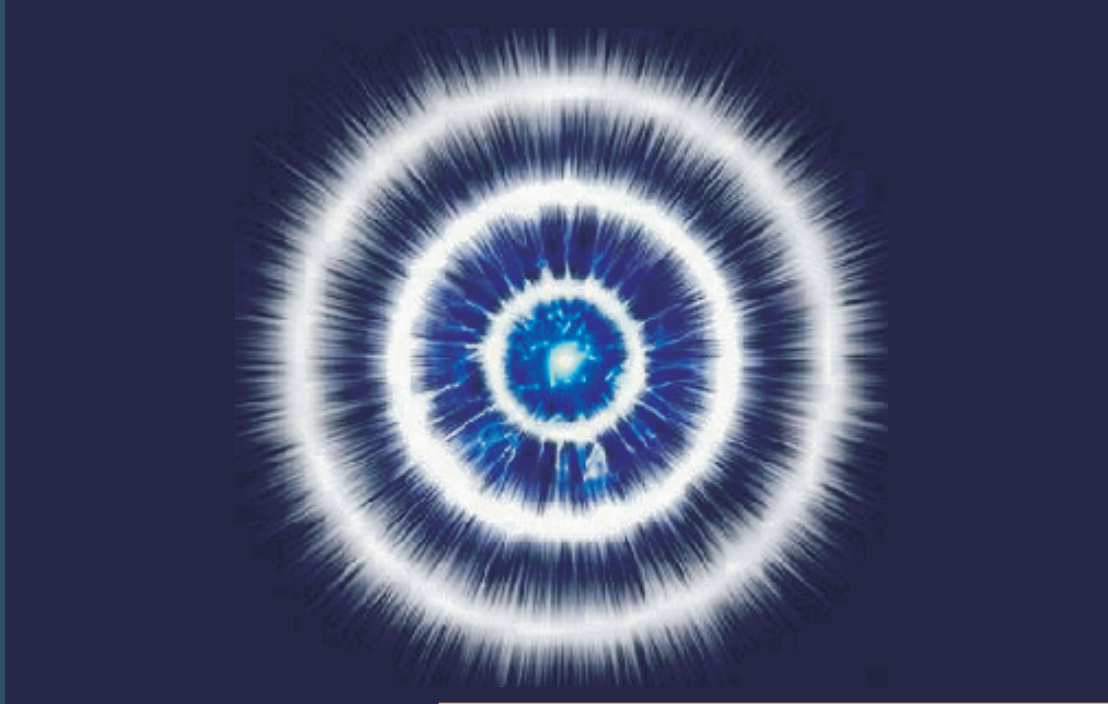

Microwave Irradiation and its Application in Textile Industries

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Introduction

Textile processing consumes huge amount of energy as in dye fixation and heat setting. Heat can be transferred to material by radiation, conduction and convection. These three ways of heat transfer can be used either separately or in combination. Saving time and energy is of immediate interest to textile industry. The introduction of new techniques which will allow less energy to be used is a highly important area of activity to consider. A relatively short section of properly designed microwave heating can increase production speeds.

In microwave processing, energy is supplied by an electromagnetic field directly to the material. This results in rapid heating throughout the material thickness with reduced thermal gradients. Volumetric heating can also reduce processing times and save energy. The microwave field and the dielectric response of a material govern its ability to heat with microwave energy. Knowledge of electromagnetic theory and dielectric response is essential to optimize the processing of materials through microwave heating.

This review addresses major basic and practical aspects of microwave irradiation as means of heat generation. Emphasis is placed on electromagnetic field dielectric response as well as application of microwave heating to textile coloration, processing equipment, and microwave/materials interactions. A future outlook is also reported within the increasing importance of microwave irradiation in textile coloration and other domains of application.

In the last three decades, the microwave oven has become an essential appliance in most kitchens. Faster cooking times and energy savings over conventional cooking methods are the primary benefits. Although the use of microwaves for cooking food is widespread, the application of this technology to the processing of materials is a relatively new development. The use of microwave energy for processing materials has the potential to offer similar advantages in reduced processing times and energy savings. In conventional thermal processing, energy is transferred to the material through convection, conduction, and radiation of heat from the surfaces of the material. In contrast, microwave energy is delivered directly to materials through molecular interaction with the electromagnetic field. In heat transfer, energy is transferred due to thermal gradients, but microwave heating is the transfer of electromagnetic energy to thermal energy and is energy conversion, rather than heat transfer. This difference in the way energy is delivered can result in many

potential advantages to using microwaves for processing of materials. Because microwaves can penetrate materials and deposit energy, heat can be generated throughout the volume of the material. The transfer of energy does not rely on diffusion of heat from the surfaces, and it is possible to achieve rapid and uniform heating of thick materials. In traditional heating, the cycle time is often dominated by slow heating rates that are chosen to minimize steep thermal gradients that result in process-induced stresses. For polymers and ceramics, which are materials with low thermal conductivities, this can result in significantly reduced processing times. Thus, there often is a balance between processing time and product quality in conventional processing. As microwaves can transfer energy throughout the volume of the material, the potential exists to reduce processing time and enhance overall quality.

