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## Microwave

In the past few decades, many significant advances in polymer chemistry, such as the novel synthetic reagents and methods, as well as the advent of an analytical apparatus and techniques, have made the organic synthesis more dynamic and effective than ever before. However, the practical aspects for carrying out laboratory-scale reactions have changed little during this period. When heating was necessary, oil baths and heating jackets were the main equipment used. These traditional heating techniques were slow and time-consuming, and sometimes can lead to overheating and decomposition of the substrate and product. To overcome these difficulties microwaves have been employed in polymer chemistry to reduce the reaction times from hours to minutes, to increase yields and selectivity.

The application of microwaves introduced in industry in the 50’s. First of all in 1966 James W. Jacobs patented a new polymerization process and Synthesized Polyurethane foams using microwave irradiation. Dietmar Anders published the first paper of a continuous vulcanization process using microwave irradiation. In 1973 E. J. Bunclark patented a microwave assisted vulcanization system of different polymer mixtures. The slow development of the technique in polymer synthesis was principally attributed to the lack of controllability and reproducibility due to using poorly designed domestic microwave ovens as reactors. Safety is another consideration since explosions have been reported [4]. However, with the availability of commercial microwave equipment intended for polymer synthesis and the development of the solvent-free techniques, microwave assisted polymer chemistry has experienced exponential growth since the mid-1990s.

After that microwaves were considered as interesting tool for synthesizing polymers and organic compounds in the research laboratories. It is also found that rate of reaction increases using microwaves irradiation which takes less time to get product even reaction completes in few seconds or few minutes. It also gives good deal of products i. e. improves reaction yields, produces cleaner chemistries and discovers new reaction pathways. This is due to of its efficient way to transfer energy to the system, in comparison with conventional conductive heating methods. Hence microwaves synthesis is a new possibility for performing chemical transformations. It is also a green technology because atom economy is zero or nearly zero. But there is a debate on its instrumentation.

### Microwave Region

A microwave is a form of electromagnetic energy defined within the frequency range between 300 MHz – 300, 000 MHz (i. e 300GHz) and with a wave length interval between 1mm – 30 cm shown in Figure 1. 1. Within this region of electromagnetic energy, only molecular rotation is affected, not molecular structure. By International agreement, four frequencies were originally reserved for the use of electromagnetic fields for industrial, scientific and medical purposes other than communications: 915±25, 2450±13, 5800±75 and 22125±125 MHz, in order to avoid interferences between the equipments. These frequencies are known as ISM (Industrial, scientific and medical) band. From these four frequencies 2450 MHz (λ= 12. 2 cm) is preferred because it has the right penetration depth to interact with laboratory scale samples [4]. In other words this region can be explained as in the electromagnetic spectrum, microwave region is located between infrared radiation and radio frequencies and corresponding to wave length of 1cm to 1m (frequencies of 30 GHz to 300 MHz respectively). Domestic and industrial microwave heaters operates at 12. 2 cm (2450 MHz) or 33. 3cm (900MHz, so that there is no interference).

### Microwave Oven

Heating effect of water is very strong at 20GHz but the domestic ovens operate at a much lower frequency, 2. 45GHz. The reason for this is that it is necessary to heat food efficiently throughout its interior. If the frequency is optimal for maximum heating rate, the microwaves are absorbed in the outer region of the food, and penetrate only a short distance. At 2. 45 GHz the microwaves penetrate into depth, makes the water molecules vibrate at the rate of two billion times per sec and the food is heated up. A microwave oven consists of a high power source, a wave guide feed and the oven cavity. The source is generally a magnetron (Figure 1. 2) in which the microwaves are generated. A magnetron is a thermionic diode having an anode and a directly heated cathode. As the cathode is heated, electrons are released and are attracted towards the anode. The anode is made up of an even number of small cavities, each of which acts as a turned circuit. The gap across of each cavity behaves as a capacitance.

### Cavities and Magnetron

A very strong magnetic field is induced axially through the anode assembly, and has the effect of bending the path of the electrons as they travel from the cathode to the anode. As the deflected electrons pass through the cavity gaps, they induce a small charge into the turned circuit, resulting in the oscillation of the cavity. Alternate cavities are linked by two small wire straps which ensure the correct phase relationship. This process of oscillation continues until the oscillation has achieved sufficiently high amplitude. It is taken off the anode via an antenna. Of the 1200W of electric line power used by the magnetron, around 600W is converted into electromagnetic energy. The remainder is converted into heat that must be dissipated through air and water cooling.

### Mechanism of microwave heating

A material can be heated by applying energy to it in the form of high frequency electromagnetic waves due to the ability of electric field to exert a force on charged particles. If the Particle present in the substance can move freely through it, a current has been induced. However, if the charge carrier bound to certain regions they will move until a counter force balanced them and the net result is a dielectric polarisation. Both conduction and dielectric polarization are sources of microwave heating. The source of microwave dielectric heating lies in the ability of an electric field to polarize charges in a material and the inability of this polarization to follow rapid reversal of an electric field.

The total polarisation is the sum of a number of individual components.

µt = µe +µa +µd +µI

µe – the electronic polarization arises from the realignment.

µa – the atomic polarisation results from the relative displacement of nuclei due to the unequal distribution of charge within the molecule.

µd – the dipole polarisation , resulting from the orientation of permanent dipoles by the electric field.

