Chapter 3

Biomaterials

3.1 Introduction

Biomaterials are defined as materials of synthetic as well as of natural origin in contact with tissue, blood, and biological fluids, and intended for use for prosthetic, diagnostic, therapeutic, and storage applications without adversely affecting the living organism and its components [1]. It is also defined as a nonviable material used in a medical device, intended to interact with biological systems [2]. Biomaterials find application in many fields. The most common application of biomaterials include artificial hip joint, kidney dialysis machine, sutures, bone plates, cardiac pacemaker, intraocular lens, augmentation mammoplasty, chin augmentation, probes, catheters etc [3].

A biofilm is a three-dimensional (3-D) community of microorganisms immobilized at a substratum and frequently embedded in an organic polymer matrix of microbial origin [4]. The chief constituents of natural biofilms typically are many species of bacteria, fungi, algae, protozoa, debris, and corrosion products, with the extra-cellular polymeric substance (EPS) matrix largely responsible for structural integrity [5]. Synthetic films that possess either or any of the characteristic features such as biodegradability, biocompatibility, bioresorption etc. can be termed as biomaterial films. Biomaterial films found significant applications in complex surgeries involving skin transplantation, plastic surgery and they even found use as a medium for drug delivery as some of these films were edible and non-toxic. They were used as transdermal patches and as capsule coatings. Gelatin, Dextran, Chondroin sulphate, Calcium pectinate, Pectin and Chitosan were some polysaccharides which were used in this context [6]. All these materials were found
suitable because of their biodegradability and easy availability. If the film is soluble in water, it can be more useful in pharmaceutical applications. The success of a biomaterial inside a body depends on both the property of the material, design, biocompatibility and external factors such as patient’s health and activities.

The biomaterial films can also be used as phantom materials in an electromagnetic simulation environment [7-10]. If their dielectric properties are comparable to the dielectric properties of body parts, those body parts can be represented by phantom materials with a matched dielectric profile in a microwave experimental setup. Biological films can be of animal or plant origin. Some of these films possess medicinal properties. It is by the virtue of the plant from which it is procured. The characteristic features of the films include its mechanical, structural, optical and electrical properties. The mechanical properties include its elasticity, stress withstanding capability etc. Structural properties like heterogeneity and porosity are also important. The optical properties include film’s transparency and refractive index. And the electrical properties which are of significant important to us are its electrical conductivity, skin depth, surface resistance, heating coefficient, dielectric constant, loss tangent and attenuation constant. The electrical conductivity tells us about the amount of electric current that can be passed through the film without destroying its properties. Though Skin depth is a term normally associated with conductors, it can be used to characterize the behavior of a film which is subjected to microwaves. The depth of penetration of the signal gives us information such as the capability of the material to reflect a wave and its wave absorption characteristics.

Material characterization using microwaves is a solid field where microwave properties of materials are studied. By studying the dielectric properties [11, 12], several biological effects can be studied. For instance, microwaves are widely used in the field of imaging for finding malicious tissues and tumours. These techniques use tissue characterization based on complex permittivity. The dielectric properties of the malignant tissue will be different from that of the normal tissue. Several advancements have been made on the dielectric property study of materials. The
properties such as permittivity and loss factor are significant in determining material properties. Microwave absorption characteristics gives the amount of microwave power absorbed by the film at different microwave frequencies and will be helpful in the study of effect of non-ionizing radiation [13] on Chitosan and Arrowroot.

### 3.2 Arrowroot

![Arrowroot Plant](image)

Ararrowroot is from the family Marantaceae [14]. The Arrowroot plant grows up to a height of 80 cm and harvested 9 to 11 months after planting. It has tuberous roots which stores starch. Arrowroot powder is valued chiefly for its thickening properties. It has neutral taste and it thickens at a lower temperature than starch of corn. Arrowroot (ARROW) is most commonly used as a chief ingredient in baby food, as substitute for talcum in baby powder and body powder. Because of its
medicinal properties [15], it is considered to be an effective antidote to vegetable poisons.

Arrowroot was first introduced to Europeans by the Arawak tribe in the Caribbean. They used it to draw poison out of the wounds inflicted from poisoned arrows. The Indians used Arrowroot as their chief food and called it aru-aru (meal of meals). An interesting pilot study on the use of arrowroot as treatment for diarrhea in irritable bowel syndrome patients by C. Cooke et al [15] shows that Arrowroot is very effective in treatment of diarrhea and plays a significant role in reducing daytime bowel frequency. Its calorie content is low. So it is a popular food among calorie-conscious dieters.