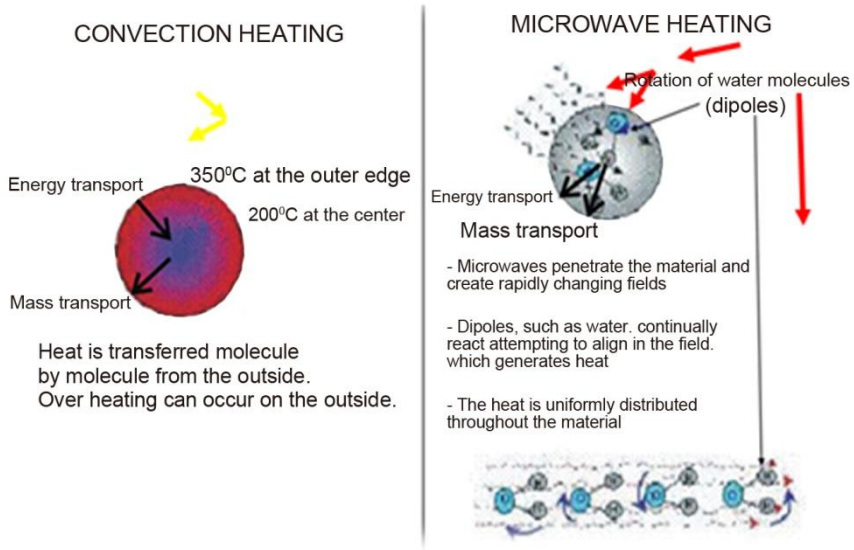


Fig. 1. *Energy transfer comparison.*

In addition to volumetric heating, energy transfer at a molecular level can have some additional advantages. Microwaves can be utilized for selective heating of materials. The molecular structure affects the ability of the microwaves to interact with materials and transfer energy. When materials in contact have different dielectric properties, microwaves will selectively couple with the higher loss material. This phenomenon of selective heating can be used for a number of purposes. In conventional joining of ceramics or polymers, considerable time and energy is wasted in heating up the interface by conduction through the substrates. With microwaves, the joint interface can be heated in-situ by incorporating a higher loss material at the interface [1]. In

multiple phase materials, some phases may couple more readily with microwaves. Thus, it may be possible to process materials with new or unique microstructures by selectively heating distinct phases. Microwaves may also be able to initiate chemical reactions not possible in conventional processing through selective heating of reactants. Thus, new materials may be created.

In recent literature, many researchers report non-thermal phenomena that have been broadly termed ‘‘microwave effects’’. Examples of the microwave effect include enhanced reaction rates of thermosetting resins during microwave curing [2] and faster densification rates in ceramics sintering [3]. Although there is considerable debate over the existence of microwave effects, many papers present unexpected results that do not seem to be a consequence of reduced thermal gradients possible within microwave processed materials. Critics of the microwave effect often claim that differences can be attributed to poor temperature measurement and control of experimental conditions that result in systematic error. Although direct heating by microwaves can offer advantages over conventional heat transfer, the different mechanism of energy transfer in microwave heating has also resulted in several new processing challenges. Because energy is transferred by the electromagnetic field, non uniformity within the electromagnetic field will result in non-uniform heating. As materials are processed, they often undergo physical and structural transformations that affect the dielectric properties. Thus, the ability of microwaves to generate heat varies during the process. Sharp transformations in the ability of microwaves to generate heat can cause difficulties with process modeling and control. Understanding the generation, propagation, and interaction of microwaves with materials is critical. Because the processing equipment determines the electromagnetic field, the design of microwave equipment is particularly important. The properties of the electromagnetic field, chemical composition of the material being processed, structural changes that occur during processing, size and shape of the object being heated, and the physics of the microwave/materials interactions all complicate microwave process [4–12].

Contents

Chapter 1

Microwaves

Microwaves, like other radio waves, are a form of electromagnetic waves. Electromagnetic waves are wavelike oscillations of electric and magnetic fields. Electric fields are what makes electric charges attract or repel. Positive or negative electric charges produce electric fields which in turn act on other charges. In a similar way, magnetic fields cause magnetic forces [13]. These fields are perpendicular to each other and continually oscillate between maximum positive and maximum negative (pointing in the opposite direction). The microwaves used to heat food in microwave ovens have a wavelength of 12.2 cm and oscillate at a frequency of 2.45 gigahertz. Giga means billion, so the electric and magnetic fields oscillate fast enough to make 2.45 billion complete cycles each second [13-15].

Microwaves are electromagnetic irradiation in the frequency range 0.3–300GHz (wavelengths of 1mm to 1m), between infrared radiation and radio frequencies. Microwave radiation was discovered as a heating method in 1946, with the first commercial domestic microwaves being introduced in the 1950s. The first commercial microwave for laboratory utilization was recognized in 1978. Over the last decade, microwave dielectric heating as an environmentally benign process has developed into a highly valuable technique, offering an efficient alternative energy source for numerous chemical reactions and processes [16]. It has many advantages compared to conventional oil-bath heating, such as (1) non-contact heating, (2) energy transfer instead of heat transfer, (3) higher heating rate, and (4) rapid start-up and stopping of the heating,(5) uniform heating with minimal thermal gradients, (6) selective heating properties, (7) reverse thermal effects (heating starting from the interior of the material body), (8) energy savings and (9) higher yields in shorter reaction time.

Microwave heating is based on dielectric heating, the ability of some polar liquids and solids to absorb and convert microwave energy into heat. In this context, a significant property is the mobility of the dipoles by either ionic conduction or dipolar polarization and the ability to orient them according to the direction of the electric field. The orientation of the dipoles changes with the magnitude and the direction of the electric field. Molecules that have a permanent dipole moment are able to align themselves through rotation, completely or at least partly, with the direction of the field. Therefore, energy is lost in the form of heat through molecular friction and dielectric loss. The amount of heat produced by this process is directly related to the capability of the matrix to align itself with the frequency of the applied electric field. If the dipole does not have enough time to realign, or reorient too rapidly with the applied field, no heating occurs [17].

The allocated frequency of 2.45GHz employed in all commercial systems is placed between these two extremes, and offers the molecular dipole time to align in the field, but not to follow the alternating field precisely. The heating characteristics of a particular material under microwave irradiation conditions are dependent on its dielectric properties.

