{13}$C NMR**

NMR spectra were recorded using Bruker II 600 spectrometer in 2% deuterated acetic acid in D$_2$O solution. The experiments were run at 70°C, temperature at which the solvent (HOD) peak does not interfere with any Gum Karaya peaks. After dissolution, approximately 1 mL of the Gum Karaya sample solution was transferred to 5 mm NMR tube. The sample tube was inserted in the magnet and allowed to reach thermal equilibrium for 10 min before performing the experiment. Then measured NMR spectrum was interpreted and compared with reported reference standard to conform the presence of Gum Karaya compound (Shaji et al., 2010).

**Mass spectroscopy study (MS)**

MALDI-TOF MS, Matrix Assisted Laser Desorption Ionization-Time Of Flight MS, involves mixing the sample with a matrix, which is then coated on to a plate or probe, and subjected to a collimated focused laser beam, causing ionization and desorption. MALDI enables analysis of molecules up to approximately 500kDa and has the advantage of producing large mass ions, with high sensitivity and little fragmentation. MALDI is now commonly coupled with Delayed Extraction (DE-MALDI), causing the produced ions to enter the Time of Flight analyzer at the same time. MALDI-TOF MS is used to determine molecular weight and may be employed in a large number of biochemical applications. Based on the interpretation of mass spectra the molecular weight of Gum Karaya was found to be 207.441 (Shaji et al., 2010).

**Standardization of Gum Karaya**

Partial deacetylation of Gum Karaya results in the production of modified Gum Karaya with desired characteristics to develop floating drug delivery systems. The degree of deacetylation is necessary to obtain a soluble product must be greater than 80–85%. Gum Karaya is commercially available in several types and grades that vary in molecular weight by 10,000–1,000,000, and vary in degree of deacetylation and viscosity (IP, 2006).

**General properties**

**Solubility**

Sparingly soluble in water; poorly soluble in 0.1N HCl and simulated gastric fluid; practically insoluble in ethanol (95%), other organic solvents, and neutral or alkali solutions at pH above approximately 6.5. Gum Karaya dissolves readily in dilute and concentrated solutions of most organic acids and to some extent, in mineral inorganic acids (except phosphoric and sulfuric acids). Upon dissolution, amine groups of the polymer become protonated, resulting in positively charged polysaccharides that are soluble in water; the solubility is affected by the degree of deacetylation.

**Viscosity**

A wide range of viscosity types are commercially available. Owing to its high molecular weight and linear, unbranched structure, Gum Karaya is an excellent viscosity enhancing agent in an acidic environment. It acts as a pseudo-plastic material, exhibiting a decrease in viscosity with increasing rates of shear. The viscosity of Gum Karaya solution increases with increasing concentration, decreasing temperature, and increasing degree of deacetylation. Viscosity of Gum Karaya (1%w/v) was performed by using Ostwald viscometer by dissolving the Gum Karaya in 2% acetic acid.

**Particle size distribution**

Particle size of powdered Gum Karaya was determined by sieve analysis. Sieves 40-180 were used and checked properly for their integrity.

**Determination of loss on drying**

Loss on drying is the loss in weight in % w/w resulting from water and volatile matter of any kind that can be driven off under specified conditions. The test is carried out on a well-mixed sample of the substance using IR moisture analyser. If the substance is in the form of large crystals, reduce the size by rapid crushing to a powder.

Weigh a glass-stoppered, shallow weighing bottle that has been dried under the same conditions to be employed in the determination. Transfer to the bottle the quantity of the sample specified in the individual
monograph, cover it and accurately weigh the bottle and the contents. Distribute the sample as evenly as practicable by gentle sidewise shaking to a depth not exceeding 10mm. Place the loaded bottle in the drying chamber (oven or desiccator) as directed in the monograph, remove the stopper and leave it also in the chamber. Dry the sample to constant weight or for the specified time and at the temperature indicated in the monograph. After drying is completed, open the drying chamber, close the bottle promptly and allow it to cool to room temperature (where applicable) in a desiccator before weighing. Weigh the bottle and the contents.

**Determination of sulphated ash**

Silica was heated to redness for 10 minutes, allowed to cool in a desiccator and weighed. The crucible 1g of the substance being examined was transferred and contents were weighed. The sample was ignited until the substance was thoroughly charred. The residue was moistened with 1mL of sulphuric acid, heated gently until the white fumes are no longer evolved and ignite at 800°C ± 25°C until all black particles have disappeared. The crucible was allowed to cool, few drops of sulphuric acid was added and heated. The operation was repeated until two successive weighing do not differ by more than 0.5 mg.