![Figure 3.2 Structures of Starch and Cellulose](image)

Arrowroot is basically starch. Starch and cellulose are two terms which are often mistaken for each other. Cellulose is a linear polysaccharide polymer with many glucose monosaccharide units. The acetal linkage is beta which makes it different from starch (figure 3.2). The beta-acetal linkage cannot be broken down by any of the enzymes in animals. Animals such as cows, goats, horses, termite etc has symbiotic bacteria in their intestinal tract. These bacteria produce necessary enzymes to digest cellulose in the gastro-intestinal tract. Hydrolysis of cellulose takes place in intestinal tract. No vertebrate including humans can digest cellulose directly. So humans are not able to digest fibrous food. The indigestible cellulose is
the fiber which acts in the smooth working of intestinal tract. But Starch contains alpha-acetal linkage which can be easily broken down by enzymes within the body. Arrowroot hence is an edible food. It is also used for making cookies, in talcum powder and in biscuits.

### 3.2.1 Preparation

(i) Arrowroot powder

Arrowroot powder is prepared from the root of Arrowroot plant. Arrowroot is a tuberous plant. So the chief food storage is in the roots. The roots are soaked in hot water and the fibrous covering is peeled off. It is then cut into small pieces and mashed to obtain pulp. The pulp is macerated in water to break down tough cells surrounding starch. It is then washed thoroughly with water and filtered. The washing is done to separate starch from fibrous material. The process is repeated until pure arrowroot powder is obtained. The separated starch is dried and later powdered to obtain Arrowroot powder. Around 20% of the original root is starch. By careful processing, up to 17-18% of the starch can be extracted. There are two kinds of powders obtained from two variants of the same plant. One has pure white color and the other has a slight yellowish tint. But both these powders are very
identical in all other properties. Both are commercially available in the market and are cheap.

(ii) Arrowroot film

Arrowroot film is prepared from Arrowroot powder in a simple process. The powder (10g) is dissolved in water (100ml) and is heated in a glass tumbler. Around 80°C, it will turn into a transparent gel. This particular soluble: solvent ratio was determined by trial and error. If the amount of powder is high, then it results in excess amount of particles. And the particles tend to coagulate and it is not possible to get a clean gel. If amount of powder is low, the gel wouldn’t get sufficient viscosity and the films prepared from the gel won’t have any mechanical strength at all. It cannot help in film formation. If we use a conventional heater to heat the solution, then it is necessary to stir the solution so that no coagulation of particles occurs. This method is more susceptible to coagulation problem as the water near to bottom of the vessel gets heated up first rather than the water near the top surface. It is possible to get a clean gel, but is difficult to achieve so. As the method suffers from this problem, we had to find other heat sources which can
provide uniform heat to all parts of the solution. As a result, we found microwave heating as the best method for the purpose. In microwave heating, the solution gets a uniform heat from all directions. We were able to obtain a clean gel easily when microwave heating was used. The prepared gel is casted on thick plastic sheets. It is not necessary to use plastic sheet, however, the surface used should be smooth so that the prepared film doesn’t get adhered to the surface.

The cast was dried using three methods - conventional shadow drying, microwave drying and hot air drying. Among these methods, shadow drying was the best method to obtain a uniform film. Microwave and hot air drying can also produce good films, but if the temperature given is even slightly high, it will result in deformed film. And the amount of temperature given depends up on the viscosity and thickness of the cast which varies. Hence we have to reduce temperature and increase drying time. But it will eat up electricity and is not as cheap as shadow drying. But the microwave and hot air drying methods has an advantage. They drastically reduce the drying time when compared to shadow drying. The choice of drying method requires a tradeoff between time and economy.

3.2.2 Dielectric Characterization

The dielectric characterization of Arrowroot film was done for S-band of microwave frequencies. Cavity perturbation method was used for the purpose. The properties such as Dielectric constant, Loss factor, Conductivity, Skin depth and Heating coefficient were measured. Microwave absorption study was also performed. These properties are very significant in microwave phantom applications of the material. Arrowroot is an edible food. Also it is biodegradable which makes it suitable for implantation purposes. It is already used in paper coatings and biodegradable plastics. The ability to form thin, transparent and tough film enables it to find applications in many other fields also.
(i) Permittivity and Loss factor

The Permittivity and Loss factor variations of Arrowroot film at various frequencies are shown in Figure 3.5. It can be seen that the dielectric constant of Arrowroot film varies between 5.25 and 6.5. Arrowroot is having low dielectric constant which is closer to the dielectric constants of certain body parts such as bone marrow, collagen, breast fat, etc. Thus Arrowroot can be considered as a phantom material for in vitro studies and can be used for implantation purposes after performing the biocompatibility studies.