When organic synthesis is performed by an external heat source, for example, an oil bath, heat is conducted from the surface into the interior of the specimen, the reaction will be slow and inefficient for transferring energy into the system, because it depends on the thermal conductivity of the different materials that must be penetrated, and results in the temperature of the reaction vessel is being higher than that of the reaction mixture. In contrast, microwave irradiation produces efficient internal heating (in core volumetric heating) by direct coupling of microwave energy with the solvents, reagents or catalysts that are present in the reaction mixture [17–19]. Microwave activation has also been widely used in various polymerization reactions, such as poly condensation, free and controlled radical polymerizations, ring-opening polymerization and polymer processing (including polymer modification, curing processes and preparation of dental materials) [18-19]. Recently, several excellent books and reviews have been published on microwave- assisted polymer synthesis and processing [20-22].

1.1 Electromagnetic Spectrum

The *electromagnetic spectrum* is the range of all possible frequencies of electromagnetic radiation. The "*electromagnetic spectrum*" of an object is the characteristic distribution of electromagnetic radiation emitted or absorbed by that particular object.

The electromagnetic spectrum extends from low frequencies used for modern radio communication to gamma radiation at the short-wavelength (high-frequency) end, thereby covering wavelengths from thousands of kilometers down to a fraction of the size of an atom (Fig. 2) [23].

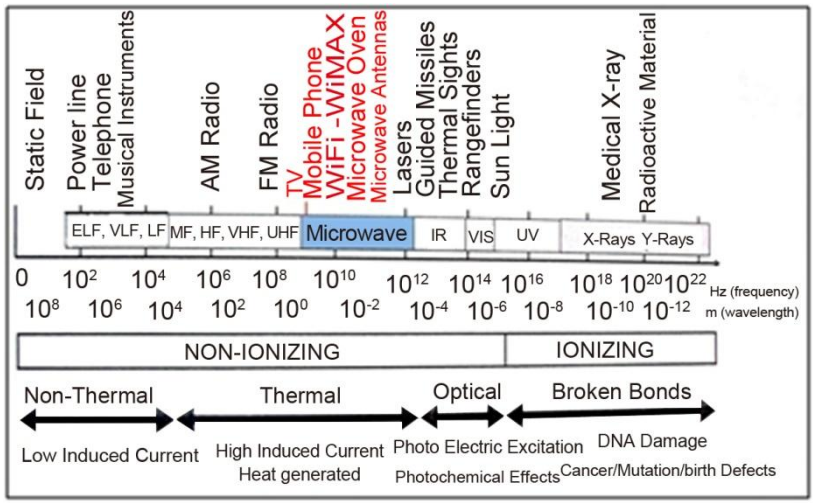


Fig. 2. *The electromagnetic spectrum.*

Table I. *Main interactions of electromagnetic radiation with matter [23].*

Region of the spectrum	**Main interactions with matter**
Radio	Collective oscillation of charge carriers in bulk material (plasma oscillation). An example would be the oscillation of the electrons in an antenna.
Microwave	Plasma oscillation, molecular rotation.
Near infrared	Molecular vibration, plasma oscillation (in metals only).
Visible	Molecular electron excitation (including pigment molecules found in the human retina).
Ultraviolet	Excitation of molecular and atomic valence electrons, including ejection of the electrons (photoelectric effect).
X-rays	Excitation and ejection of core atomic electrons, Compton scattering (for low atomic numbers).
Gamma rays	Energetic ejection of core electrons in heavy elements, Compton scattering (for all atomic numbers), excitation of atomic nuclei, including dissociation of nuclei.
High-energy gamma rays	Creation of particle-antiparticle pairs. At very high energies a single photon can create a shower of high-energy particles and antiparticles upon interaction with matter.

1.2 Fundamentals of Microwaves

Microwave frequencies occupy the electromagnetic spectrum between radio frequencies and infrared radiation with the frequencies of 300 GHz to 300 MHz, (as mentioned before) which corresponds to the wavelengths of 1 mm to 1 m, respectively (Fig.3).Their major applications fall into two categories, depending on whether they are used for transmission of information (telecommunication) or transmission of energy. However, the extensive application of microwaves in the field of telecommunication (e.g., most of the wavelengths in the range of 1 cm to 25 cm are used for mobile phones, radar, and radar line transmissions) has caused only specially assigned frequencies to be allocated for energy transmission (i.e., for industrial, scientific, or medical applications). Currently, to minimize interferences with telecommunication devices, these household and industrial microwave applicators are operated only at a few precise frequencies with narrow tolerance that are allocated under international regulations. For example, the most common microwave applicators (i.e., domestic microwave ovens) use the frequency of 2.45 GHz. This is probably why most commercially available microwave reactors devoted for chemical use operate at the same frequency; however, some other frequencies are also available for heating [24].