**Glass Transition Temperature**

The glass transition temperature (Tg) of the dry material was determined using Model DSC-50 Shimadzu automatic analyzer. Sample size varied between 5 and 10mg, heating rate was maintained at 40K min⁻¹. The temperature interval was set to -50 to 150°C. The Tg was determined by calculating the temperature of the half step height.

**Limit test for chlorides**

Gum Karaya solution was prepared as directed in the monograph and transfer to a Nessler cylinder. 10 mL of dilute nitric acid was added to this, further diluted to 50 mL with water and 1 mL of 0.1M silver nitrate was added. The solution was stirred immediately with a glass rod and allowed to stand for 5 minutes and protected from light. When viewed transversely against a black background any opalescence produced is not more intense than that obtained by treating a mixture of 10.0 mL of chloride standard solution (25 ppm cl) and 5 mL of water in the same manner.

**Limit test for heavy metals**

The limit for heavy metals is indicated in the individual monographs in terms of ppm, i.e., the parts of lead, Pb, per million parts (by weight) of the substance being examined.

**Standard solution:** 1mL of lead standard solution (20 ppm Pb) was pipetted and transferred in to 50mL Nessler cylinder and diluted with water to 25 mL. pH was adjusted with dilute acetic acid between 3.0 and 4.0.

**Test solution:** 25 mL of the test solution was prepared in 50mL Nessler cylinder as directed in the monograph by dissolving the specified quantity of the substance being examined in sufficient water to produce 25mL, pH was adjusted with dilute acetic acid between 3.0 and 4.0, dilute with water to about 35 mL and mixed well.

**Procedure:** 10 mL of freshly prepared hydrogen sulphide solution was added to each of the cylinders containing the standard solution and test solution respectively, mixed well, diluted to 50 mL, with water, allowed to stand for 5 minutes and viewed downwards over a white surface; the colour produced with the test solution was not more intense than that produced with the standard solution.

**Limit test for arsenic**

Limit test of arsenic was performed to determine the presence of trace amounts of arsenic by converting the arsenic in a substance under test to arsine, which was then passed through a solution of silver diethyl dithiocarbonate to form a red complex. The red complex so produced is compared visually, to the colour produced similarly in a control containing an amount of arsenic equivalent to the limit given in the individual monograph. The test was performed with arsenic test apparatus.

**Procedure:** Standard was prepared by pipetting 3mL of standard arsenic solution in to a generator flask and diluted to 35mL with water. Test was prepared as per individual monograph. 20mL of sulfuric acid, 2mL of potassium iodide, 0.5mL of strong acid stannous chloride and 1mL of isopropyl alcohol were mixed with standard and test solutions separately. Allowed to stand at room temperature for 30min. Then 3mL of silver diethyl dithiocarbonate and 3g of granular zinc were added to the mixture and allowed to evolution of hydrogen and the colour development to proceed at room temperature for 45min. Any red colour produced by the test preparation does not exceed that produced by the standard preparation.

**Sterility test**

The sterility test was carried out as per Indian pharmacopoeia. Sterility test was intended for detecting the presence of viable forms of bacteria, fungi and yeasts in the sample. The test was performed based on the principle that if bacteria or fungi placed in a medium which provides nutritive material and water and kept at room temperature, the organism will grow and their presence can be indicated by turbidity in the clear medium.

A simple direct inoculation method was used to carry out sterility test. In this method the specified quantity of sample was introduced under aseptic condition into a
test tube of culture medium. Then it was incubated at 25°C and 45%RH for 14 days and the growth of microorganisms in the medium was observed. The culture medium was examined during and at the end of incubation period to find out any microbial growth.

**Determination of Bulk density and tap density**

Apparent bulk density (ρ_b) was determined by pouring the material into a graduated cylinder. The bulk volume (V_b) and weight of the powder (M) was determined. The bulk density was calculated using the formula,

\[ \rho_b = \frac{M}{V_b} \]

The measuring cylinder containing a known mass of powder was tapped for a fixed time. The minimum volume (V_t) occupied in the cylinder and the weight (M) of the blend was measured. The tapped density (ρ_t) was calculated using the following formula,

\[ \rho_t = \frac{M}{V_t} \]

**Compressibility Index**

The simplest way for measurement of free flow of powder is compressibility, an indication of the ease with which a material can be induced to flow is given by compressibility index (I) which is calculated as

\[ I = \left( \rho_b - \rho_t / \rho_b \right) \times 100 \]

\( \rho_b \) = tapped density  
\( \rho_t \) = initial bulk density  
The value below 15% indicates a powder which usually give rise to good flow characteristics whereas above 25% indicate poor flowability.