![Figure 3.5 Variation of Permittivity and Loss factor of Arrowroot film at microwave frequencies](image)

Loss factor of a material is associated with the dissipation or power loss within the dielectric. The conductivity of the material is a direct function of loss factor and for practical purposes both the conductivity and loss factor are considered indistinguishable [16]. Figure 3.5 shows the variation of dielectric loss. When a field is applied to a dielectric, it tends to polarize the material. The degree of polarizability depends on the relaxation time of the molecules of the medium [11]. For Arrowroot film, the molecules are tightly packed so that the molecules achieve equilibrium with the applied field rapidly. As the relaxation phenomenon is
quick, it has low dielectric loss. Hence the film has a comparatively small loss factor. Thus microwave power is less dissipated in Arrowroot film.

(ii) Conductivity, Skin depth and Heating Coefficient

The dielectric parameters other than permittivity and loss factor are also important for determining material characteristics. The measured properties such as conductivity, skin depth, and heating coefficient are shown in Table 3.1. The conductivity of the material is responsible for the loss within the material. The skin depth determines the depth of penetration of electromagnetic wave within a substance. The skin depth of the material is inversely proportional to the conductivity of the material. The variation of microwave heating coefficient is also shown. The heating coefficient increases with increase in frequency. This means more energy is dissipated via dielectric heating mechanism at low frequencies.

Table 3.1. Dielectric parameters of Arrowroot film

<table>
<thead>
<tr>
<th>Frequency (MHz)</th>
<th>Conductivity (Sm⁻¹)</th>
<th>Skin depth (m)</th>
<th>Heating coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>2439</td>
<td>0.3317</td>
<td>0.0176</td>
<td>0.4092</td>
</tr>
<tr>
<td>2685</td>
<td>0.2729</td>
<td>0.0185</td>
<td>0.5474</td>
</tr>
<tr>
<td>2971</td>
<td>0.2811</td>
<td>0.0174</td>
<td>0.5880</td>
</tr>
</tbody>
</table>

3.2.3 Applications

The dielectric properties of the film sample reveals that it has low dielectric constant which is a typical characteristic of body tissues collagen, bone marrow and human abdominal wall fat. Table 3.2 shows the dielectric chart of these tissues.

The newly developed film may be used as phantom material representing these tissues. For in vitro studies involving these tissues, the film could be useful as an alternative to expensive counterparts. Apart from its use as phantom material in
microwave imaging or SAR studies, it can be of more use in medical field. The remarkable properties of the film such as its transparency, medicinal properties and edibility can be made useful if it is used as capsule material for drugs. We suggest the use of a drug delivery system entirely based on Arrowroot film. The Arrowroot film could be easily molded into any shape and is also cost effective at the same time. Drug delivering systems presently use gelatin, dextran, chondroitin sulphate, calcium pectinate, pectin and chitosan as carriers for oral delivery of drugs. These materials are also used for bimodal delivery of drugs [6]. Arrowroot film is insoluble in water, but it swells when dipped in water for a while. The bimodal delivery can be achieved by incorporating hydrophilic degradable polysaccharides in Arrowroot film. The film can ensure sustained drug delivery through this method. It could also be used as transdermal patches which embed drugs in it for sustained delivery over a period of time. Another proposed application is eco-friendly band-aids. The soaking property of the film can be made useful for embedding hydrophilic drugs in it. Low cost, easy preparation and most importantly biocompatible features makes this material a better choice.

Table 3.2 Dielectric properties of certain body tissues

<table>
<thead>
<tr>
<th>Body part</th>
<th>Dielectric constant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Collagen</td>
<td>5.5-6.5</td>
</tr>
<tr>
<td>Bone marrow</td>
<td>4.2-5.8</td>
</tr>
<tr>
<td>Human abdominal wall fat</td>
<td>4.92</td>
</tr>
</tbody>
</table>