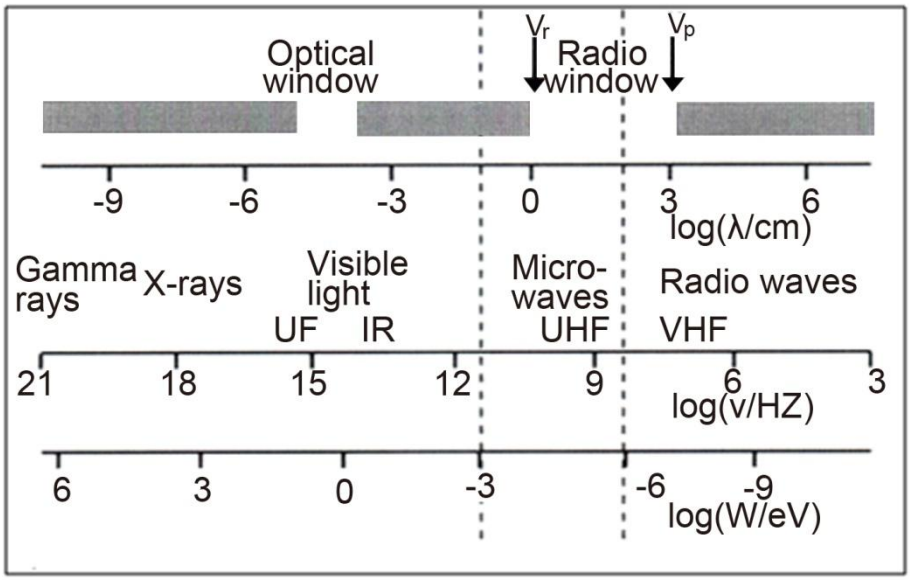


Fig. 3. *Spectrum of electromagnetic radiation: λo, wavelength in free space; W, hν quantum energy; v_r, lowest resonance frequency in the rotational spectrum of water; and v_p, plasma frequency of the ionosphere.*

1.3 Interaction of Microwaves with Materials[24]

When a piece of material is exposed to microwave irradiation, microwaves can be:

a) Reflected from its surface if it is an electrical conductor (e.g., metals, graphite, etc.),
b) Penetrate the material without absorption in the case of good insulators with good dielectric properties (e.g., quartz glass, porcelain, ceramics),
c) Absorbed by the material if it is a lossy dielectric (i.e., a material that exhibits so-called dielectric losses, which in turn results in heat generation in a quickly oscillating electromagnetic field, such as water).

When a strongly conducting material (e.g., a metal) is exposed to microwave radiation, microwaves are largely reflected from its surface. However, the material is not effectively heated by microwaves; in response to the electric field of microwave radiation, electrons move freely on the surface of the material, and the flow of electrons can heat the material through a resistive (ohmic) heating mechanism (Fig. 4a). In the case of insulators (e.g., porcelain), microwaves can penetrate the material without any absorption, losses, or heat generation. They are transparent to microwaves (Fig4b). For some dielectrics, the reorientation of either permanent or induced dipoles during passage of microwave radiation, which is electromagnetic in nature, can give rise to absorption of microwave energy and heat generation due to the so-called dielectric heating mechanism (Fig. 4c).

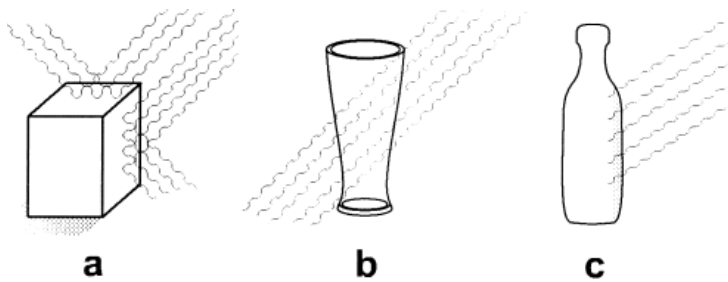


Fig. 4. *Interaction of microwaves with different materials: (a) electrical conductor, (b) insulator, and (c) lossy dielectric.*

Dependent on the frequency, the dipole may move in time to the field, lag behind it, or remain apparently unaffected. When the dipole lags behind the field (polarization losses), interactions between the dipole and the field lead to

an energy loss by heating (i.e., by dielectric heating mechanism), the extent of which is dependent on the phase difference of these fields (Fig.5b).

In fact, the electric field component of microwave radiation is responsible for dielectric heating mechanisms because it can cause molecular motion by either migration of ionic species (conduction mechanism) (Fig. 5a) or rotation of dipolar species (dipolar polarization mechanism) (Fig 5b).

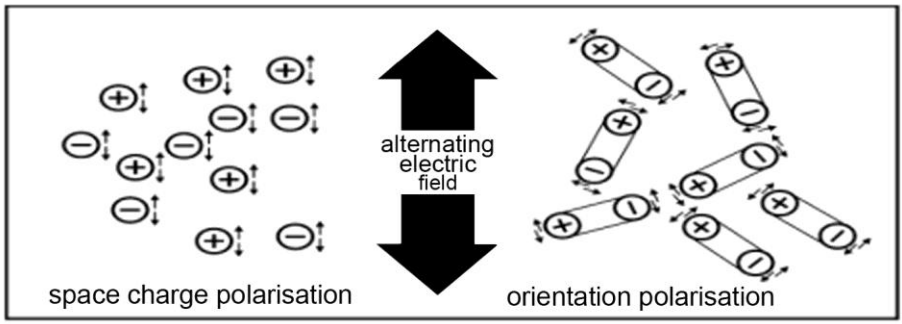


Fig. 5. *Interaction of charge particles and dipolar molecules with electromagnetic radiation: space charge polarization (a) and orientation-polarization (b), respectively.*

In a microwave field, the electric field component oscillates very quickly (at 2.45 GHz, the field oscillates 4.9 x 10^9 times/sec), and the strong agitation, provided by cyclic reorientation of molecules, can result in an intense internal heating that can lead to heating rates in excess of 10 ℃/sec when microwave radiation of a kilowatt-capacity source is used [25].

In practice, most good dielectric materials are solid and examples include ceramics, mica, glass, plastics, and the oxides of various metals, but some liquids and gases can serve as good dielectric materials as well. For example, deionized water is a fairly good dielectric; however, possessing polar molecules (i.e., a dipole moment) can couple efficiently with microwaves to lead to heat generation due to polarization losses. Thus, such substances that are counted among dielectrics but exhibit some polarization losses that result in dielectric heating are also called dielectric lossy materials or in general lossy materials. On the other hand, n-hexane, having a symmetrical molecule, does not possess a dipole moment and does not absorb microwaves.