**Stability and Storage Conditions**

Gum Karaya powder is a stable material at room temperature, although it is hygroscopic after drying.

**RESULT AND DISCUSSION**

**Deacetylation of Gum Karaya**

To avoid long heating times, Gum Karaya was prepared by deacetylation in 45% sodium hydroxide solution using microwave radiation technology. Microwave heating, as an alternative to conventional heating techniques, has been proved more rapid and efficient for chemical reactions. To speed up the process, the Gum Karaya was steeped in 45% sodium hydroxide for 24 h at room temperature before subjecting to microwave radiation. The degree of deacetylation for poorly soluble Gum Karaya was determined by potentiometric titration method. DDA% was determined at various time intervals of 2, 4, 6, 8, 10, 12, 14, 16, 18 and 20 min have shown 12, 18, 24, 32, 48, 52, 64, 77, 83 and 92% respectively. Effect of time on the DDA% under microwave heating method for Gum Karaya found to be increasing with time. The deacetylation percentage above 60 was obtained after 12 min in microwave heating. 64% of DDA was obtained at 14 min, at this stage the Gum Karaya is having all desired properties required for developing FDDS, which is confirmed by determining density, swelling index, viscosity, solubility. The results obtained for these parameters are given in Table 1. The DDA% with time is shown in Figure 1.

**FTIR analysis**

IR Spectra of Gum Karaya is shown in Figure 2. A single band was observed in spectrum at 3441.56 cm\(^{-1}\) which is commonly assigned to the stretching of the CO group hydrogen bonded to amide group of the neighboring intra-sheet chain. The strong band at 1430 cm\(^{-1}\) is seen in the spectrum while a distinct band at 1416 cm\(^{-1}\) occurs in the spectrum of Gum Karaya. The band due NH stretching at 3264 cm\(^{-1}\) and 3107 cm\(^{-1}\) can be seen clearly in the spectrum assigned these bands to CO-NH intermolecular bonding and H bonded NH group. OH-out-of plane bending at 703 cm\(^{-1}\) and NH-out-of plane bending at 750 cm\(^{-1}\) can be observed in the spectrum. This is due to a relatively low crystalline and loosely ordered structure showing weaker inter- and intramolecular hydrogen bonding in Gum Karaya.

**Differential Scanning calorimetric analysis (DSC)**

A DSC curve of Gum Karaya was shown in Figure 3. The curve shows that weight loss occurs in two stages. The first stage starts around 91.6°C and the second stage starts around 316°C. The first stage is assigned to the loss of water because polysaccharides usually have a strong affinity for water and therefore may be easily hydrated.

The second one corresponds to the thermal decomposition of Gum Karaya. The decomposition temperature of Gum Karaya was found to be 316°C. This result indicates that Gum Karaya exists as a stable structure toward thermal decomposition.

**X-Ray powder diffractometry of Gum Karaya**

XRD analysis was applied to detect the crystallinity of the modified Gum Karaya. The XRD pattern of Gum Karaya, show five sharp crystalline reflections at 10.4°, 20.8°, 30.2°, 34.1° and 41.2°.

X-ray diffraction exposed the characteristics of Gum Karaya more clearly due to the different arrangements adopted by these polymorphs. Figure 4 shows XRD patterns of Gum Karaya. The XRD profile of Gum Karaya exhibits well resolved and intense peaks while a broad diffuse scattering and less intense peaks were found for Gum Karaya at 10.4° and 20.8°, crystallinity percent was found to be 62.4 and 44.6 respectively. This indicates that Gum Karaya is a partially crystalline polymorph because of its antiparallel compact structure.

The crystallinity percentage was calculated by
dividing the area of the crystalline peaks by the total area under the curve.

**Scanning electron microscopy (SEM)**

Figure 5 shows SEM photographs of powder Gum Karaya. A very uniform with a lamellar organization and dense structure was observed clearly for Gum Karaya.

**13C NMR analysis**

Chemical composition of Gum Karaya was obtained by 13C NMR spectrum using 2% deuterated acetic acid in D2O as solvent. Figure 6 shows the 13C NMR spectrum (600 MHz) of Gum Karaya. The absence of methyl proton resonance from protein between 1.0 and 1.5 in 13C NMR spectrum gives a good indication to the purity of Gum Karaya sample.

**Mass spectroscopy**

Figure 9 shows the mass spectrum of Gum Karaya was measured by MALDI, which enables analysis of molecules approximately up to 500KDa. Molecular weight of the Gum Karaya was calculated by interpreting the mass spectrum was found to be 207.44.