3.2.4 Advantages and disadvantages

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>✓ <em>Easy availability</em></td>
<td>▪ <em>Low mechanical strength</em></td>
</tr>
<tr>
<td>✓ <em>Medicinal properties</em></td>
<td>▪ <em>Varying thickness in different batches</em></td>
</tr>
<tr>
<td>✓ <em>Edibility</em></td>
<td></td>
</tr>
<tr>
<td>✓ <em>Low cost</em></td>
<td></td>
</tr>
<tr>
<td>✓ <em>Easy preparation</em></td>
<td></td>
</tr>
<tr>
<td>✓ <em>Transparency</em></td>
<td></td>
</tr>
<tr>
<td>✓ <em>Biodegradability</em></td>
<td></td>
</tr>
<tr>
<td>✓ <em>Eco-friendly</em></td>
<td></td>
</tr>
</tbody>
</table>

3.2.5 Conclusions

The Arrowroot film has been developed and its dielectric properties were analyzed. The prepared films are transparent and can be made into any thickness. They are moldable, edible and biodegradable. The material could find potential applications such as phantom material in microwave imaging, as capsule material in pharmaceutical applications, as transdermal patch material and as eco-friendly Band-Aids. The dissolution properties of the Arrowroot film and its feasibility of use as a drug coating material can only be verified with the use of a dissolution test apparatus and the analysis of data using High Performance Liquid Chromatography (HPLC) system.

3.3 Chitosan

Chitosan (poly [β-1-4) D-glucosamine) is the second abundant polysaccharide and a cationic polyelectrolyte present in nature [17]. It is one of the most versatile biopolymers known to the science community. Its applications are numerous. It is used in applications such as natural seed treatment, ecologically friendly bio
pesticides, plant growth enhancement, water filtration, self-healing coatings, bandages, clarification, chromatography, paper and textiles, photography, transdermal drug delivery etc. [18-22]. It is also used as a soluble dietary fiber owing to its edibility. The property of long chain molecules of dissolved Chitosan to wrap the solid particles suspended in liquids and to bring them together and agglomerate makes it suitable as a coagulant acid. Chitosan facilitates better collagen growth in implants. Its amino group has a pKa value of 6.5 which results in protonation in acidic to neutral solution. This makes Chitosan water soluble and enhances its bio adhesive properties.

Another property of Chitosan is its ability to form gel in diluted acidic solvents. The Chitosan chain contains free amine group which will undergo protonation on treatment with diluted aqueous solution of acids resulting in formation of corresponding Chitosan salt in the solution. Chitosan also has been found to have antimicrobial, hypocholesterolemic, and wound healing properties [23-25]. Chitosan is characterized in many ways such as quality, purity, molecular weight, viscosity, degree of deacetylation and physical forms. The deacetylation process is done by treatment of Chitin with an alkaline solution. Chitin when deacetylated more than 75% gives Chitosan. The degree of deacetylation can be determined by any of the tests such as ninhydrin test, near-infrared spectroscopy, infrared spectroscopy, linear potentiometric titration, nuclear magnetic resonance spectroscopy, hydrogen bromide titrimetry, and first derivative UV-spectrophotometry [26]. Chitosan molecules are positively charged. So it readily binds to negatively charged surfaces. This property of Chitosan is made useful in drug delivery applications. It could carry polar drugs across epithelial surfaces. An exhaustive study on the dielectric behaviour of Chitosan at microwave frequencies has been made and its suitability as biopolymer is analyzed.

### 3.3.1 Preparation

Chitosan is a biopolymer which is obtained from the shells of sea crustaceans like shrimp and crab [27]. These shells are first oven dried to reduce
the moisture content. A final moisture content of 10-12% is preferred. The shells are crushed and boiled for an hour in 4% NaOH solution to dissolve proteins and sugars. It is again crushed to fine pieces. The sample is then treated/soaked with 1% solution of HCl for 24 hours to remove calcium carbonate and other minerals. In order to decompose the albumin which is present in the demineralized sample to water soluble amino acids, the sample is treated with 2% NaOH solution for an hour. Thus we obtain Chitin. The sample is washed in deionized water and cleaned. The Chitin on deacetylation by treatment with NaOH gives Chitosan. For deacetylation, 50% NaOH solution is added to the sample and the sample is boiled at 100°C for two hours. Then it is allowed to cool to room temperature. The sample is then washed thoroughly in 50% NaOH and is filtered to obtain the residue, which is Chitosan. It is then kept in an oven and is heated at 120°C for 24 hours. The Chitosan powder thus obtained is kept in air tight containers.