To apply microwaves to carry out chemical processes, it is most important to find at least one component that is polarizable and whose dipoles can reorient (couple) rapidly in response to changing electric field of microwave radiation. Fortunately, a number of organic molecules and solvents fulfill these

requirements and are the best candidates for microwave applications.

The first step is to analyze the reaction components together with their dielectric properties, among which the most important is dielectric constant (ε_r), sometimes called electric permeability. Dielectric constant (ε_r) is defined as the ratio of the electric permeability of the material to the electric permeability of free space (i.e., vacuum), and its value can derived from a simplified capacitor model (Fig. 6).

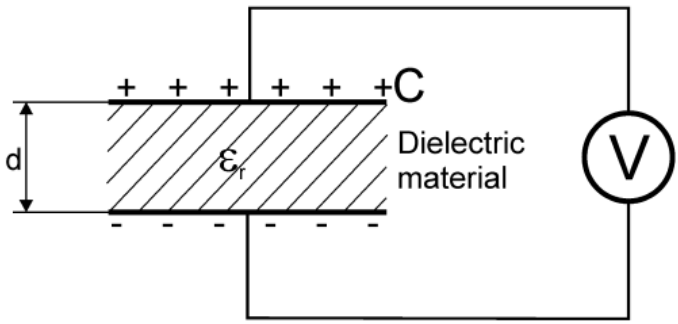


Fig. 6. *An electrical capacitor consisting of two metal plates separated by an insulating material called a dielectric.*

When the material is introduced between two plates of a capacitor, the total charge (C_0) stored in the capacitor will change (C) (Eq. 1.1). The change depends on the ability of the material to resist the formation of an electric field within it and, finally, to get polarized under the electric field of the capacitor.

$$\varepsilon_r = C/C_0 \tag{1.1}$$

where C_0 is the capacitance of the capacitor with vacuum and C is the capacitance of the capacitor with the material.

Thus, dielectric constants (ε_r) that determine the charge holding ability of the materials are characteristic for each substance and its state and vary with temperature, voltage, and, finally, frequency of the electric field. Dielectric constants for some common materials are given in Table II.

Table II. *Dielectric constants (ε_r) of some common materials at 20 ℃.*

Material	Dielectric constant (ε_r)	Material	Dielectric constant (ε_r)
Vacuum	1	Titanium dioxide	100
Air (1 atm)	1.00059	Water	80
Air (100 atm)	1.0548	Acetonitrile	38
Glass	5-10	Liquid ammonia(-78 ℃)	5
Quartz glass	5	Ethyl alcohol	25
Porcelain	5-6	Benzene	2
Mica	3-6	Carbon tetrachloride	2
Rubber	2-4	Hexane	2
Nylon	3-22	Plexiglass	3
Paper	1-3	Polyvinyl chloride	3
Paraffin	2-3	Polyethylene	2
Soil (dry)	2.5-3	Teflon	2
Wood(dry)	1-3	Polystyrene (foam)	1.05

Air has nearly the same dielectric constant as vacuum (ε_r = 1.00059 and 1.00000, respectively). Polar organic solvents (i.e., water, acetonitrile, ethyl alcohol) are characterized by relatively high values of dielectric constants and, in turn, can be heated by dielectric heating mechanism under microwave irradiation. Nonpolar organic solvents (i.e., benzene, carbon tetrachloride, *n*-hexane) have low dielectric constants and, in fact, show negligible heating effects under microwave irradiation. Most plastics range in the low values of dielectric constants (i.e., between 2 and 3); therefore, some of these materials, besides glass and quartz glass, are used to manufacture reaction vessels for microwave application due to their good chemical as well as temperature resistance (e.g., Teflon, PEEK). Thus, heating a material in microwave ovens is based upon the ability of some liquids as well as solids to absorb and to transform electromagnetic energy into heat.

The heating rate (i.e., temperature increase) of the material under microwave irradiation also depends on the shape and size of the sample. Eventually, the sample size, penetration depth, and heating rate are strongly coupled during microwave irradiation and may finally result in more homogeneous or heterogeneous heating of the material, which in turn can result in overheating of

the material and creation of so-called hot spots in the latter case [23].

It is worth stressing that microwaves in comparison with conventional heating methods are the means of volumetric heating of the material that gives rise to a very rapid energy transfer into the material being heated. In conventional heating, heat flow is initiated on the material's surface and the rate of heat flow into the center is dependent on the material's thermal properties and the temperature differentials. A conventional oven is required to be heated to temperatures much higher than is required by the material itself (Fig. 7).

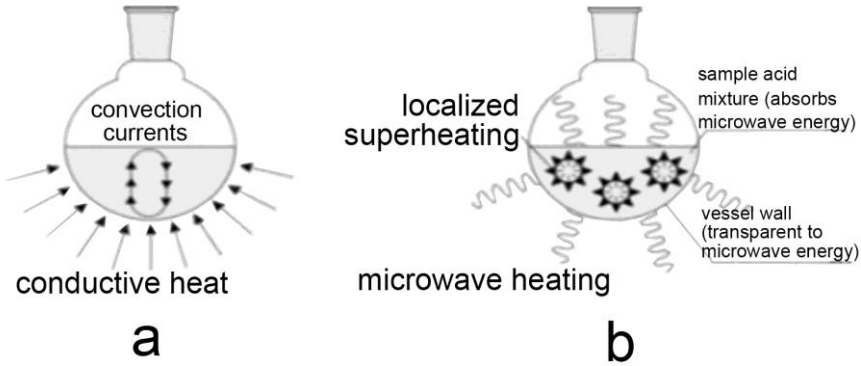


Fig. 7. *Different heating mechanisms for conventional (a) and microwave heating (b).*