**Standardization of Gum Karaya powder**

Gum Karaya powder was subjected for standardization using many parameters such as appearance, odor, taste, solubility, viscosity, particle size distribution, incompatibility, loss on trying, ash content, glass transition temperature, limit tests, pH, sterility test, density, and degree of deacetylation.

The appearance of Gum Karaya was White to buff fine powder it does not have any characteristic odour and taste. The pH was found to be 4.9, which indicates more compatible with stomach environment. Density was low as 0.95g/mL which may be helpful in the development of floating system.

Gum Karaya was poorly soluble in water; practically insoluble in ethanol (95%), other organic solvents, and neutral or alkali solutions at pH above approximately 6.5. Gum Karaya dissolved readily in dilute and concentrated solutions of acetic acid and sparingly soluble in hydrochloric acid.

The viscosity of Gum Karaya (1% w/v) was performed by using Ostwald viscometer. Viscosity was found to be 464 ± 6.64cps which confirms Gum Karaya is an excellent viscosity enhancing agent in an acidic environment.

Gum Karaya was complied with all other evaluated parameters such as viscosity, limit test for chloride heavy metals and arsenic, loss on trying and ash content were found to be 464 ± 6.62 cps, <25ppm, <20ppm, <10ppm, 8% and 0.8% respectively.

The apparent particle, tap and bulk densities of Gum Karaya were shown within the limit. Bulk and tap density are dependent on particle density which indicated good flowability of the powder and hence while using both these values the Carr index was calculated. The lower Carr index indicates the better flowability of the powder. These was no physical changes observed during storage condition which indicates Gum Karaya is more stable at room temperature

The sterility test indicated no evidence of growth hence the Gum Karaya sample being examined was passes the test for sterility. Results obtained for all standardization parameters are given in Table 2.

### Table 1. Degree of deacetylation of Gum Karaya at various time interval and results obtained for the evaluation of preliminary parameters

<table>
<thead>
<tr>
<th>Time in min</th>
<th>DDA %</th>
<th>Density g/cm3</th>
<th>Swelling index %</th>
<th>Viscosity cps</th>
<th>Solubility in 0.1N HCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>21</td>
<td>4.26 ± 1.25</td>
<td>22.74 ± 2.58</td>
<td>124 ± 4.65</td>
<td>Insoluble</td>
</tr>
<tr>
<td>8</td>
<td>33</td>
<td>3.68 ± 2.15</td>
<td>28.64 ± 4.62</td>
<td>259 ± 5.54</td>
<td>Insoluble</td>
</tr>
<tr>
<td>10</td>
<td>45</td>
<td>3.25 ± 0.58</td>
<td>32.96 ± 1.25</td>
<td>354 ± 6.65</td>
<td>Insoluble</td>
</tr>
<tr>
<td>12</td>
<td>56</td>
<td>2.68 ± 0.97</td>
<td>45.15 ± 3.62</td>
<td>346 ± 4.65</td>
<td>Insoluble</td>
</tr>
<tr>
<td>14</td>
<td>65</td>
<td>1.45 ± 0.64</td>
<td>74.52 ± 2.56</td>
<td>385 ± 4.23</td>
<td>Slightly soluble</td>
</tr>
<tr>
<td>16</td>
<td>72</td>
<td>0.95 ± 0.65</td>
<td>97.58 ± 0.54</td>
<td>462 ± 7.56</td>
<td>Poorly soluble</td>
</tr>
<tr>
<td>18</td>
<td>79</td>
<td>0.96 ± 2.56</td>
<td>95.94 ± 1.64</td>
<td>476 ± 8.62</td>
<td>Sparingly soluble</td>
</tr>
<tr>
<td>20</td>
<td>88</td>
<td>0.92 ± 1.85</td>
<td>82.68 ± 6.67</td>
<td>514 ± 3.45</td>
<td>Freely soluble</td>
</tr>
</tbody>
</table>