Chitosan is soluble in weak acids due to the low value of pKa. Chitosan powder (1gm) was dissolved in 50 ml dilute acetic acid (14%). The solution was stirred for 4 hours until Chitosan was completely dissolved. The thick gel obtained was then taken in small capillary tubes of approximately 0.8 mm diameter for
cavity perturbation measurements. The Chitosan gel was casted and dried in shadow to obtain thin films of varying thickness. The film was cut into thin strips of 3-5 mm width and 50mm length. The thickness of the films ranged from 0.2mm to 0.8mm. The dielectric characterization of the film was done for S-band of microwave frequencies.

### 3.3.2 Dielectric Characterization

The dielectric characterization of Chitosan gel and film at microwave frequencies was done using cavity perturbation method. The dielectric properties were evaluated for S-band of microwave frequencies. The aim of the study was to find whether the dielectric parameters resemble the dielectric parameters of any body tissues. If the dielectric parameters are comparable, then these could be used as an alternative to the particular body part in microwave tomography and SAR applications.

(i) Permittivity and Loss factor

![Figure 3.7 Variation of complex permittivity of Chitosan film and gel with frequency.](image)

The permittivity and Loss factor variation of Chitosan film and gel are as shown in figure 3.7. It is observed that Chitosan gel exhibits higher permittivity
and loss factor values as compared to Chitosan film owing to the increased water content. The permittivity and loss factor values of Chitosan film don’t show prominent variation for frequency range 2-3 GHz. The large variation in permittivity of the gel for this range suggests that the gel is highly polarized by the incident wave. The dielectric constant of Chitosan film is low because of its lower polarizability. The polarizability of the gel is due to the presence of water which is a polar material. Thus it can be concluded that the permittivity can be altered to any desirable value by varying the water content of the gel (by varying the pH). The Chitosan film shows very low value for permittivity and loss factor. This means that the film is less polarized and dielectric relaxation for the film is very short at microwave frequencies.

(ii) Conductivity, Skin depth and Loss Tangent

The microwave conductivity of a material depends on the dielectric loss factor. It in turn depends on the polarizability of the material and the relaxation time. As Chitosan film is a non-polar material, it has a very low loss factor. And

Table 3.3. Dielectric parameters of Chitosan film and gel

<table>
<thead>
<tr>
<th>Form of Chitosan</th>
<th>Frequency (GHz)</th>
<th>Conductivity $\sigma$ (S/m)</th>
<th>Skin depth $\delta_s$ (m)</th>
<th>Tan $\delta$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Film</td>
<td>2.430</td>
<td>$3.7 \times 10^{-2}$</td>
<td>$5.30 \times 10^{-2}$</td>
<td>$4.43 \times 10^{-2}$</td>
</tr>
<tr>
<td></td>
<td>2.680</td>
<td>$6.73 \times 10^{-2}$</td>
<td>$3.75 \times 10^{-2}$</td>
<td>$7.17 \times 10^{-2}$</td>
</tr>
<tr>
<td></td>
<td>2.970</td>
<td>$1.11 \times 10^{-1}$</td>
<td>$2.77 \times 10^{-2}$</td>
<td>$1.05 \times 10^{-1}$</td>
</tr>
<tr>
<td>Gel</td>
<td>2.430</td>
<td>$1.06 \times 10^{-1}$</td>
<td>$3.13 \times 10^{-2}$</td>
<td>$4.05 \times 10^{-2}$</td>
</tr>
<tr>
<td></td>
<td>2.680</td>
<td>$2.67 \times 10^{-1}$</td>
<td>$1.88 \times 10^{-2}$</td>
<td>$6.06 \times 10^{-2}$</td>
</tr>
<tr>
<td></td>
<td>2.970</td>
<td>$5.43 \times 10^{-1}$</td>
<td>$1.25 \times 10^{-2}$</td>
<td>$1.09 \times 10^{-1}$</td>
</tr>
</tbody>
</table>

hence the microwave conductivity is very low for Chitosan film as compared to Chitosan gel. This is evident from Table 3.3. The Skin depth measures the depth of penetration of the microwave signal into the sample. For a highly conducting
material, much of the wave gets reflected back without penetrating deeply into the sample.

It can be observed from Table 3.3 that the Skin depth becomes low as microwave conductivity increases. As Chitosan film is homogeneous, interfacial polar charges will be very low in number. It will result in a low dielectric constant value. \( \tan \delta \) is the loss tangent of the film and gel. It is observed that the Chitosan gel is lossier due to the polar nature of the sample.

### 3.3.3 Applications

The film and gel forms of Chitosan can find significant application as phantom materials in microwave imaging. They have dielectric properties similar to that of certain body parts. The dielectric property of the gel depends on the water content in it. Thus by varying the water content, phantoms for various body parts can be prepared. A comparison of the dielectric properties of Chitosan and the corresponding body parts is shown in Table 3.4.