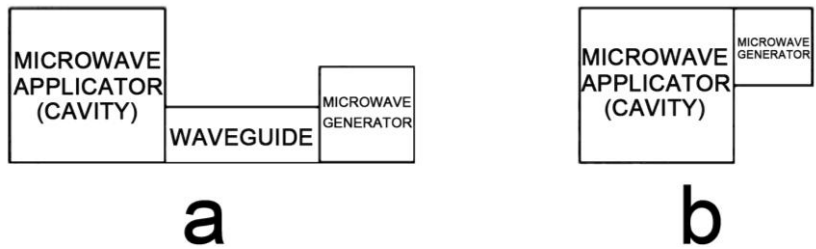


Fig. 8. *Schemes of microwave devices with (a) and without (b) transmission line.*

1.4 Microwave Equipment

Microwave devices that are dedicated to carry out chemical processes are similar to other microwave systems that consist of (Fig.8):

1) Microwave power source (generator),
2) Transmission line (waveguide) that delivers microwaves from the generator into an Applicator,

3) Microwave applicator (cavity).

1.4.1 Microwave Generators

The main types of microwave power sources are magnetrons and klystrons. Magnetrons, which are commonly used in microwave ovens, are mass produced and thus are cheap and easily available on the market. Therefore, it is common practice to use the same magnetrons for laboratory and industrial microwave processing. In general, magnetrons are vacuum devices consisting of an anode and a cathode, and the anode is kept at a higher potential than the cathode. As soon as the cathode is heated, electrons are emitted from it and are accelerated toward the anode by the electric field. At the same time, external magnets mounted around the magnetron anode block create the magnetic field parallel to the axis of the cathode. This magnetic field forces the electrons to rotate around the cathode before they can reach the anode. The rotating electrons form a rotor moving around the cathode synchronously in the way that they decelerate and thus transform their energy into microwave oscillations in the cavities cut in the anode blocks. A typical anode block consists of an even number of small cavities that form a series of microwave circuits, which are tuned to oscillate at a specified frequency dependent on the dimensions and shape of the cavities. Finally, microwave energy from one of the resonant cavities is coupled to the output transmission line, usually terminated with an output antenna (Fig. 9).

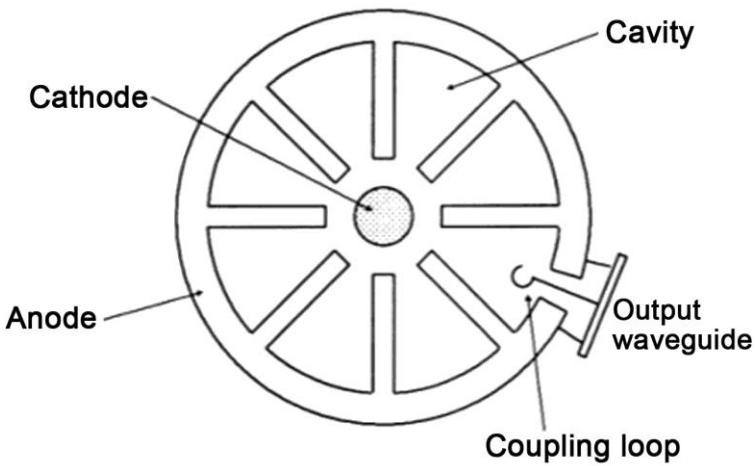


Fig. 9. *Schematic diagram of a magnetron shown in cross section. Reprinted from National Academy Press {26}. Microwave Processing of Materials. National Academy Press, with permission.*

1.4.2 Transmission Lines (Waveguides)

Microwaves can be easily transmitted through various media. Hence, an applicator can be remote from the power source and connected to it via the transmission line, in which microwaves can be propagated using three types of modes:

1) Transverse electromagnetic mode (TEM).
2) Transverse electric (TE).
3) Transverse magnetic (TM).

1.4.3 Microwave Applicators (Cavities)

Microwave applicators may appear in many different shapes and dimensions, and in fact their design is critical to processes run under microwave conditions since within applicators the microwave energy must be efficiently coupled to the material.

Every efficient application of microwave energy to perform chemical syntheses requires reliable temperature measurement as well as continuous power feedback control, which enable heating of reaction mixtures to a desired temperature without thermal runaways. Moreover, power feedback control systems that are operated in most microwave reactors enable a synthesis to be carried out without knowing the dielectric properties and/or conductive properties of all the components of the reaction mixture in detail. On the other hand, temperature control during microwave irradiation is a major problem that one faces during microwave-assisted chemical reactions. Maintaining good thermal contact with the material being heated is crucial when heating using microwave irradiation and it is important that temperature probes produce a minimum perturbation to the existing field in a microwave cavity. In general, temperature in microwave field can be measured by means of:

- Fiberoptic thermometer
- Shielded thermocouple
- Pyrometer (infrared sensor)

Fiberoptic thermometers can be applied up to 300 ℃ but are too fragile for real industrial applications. In turn, optical pyrometers and thermocouples can be used, but pyrometers measure only surface temperatures, which can be lower than the interior temperatures in reaction mixtures. Application of thermocouples, which in the case of microwaves are metallic probes, screened

against microwaves, can result in arcing between the thermocouple shield and the cavity walls, leading to failures in thermocouple performance.

The temperature of microwave-irradiated samples can be also measured by inserting either a thermocouple or thermometer into the hot material immediately after turning off microwave power. This rather simple procedure may sometimes help if no other means of temperature measurements is provided. However, it must be stressed that the temperature of many materials can drop quickly as soon as microwave power is switched off [23,26].