µI – the interfacial polarization they experience

The major factor is dipole polarization. It is due to the dipole moment which in turn results from the differing electronegativities. At low frequencies, the time taken by the electric field to change direction is longer than the response time of dipoles and the dielectric polarisation keeps in phase with the electric field. The field provides energy necessary to make the molecules rotate into alignment. Some of the energy is transferred to the random motion each time dipole is knocked out of alignment and then realigned. The transfer of energy is so small, that the temperature hardly rises. If the electric field oscillates rapidly, it changes direction faster than the response time of the dipoles. Since the dipoles do not rotate, no energy is absorbed and the material does not heat up. In the microwave range of frequencies, the time in which the field changes is about the same as the response time of the dipoles. They rotate because of the torques, but resulting polarization lags the changes of the electric field. When the field is at a maximum strength, polarization may still be low. It keeps rising as the field weakens. The lags indicate that the material absorbs energy from the field and is heated.

## Microwave reactors for synthesis

### There are two types of reactors used for microwave assisted synthesis

(1) Multimode Reactors,(2) Monomode Reactors

Domestic microwave ovens as multimode reactors Figure 1. 3 are the most common instruments used in polymer synthesis since they are comparatively inexpensive and readily available. Using domestic microwave cavity has done a lot of satisfying different synthesis. However, the multimode reactors provide a field pattern with areas of high and low field strength, commonly referred to as “ hot and cold spots.” This non-uniformity of the field leads to the heating efficiency varying drastically between different positions of the sample. In addition, domestic microwave ovens lack the ability to monitor and control temperature. All of these characteristics can lead to poor experimental reproducibility. In order to carry out more accurately and safely different reactions, microwave domestic ovens can be simply modified by piercing a hole on the top of cavity. This allows the introduction of tube (acting as a air cooler) surrounded by a water cooler to maintain reactions under solvent reflux, or under inert atmosphere or allowing the addition of compounds in multistep procedures.

The single mode cavity, as the name implies, allows only a single mode to enter the cavity by waveguide. A properly designed monomode (Figure 1. 4) reactor can prevent the formation of “ hot and cold” spots. This advantage is very important for synthesis since the actual heating pattern can be controlled. Therefore, higher reproducibility and predictability are achieved. In addition, higher power levels can be obtained in single mode systems. Nowadays, more sophisticated monomode reactors are available, such as the CSIRO continuous microwave reactor and the CSIRO microwave batch reactor. These reactors allow temperature control via changing power and temperature monitoring with preinstalled digital thermometers. Moreover, some ovens even interface with Computers for reaction monitoring.

## Biodegradable and natural polymer

### Guar gum

Biopolymers are natural, non-toxic, polymeric substances, formed by organisms. They are a product of metabolic processes within cells that include complex, enzyme- induced polymerization of monomers. According to the IUPAC definition, these macromolecules include proteins, polysaccharides and nucleic acids (IUPAC, 1997). Except for nucleic acids, that convey genetic information, most biopolymers constitute structural or reserve material in cells. Others serve as protective gels, adhesives, etc. or are stress-induced exudates. Most biopolymeric materials are extracted from plant and animal products. A wide range of biopolymers is also derived from bacterial and fungal exudates. There exist another group of biopolymers that are synthetic and obtained by polymerization of natural substances, for example (poly) lactic acid. Additionally, some authors understand the term biopolymer as a biodegradable polymer. In this scenario we discuss our study material Guar gum extracted from guar bean.

In early time man cultivated guar in Indo-Pakistan sub continent for numerous generations, but recently guar has remained as a minor crop. Now it seems destined to assume a larger role among the domesticated plants that supply the food and needs of man. The discovery that guar seed endosperm could be a source of useful industrial gum brought this little known crop world recognition and started on its way to major crop prominence [6]. Guar gum has grown rapidly in industrial usage since its first industrial trail during the world war-II. Guar was first investigated as a source of gum in 1945 [7]. Guar seed production is concentrated in India, Pakistan and United States. Most guar is produced in regions in which the average rainfall is 75 cm or less per year

A gum may be generally defined as any water soluble polysaccharide that is extractable from land or marine plants or from microorganisms and that possesses the ability to contribute viscosity or gelling ability to their dispersions. This definition, however, excludes starches and pectins in this group. Common gums from plant include the seed galactomannans of guar and locust bean; the plant exudates, gum Arabic and gum tragacanth; and the sea weed derived gums, agar, carrageenan, and algin. Guar gum or guaran, is the endosperm polysaccharide of the seed of Cyamopsis tetragonolobus, family leguminosae, that grows naturally in India and Pakistan and has been introduced as cash crop in the United States in 1905 [8]. Guar plant belongs to family Fabaceae or Leguminaceae. Common name, is guar, from “ sankrist word” “ go” or “ gav”. Local name is cluster bean. Latin name is Cyamopsis tetragonolobus or C. psoraloides.

Guar gum (GG) derived from the seeds of guar plant Cyamoposis tetragonolobus (Leguminosae) is a natural nonionic branched polymer with β-D-mannopyranosyl units linked (1–4) with single membered α-D-galactopyranosyl units occurring as side branches. The number of branches in guar is responsible for both its easier hydration and its different hydrogen-bonding activity as compared to that of locust bean gum [9]. Native GG and its derivatives have been used in many fields (e. g. thickening agent, ion exchange resin and dispersing agent, etc.). Guar gum is a low cost, easily available, non-toxic and biodegradable polysaccharide. Its potential in hydrogel chemistry and new generation formulation technology still lay less exploited as compared to other backbones like starch, cellulose, chitosan, xanthan gum etc. The guar seed is dicotyledonous, having a diameter of approximately 1/8 in (8 mm). The hull is loosened by water soaking and removed by grinding. The difference in hardness of the various seed components is utilized, and purification is accomplished by multi stages of grinding and sifting.