<table>
<thead>
<tr>
<th>Human Organ</th>
<th>Dielectric constant</th>
<th>Equivalent Phantoms</th>
</tr>
</thead>
<tbody>
<tr>
<td>Collagen/Fat tissue</td>
<td>5.5 - 6.5</td>
<td>Chitosan Film</td>
</tr>
<tr>
<td>Brain/Breast tissue</td>
<td>26 - 29</td>
<td>Chitosan Gel</td>
</tr>
</tbody>
</table>

The dielectric constant of Collagen/fat tissue falls in the range 5.5-6.5 which corresponds to the dielectric constant of prepared Chitosan film. The equivalent phantom for Brain/Breast tissue can be implemented with Chitosan gel. The study shows that the phantoms corresponding to other body parts can also be developed from Chitosan gel with various pH values. The Chitosan film and gel both are very easy to prepare, low cost and biodegradable. Thus they are ideal candidates for use as phantom materials in microwave imaging applications.
3.3.4 Conclusions

The dielectric characterization studies of Chitosan gel and film were performed. Both the gel and film are transparent and biodegradable. It was found that both of them have dielectric properties similar to that of certain body parts which enables them to be used as phantom materials in microwave imaging applications.

3.4 Arrowroot-Chitosan film

The quest for thin, tough and transparent biocompatible films led to the development of new materials in medical field [28-30]. These films found use as contact lens materials, encapsulation of drugs, as transdermal patches etc. Biocompatibility, ease of preparation, low cost and availability are the important constraints which are to be taken into consideration while preparation of films. The mechanical properties of the film determine the strength of the film. A work on the influence of thickness on mechanical properties for starch films was done by A. Jansson and F. Thuvander [31]. They tested the films in tension and the films were characterized in terms of strength, stiffness and failure strain. Another important work was done for improving the performance of starch film by the use of cellulose micro fibrils [32]. It has been found that the water sensitivity linearly decreases with the cellulose micro fibril content.

The main drawback of Arrowroot film was its comparatively poor mechanical stability. The film was transparent and biocompatible. But it suffered from poor mechanical stability. To increase the mechanical strength of the film, Chitosan gel was added to Arrowroot gel during preparation. The resultant gel was casted to obtain films of varying thickness. The mechanical, morphological and dielectric properties of the film were studied. Dielectric property studies are performed at frequencies from 20 Hz to microwave ranges. The mechanical properties of the film such as stress and strain are also analyzed. Morphological properties are analyzed with the aid of SEM measurements.
The mechanical, morphological and dielectric properties of the film are analyzed. The mechanical properties are procured from Stress/Strain measurement. The morphological characteristics and porosity of the film are determined from SEM measurements. A simple and sure method to find the biocompatibility of a material is to study the dielectric properties of the material. All biomaterials are prone to microwaves. The dielectric properties of the film at low and high frequencies are analyzed with the aid of Impedance and Network analyzers.

3.4.1 Preparation

Arrowroot powder (10gm) is dissolved in water (100ml) and is heated in a glass tumbler using a microwave oven. Around 80°C, it will turn into a transparent gel. Chitosan powder (1gm) was dissolved in 20 ml glacial acetic acid (99-100%) which was diluted with 2ml of water. The gel formed was added to Arrowroot gel and mixed thoroughly. The mix was casted and dried in shadow to obtain thin films of varying thickness. The film was cut into thin strips of 3-5 mm width and 50mm length. The thickness of the films ranged from 0.15mm to 0.8mm. The dielectric characterization of the film was done for 20Hz-2MHz range as well as at S-band of microwave frequencies.

3.4.2 Characterization

(i) Dielectric properties

The dielectric characterization of the film was performed at frequency ranges from 20Hz -2MHz and 2GHz-3GHz. Measurement at low frequencies were done using Agilent E4980A Impedance Analyzer (20Hz to 2MHz) with the aid of Agilent 16451B dielectric material test fixture. The measurements at microwave frequencies were performed using Agilent 8714 ET vector network analyzer.
(a) *Characterization at low frequency*

Low frequency dielectric property measurements were carried out to find the behavior of film at low frequencies. The properties dielectric constant and loss factor are the most important properties. As shown in figure 3.8, the Arrow-Ch film has low permittivity at small frequencies. After 1 KHz, the films show more or less

![Figure 3.8. Variation of Dielectric constant at low frequencies](image1)

![Figure 3.9. Variation of Loss factor at low frequencies](image2)
the same characteristics. Figure 3.9 shows the variation of loss factor with respect to frequency. It can be seen that after 100 KHz both are showing the same response. The material is lossy at low frequencies.