1.5 Methods for Performing Reactions under Microwave Irradiation

Two pioneering works for the synthesis under microwave conditions that were published almost 20 years ago described several organic syntheses that were completed in household microwave ovens with high yield when conducted in sealed vessels [27,28]. They used commercially available screw-up pressure vessels made of glass, Teflon, and PTFE (i.e., being transparent for microwaves). This strategy has been successfully applied to a number of syntheses, but it always generates a risk of hazardous explosions. Later, there were reported 45 different reaction procedures with a commercial microwave oven and poly(ethylene terephthalate) vessels designed for acid digestion. Since then, different techniques have been developed.

The simplest method for conducting microwave-assisted reactions involves irradiation of reactants in an open vessel. Such a method, termed microwave-organic reaction enhancement (MORE), was developed [29]. During the reaction, reactants are heated by microwave irradiation in polar, high-boiling solvent so that the temperature of reaction mixture does not reach the boiling point of a solvent. Despite the convenience, a disadvantage of the MORE technique consists in limitation to high boiling polar solvents such as DMSO, DMF, *N*-methylmorpholine, diglyme, etc.

Microwave heating has been proved to be of benefit particularly for the reactions under “dry” media in open vessel systems (i.e., in the absence of a solvent, on solid support with or without catalysts) [30]. Reactions under “dry” conditions were originally developed in the late 1980s [31]. Solvent less systems under microwave irradiation offer several advantages. The absence of solvent reduces the risk of explosions when the reaction takes place in a closed vessel. Moreover, aprotic dipolar solvents with high boiling points are

expensive and difficult to remove from the reaction mixtures [23].

1.5.1 Microwave Irradiation vis-a-vis to Conventional Heating

In conventional or surface heating, the process time is limited by the rate of heat flow into the body of the material from the surface as determined by its specific heat, thermal conductivity, density and viscosity. Surface heating is not only slow, but also non-uniform with the surfaces, edges and corners being much hotter than the inside of the material. Consequently, the quality of conventionally heated materials is variable and frequently inferior to the desired result.

Imperfect heating causes product rejections, wasted energy and extended process times that require large production areas devoted to ovens. Large ovens are slow to respond to needed temperature changes, take a long time to warm up and have high heat capacities and radiant losses. Their sluggish performance makes them slow to respond to changes in production requirements making their control difficult, subjective and expensive [32-35].

Conversely, with microwaves, heating the volume of a material at substantially the same rate is possible. This is known as volumetric heating. Energy is transferred through the material electro-magnetically, not as a thermal heat flux. Therefore, the rate of heating is not limited and the uniformity of heat distribution is greatly improved. Heating times can be reduced to less than one percent of that required using conventional techniques [32, 34, 36].

1.5.2 The Advantages of Microwave

Because volumetric heating is not dependent on heat transfer by conduction or convection, it is possible to use microwave heating for applications where conventional heat transfer is inadequate. One example is in heterogeneous fluids where the identical heating of solids and liquids is required to minimize over-processing. Another is for obtaining very low final moisture levels for product without over-drying. Other advantages include [37]:

- Microwaves generate higher power densities, enabling increased production speeds and decreased production costs;
- Microwave systems are more compact, requiring a smaller equipment space or footprint. Microwave energy is precisely controllable and can be turned on and off instantly, eliminating the need for warm-up and cool-down;

- Lack of high temperature heating surfaces reduces product fouling in cylindrical microwave heaters. This increases production run times and reduces both cleaning times and chemical costs;
- Microwaves are a non-contact drying technology. One example is the application of IMS planar dryers in the textile industry, which reduce material finish marring, decrease drying stresses, and improve product quality;
- Microwave energy is selectively absorbed by areas of greater moisture. This results in more uniform temperature and moisture profiles, improved yields and enhanced product performance;
- The use of industrial microwave systems avoids combustible gaseous by-products, eliminating the need for environmental permits and improving working conditions.

1.5.3 Disadvantages of Microwave

1) Limited transmission capacity;
2) Certain topographical conditions must be observed and in certain circumstances erection of special structures (pylons) is required;
3) Relay stations necessary for long distances;
4) Disruptions can be caused by the weather.

1.5.4 Harmful Effects of Microwave

In its interaction with matter, microwave energy may be:

- Reflected as in case of metal;
- Transmitted as in case of glass;
- Absorbed as in case of living tissues.

The latter case is the most important consequence of human exposure to microwave radiation. If the environmental temperature and humidity are too high, the person's body temperature will increase. Thus, the biological thermal stress from whole body exposure at power densities on the order of 1-10 mW/cm^2 depends strongly on the environmental temperature and humidity [37, 38]. Most of the documented harmful biological effects on man from microwaves are attributed to hyperthermia. These include damage mainly to the eyes and to the testicles which are not able to efficiently dissipate absorbed energy at a rate greater than 10 W/m^2.

Among the symptoms which may be observed for workers chronically exposed to microwaves are: increased fatigability, periodic or constant headaches, extreme irritability, sleeping during work and decrease in olfactory sensitivity.

Generally, the possible health hazard due to exposure to microwaves is highly dependent on the field strengths, frequencies and likely duration of exposure. On the other hand, metallic implants in human bodies may act as antennas in microwave radiation field and possibly cause adverse health effects by heating local tissues. For example, microwave radiation may interact with cardiac pacemakers and steps have to be taken to prevent this interference.