### Table 2. Results of standardization of Gum Karaya

<table>
<thead>
<tr>
<th>Parameters evaluated</th>
<th>Reference standard</th>
<th>Results obtained</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>White to buff fine powder</td>
<td>White to buff fine powder</td>
</tr>
<tr>
<td>Odour and Taste</td>
<td>None</td>
<td>No characteristic odour and taste observed</td>
</tr>
<tr>
<td>Solubility</td>
<td>Insoluble to freely soluble depends on %DDA</td>
<td>Poorly soluble at 64 % DDA</td>
</tr>
<tr>
<td>Viscosity</td>
<td>Less than 100cps</td>
<td>655 ± 3.62 cps</td>
</tr>
<tr>
<td>Particle size distribution (µm)</td>
<td>40-180 mesh</td>
<td>40-80 mesh</td>
</tr>
<tr>
<td>Incompatibility</td>
<td>Nil</td>
<td>Nil</td>
</tr>
<tr>
<td>Loss on trying</td>
<td>Not more than 20%</td>
<td>12%</td>
</tr>
<tr>
<td>---------------------</td>
<td>-------------------</td>
<td>-----</td>
</tr>
<tr>
<td>Ash content</td>
<td>&lt;1.5%</td>
<td>0.9%</td>
</tr>
<tr>
<td>Glass transition temperature</td>
<td>NA</td>
<td>93.2°C</td>
</tr>
<tr>
<td>Limit test for chloride</td>
<td>&lt;25ppm</td>
<td>&lt;25ppm</td>
</tr>
<tr>
<td>Limit test for heavy metals</td>
<td>&lt;20ppm</td>
<td>&lt;20ppm</td>
</tr>
<tr>
<td>Limit test for Arsenic</td>
<td>&lt;10ppm</td>
<td>&lt;10ppm</td>
</tr>
<tr>
<td>Acidity/Alkalinity (pH)</td>
<td>4-5</td>
<td>4.9</td>
</tr>
<tr>
<td>Sterility test</td>
<td>Absence of viable content</td>
<td>Absence of viable content</td>
</tr>
<tr>
<td>Molecular weight</td>
<td>9,500,000</td>
<td>&gt;8,500,000</td>
</tr>
<tr>
<td>Degree of deacetylation %</td>
<td>50-120%</td>
<td>64%</td>
</tr>
<tr>
<td>Bulk density (g/cm³)</td>
<td>0.366 ± 0.012 (g/cm³)</td>
<td>0.463 ± 0.009 (g/cm³)</td>
</tr>
<tr>
<td>Tap density (g/cm³)</td>
<td>0.334 ± 0.025 (g/cm³)</td>
<td>0.395 ± 0.018 (g/cm³)</td>
</tr>
<tr>
<td>Compressibility Index (%)</td>
<td>24.70 ± 1.52 (%)</td>
<td>16.36 ± 0.97 (%)</td>
</tr>
<tr>
<td>Stability</td>
<td>Stable at room temperature</td>
<td>Stable at room temperature</td>
</tr>
<tr>
<td>Storage condition</td>
<td>Gum Karaya should be stored in a tightly closed container in a cool, dry place.</td>
<td>Gum Karaya should be stored in a tightly closed container in a cool, dry place.</td>
</tr>
</tbody>
</table>

**Figure 1. Degree of deacetylation**

![Figure 1](image1.png)

**Figure 2. IR Spectra of Gum Karaya**

![Figure 2](image2.png)

**Figure 3. DSC Thermogram of Gum Karaya**

![Figure 3](image3.png)

**Figure 4. XRD patterns of Gum Karaya**

![Figure 4](image4.png)
Figure 5. SEM micrographs of Gum Karaya
CONCLUSION

Commercially available Gum Karaya was purchased from local market. In FTIR spectra, the amide band is split for Gum Karaya. The XRD, SEM results indicate that Gum Karayais a crystalline polymorph because of its parallel structure.

Gum Karaya was hydrolyzed using microwave heating method. Gum Karaya produced from microwave heating reduced the time of deacetylation within few minutes (~14 min) to reach to the DDA% of 64%.

Gum Karaya is an abundant natural polymer, obtained from sterculia gum, is the dried exudate of Sterculia Urens, a tree native to India. The physical and chemical properties of Gum Karaya, such as inter-and intramolecular hydrogen bonding and the cationic charge in acidic medium, makes this polymer attractive for the development of floating drug delivery system. Being a natural polymer and having all desired properties, Gum Karaya can be used as a good candidate for oral floating drug delivery. Also, because of its favorable biological properties such as non-toxicity, biocompatibility, and biodegradability, Gum Karaya is a promising candidate for the enhancement of absorption of drugs through flotation using floating drug delivery system. As a result of the physical, chemical, and biological properties, Gum Karaya can is used in different formulations for drug delivery in the GI tract.

Based on the results obtained from characterization and standardization it can be concluded that modified Gum Karaya has all desired properties that are required to develop FDDS. These properties allow for the application of Gum Karaya to be used for proposed floating drug delivery system.

REFERENCES


Anderson DMW, Howlett JF and McNab CGA. Amino acid composition of proteinaceous component of gum karaya (Sterculia spp). Food Additives and Contaminants. 1985; 2: 159-64.


Food Chemicals Codex, 4th edn, 1996.


Indian Pharmacopoeia. The Controller of Publication. Delhi, 2, 1996, 735.