(b) Characterization at microwave frequencies

The cavity perturbation technique gives the permittivity and loss factor at microwave frequencies as a function of shift in resonant frequency and variation in quality factor of the wave guide cavity. From the permittivity and loss factor we can obtain other important parameters such as conductivity, skin depth, attenuation constant etc. The measured parameters are shown in Table 3.5.

<table>
<thead>
<tr>
<th>Frequency (GHz)</th>
<th>Dielectric constant, $\varepsilon_r$</th>
<th>Loss factor, $\varepsilon_r''$</th>
<th>Conductivity, $\sigma$ (Sm$^2$)</th>
<th>Skin depth, $\delta$ (m)</th>
<th>Heating Coefficient, $J$</th>
<th>Attenuation Constant, $\alpha$ (m$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>A-C</td>
<td>A</td>
<td>A</td>
<td>A-C</td>
<td>A</td>
<td>A-C</td>
</tr>
<tr>
<td>2.440</td>
<td>6.15</td>
<td>5.78</td>
<td>2.20</td>
<td>1.61</td>
<td>.299</td>
<td>.218</td>
</tr>
<tr>
<td>2.685</td>
<td>5.28</td>
<td>5.56</td>
<td>1.73</td>
<td>1.50</td>
<td>.259</td>
<td>.225</td>
</tr>
<tr>
<td>2.972</td>
<td>5.22</td>
<td>5.36</td>
<td>1.70</td>
<td>1.63</td>
<td>.281</td>
<td>.269</td>
</tr>
</tbody>
</table>

It can be seen from Table 3.5 that both the films are exhibiting more or less the same characteristics at ISM band of microwave frequencies. The dielectric constants of both the films are low. The Arrow-Ch films used for measurements are prepared by the addition of 5% Chitosan gel to 10% Arrowroot gel. The dielectric constant of Arrow-Ch film can be further lowered to the desired value by increasing the amount of Chitosan in the film. The overall low value of the real part of complex permittivity suggests that the films are hydrophobic. The presence of water or moisture content would have increased the dielectric constant. Hence the films are non-polar in nature. Due to the low dielectric constant, the charge holding capacity of the films will be nominal. The Loss factor and hence the conductivity are slightly low for Arrow-Ch film as compared to Arrow film at low frequencies.
The dielectric loss highly depends on the relaxation process which involves local motion of polar groups. The dielectric loss occurs due to the friction between the molecular chains during this process [33]. The Arrow-Ch film has a structured nature due to the presence of the Chitosan matrix. Hence the friction between chains will be nominal for Arrow-Ch film. This accounts for the lower value of dielectric loss.

The conductivity varies as a function of frequency and dielectric loss of the material. As the dielectric loss is low for Arrow-Ch film, the microwave conductivity is also low for this film. The Skin depth gives a measure of the depth of penetration of the microwave signal. The Skin depth varies inversely with conductivity for a fixed frequency. Hence the Arrow-Ch film exhibits a higher Skin depth. The microwave heating coefficient is high for Arrow-Ch film. This implies that the material is not good for dielectric heating purposes. The attenuation constant represents the amount of attenuation of the wave within the film. The Arrow-Ch film has less attenuation as compared to Arrow film. Hence the wave penetrates more into the film and this can be verified from the value of Skin depth.

Figure 3.10 Absorption variation of Arrowroot and Chitosan films with frequency
The microwave absorption characteristics of Arrowroot and Chitosan films are as shown in figure 3.10. The Absorption coefficient (A) gives a measure of microwave power absorbed by a material. The minimum value of absorption coefficient is 0 and maximum value is 1. The value of absorption is highly dependent on frequency of the incident radiation. Several other important measurements such as Specific Absorption Rate (SAR) depend on the amount of absorbed microwave power. The dielectric heating is also depending on the amount of absorbed power. If microwave absorption is high, it can result in dielectric heating of the material. It can be observed from the figure that Arrowroot and Chitosan films exhibit similar microwave absorption characteristics. The amount of microwave absorption depends on the thickness of the film used.

(ii) Morphological properties

The SEM images of Arrow and Arrow-Ch films are shown in figures 3.11 & 3.12. Figure 3.11 gives 2000 times magnified view of the surface of the film whereas figure 3.12 gives 1500 times magnified view. As we can see, there are no pores on the surface of the films. Both films are also devoid of air gaps. Due to the

Figure 3.11. SEM image of Arrowroot film
lack of pores, they can be used as capsule material for embedding drugs. The embedded drugs will not interact with atmosphere or get contaminated by any other external factors. The film itself will not interact with air if kept in a dry container. The films prepared in the lab were aged for more than 14 months and still, the films were intact, maintaining the properties.

The capsules prepared from the film can be used either for sustained delivery or burst delivery of the drug in the required areas of the digestive system. We can also make the film porous if necessary. Such porous films can be used as transdermal patches for drug delivery through skin. Both films can be used as capsule coatings but the better tensile properties exhibited by Arrow-Ch film gives it a slight edge over Arrow film.

(iii) Mechanical Properties

The Stress-Strain relationship gives important information about the mechanical stability of the film. The Stress-strain graphs of the prepared films are shown in figure 3.13.
Figure 3.13. Stress-Strain curves of Arrowroot and Arrowroot-Chitosan film (0.8mm thick)

The graph follows an almost linear path for both Arrow and Arrow-Ch. However, it can be seen that the maximum yield point is reached for Arrow film for a smaller value of strain. This means Arrow film is less elastic compared to Arrow-Ch film. The Arrow-Ch film was strained more before it ruptured while Arrow film broke easily. Arrow-Ch film is more flexible and elastic. So it can be used for many biomedical applications where stress resilient features are preferred. Some significant applications may include eco-friendly Band-Aid’s, capsule cover, transdermal patch etc.

The Strain energy measured for Arrow-Ch film is 1205 J/m³ whereas that of Arrow film is 1237.5 J/m³. It means Arrow film consumes more energy with respect to Arrow-Ch film to strain to the maximum yield point. On a close inspection of the graph we can see that high stress is applied to Arrow film to achieve the same Strain as that of Arrow-Ch film. Therefore Arrow-Ch film has low strain energy value.
3.4.3 Applications

The dielectric properties of the prepared film are the same as that of certain biological constituents of human body. A comparative study of human tissues (37°C) at 3GHz [34] and their suggested equivalent phantoms is shown in table 3.6. The dielectric constant of the film can be fine-tuned by varying the amount of Chitosan in the film. This is one of the advantages of using Arrow-Ch film rather than standalone Arrow film. Hence Arrow-Ch film can be used as a phantom material representing these body tissues in scientific/medical applications.

The Arrowroot-Chitosan film shows better mechanical properties as compared to Arrowroot film. Thus it can be used in applications which demand good mechanical properties. The low dielectric constant enables it to be used in microwave phantom applications. It is also suggested to be used in eco-friendly Band-Aids and as capsule material in medical field.

Table 3.6. Dielectric properties of human tissues at 3 GHz and their equivalent phantoms

<table>
<thead>
<tr>
<th>Biological tissue at 37°C</th>
<th>Dielectric constant of standard sample</th>
<th>Equivalent phantoms in terms of ratio of gels used in preparation (Arrowroot: Chitosan)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Collagen</td>
<td>5.5-6.5</td>
<td>5:1</td>
</tr>
<tr>
<td>Bone marrow</td>
<td>4.2-5.8</td>
<td>5:1</td>
</tr>
<tr>
<td>Fat</td>
<td>5.28</td>
<td>5:2</td>
</tr>
<tr>
<td>Breast fat</td>
<td>5.15</td>
<td>5:2</td>
</tr>
<tr>
<td>Human abdominal wall fat</td>
<td>4.92</td>
<td>5:3</td>
</tr>
</tbody>
</table>

3.4.4 Conclusions

Arrowroot-Chitosan film was synthesized. The dielectric, mechanical and morphological characterization of Arrowroot-Chitosan film was done. It has been found that the film can be used as phantom material in microwave compatibility
applications. The dielectric properties of the film can be tuned to the desired value by adjusting the % composition of Chitosan in the film. The Arrow-Ch film has better stress withstanding capability than Arrowroot film. The structural properties of the film are increased by the addition of Chitosan. The film exhibits better strain energy values which say that the film can strain more for an applied stress. The elasticity of the film allows it to be used in applications where mechanical stability is of prime concern. It is also devoid of air gaps and pores. The films prepared are biodegradable as both Chitosan and Arrowroot are edible. With its unique dielectric, mechanical and morphological characteristics, Arrow-Ch film proves to be a versatile material and can find use in many diverse fields.
References