1.5.5 Microwave Safety

Using patented applicator design geometries and a unique slotted choking mechanism, IMS technology reduces microwave leakage from system entry and exit points to virtually non-detectable levels for both their planar and cylindrical heating systems. This poses no threat of electro-magnetic radiation to the health and safety of equipment operators. IMS heaters and dryers operate at a twenty times higher level of electromagnetic emission safety than that specified by the FDA for domestic microwave systems. As a further precaution, all IMS control systems are supplied with safety interlocks and leakage detectors that shut down power instantaneously in the event of equipment malfunction or misuse.

1.5.6 Economical Aspects

A common misconception is that microwave heating is always more expensive than heating by conventional techniques. The actual answer depends on the application. In some cases, microwaves can be 50% more efficient than conventional systems, resulting in major savings in energy consumption and cost.

When used as a Pre-Dryer in combination with conventional gas or oil heated air dryers, IMS microwave systems allow overall production capacities to be increased by 25 to 93%. This is because the pre-dryer performs three functions, namely:

- Removes residual moisture;
- Preheats moisture to the evaporative temperature;
- Equalizes the moisture level of product to the conventional dryer;
- With current energy costs, the return on capital invested in an IMS

pre-dryer can vary from 12 to 24 months.

When used as a Post-Dryer in combination with conventional gas or oil-heated dryers, IMS microwave systems are disproportionately more efficient than conventional dryers at achieving final moisture levels of less than 20%. This is because the lower the moisture level, the more difficult it is to drive moisture from the center of material to the surface by conventional heat conduction and convection processes. An IMS post-dryer provides:

- Uniformity of moisture control and surface temperature of the final product;
- Higher production efficiency due to increased process speeds;
- Improved product quality resulting from reduced surface temperatures, compared with conventional post-dryer designs;
- Return on capital invested in an IMS post-dryer usually varies from 12 to 24 months.

In addition to the applications above, IMS planar units are often used as Stand-Alone Dryers.

These may be the most economical solution where minimal equipment floor space or footprint is available for a new application, or when expansion of existing production facilities would require building modifications to accommodate a conventional drying system.

In the case of liquid heating, the production cost of providing sensible heat transfer from microwave energy is approximately one third higher than using steam in a conventional heat exchanger. However, this is offset by several factors, including:

- The reduced capital investment in steam boilers, steam trains, condensate collection and water treatment plant;
- The ability to use high power densities enables microwave heaters to substantially increase production rates;
- Uniform energy distribution minimizes fouling depositions in even the most viscous products. This is particularly important with thermally sensitive materials such as chemical polymers, food ingredients, nutraceuticals, biotech products & pharmaceuticals;
- With volumetric heating of multiphase products, solids loadings of 70% or higher can be processed since the carrier fluid is not used as the primary

heat delivery medium;

- The shorter residence times achievable with microwave heating improve product quality. Compared to conventional heating, IMS heated food products tend to retain a higher percentage of flavors and nutrients.

1.5.7 Maintenance

In addition to downtime for cleaning and inspection, conventional dryers and heat exchangers need periodic servicing with an expensive inventory of parts and a highly trained labor force. Apart from periodic examination for wear on the belt of a planar system or the tube in a cylindrical heater, the only part that requires maintenance on an IMS system is the magnetron. In the event of a malfunction or misuse through incorrect operation, this can be replaced and often repaired.

Although the operating life of a 915 MHz commercial magnetron can be greater, IMS recommends that the magnetron be replaced after 8,000 hours of operation. This translates to a maintenance cost of about US$1 per operating hour.

Low power 2,450 MHz magnetrons cannot be repaired, but larger units usually can be. A typical operating life for magnetrons at this frequency is 6,000 hours, although some vendors limit their warranty to 6 months or 500 hours.

Chapter 2

Microwave Coloration

Microwave dyeing takes into account the dielectric and the thermal properties of matter. The dielectric property refers to the intrinsic electrical properties that affect the dyeing by dipolar rotation of the dye and influences the microwave field upon the dipoles. The aqueous solution of dye has two components which are polar, in the high frequency microwave field oscillating at 2450 MHz. It influences the vibrational energy in the water molecules and the dye molecules.

The heating mechanism is through ionic conduction, which is a type of resistance heating. Depending on the acceleration of the ions through the dye solution, it results in collision of dye molecules with the molecules of the fiber. The mordant helps and affects the penetration of the dye and also the depth to which the penetration takes place in the fabric. This makes microwave superior to conventional dyeing techniques.

Microwave promoted organic reactions are known as environmentally Benign methods that can accelerate a great number of chemical processes. In particular, the reaction time and energy input are supposed to be mostly reduced in the reactions that are run for a long time at high temperatures under conventional conditions [39]. Microwave is a volumetric heating (fast), whereas conventional is a surface heating (slow), as shown in Fig.10. This fact has been realized in textile coloration by many authors [40].

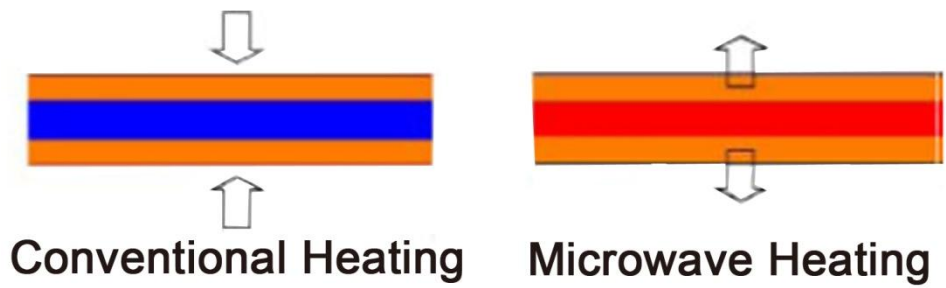


Fig. 10. *Microwave heating (volumetric) versus conventional heating (surface).*

2.1 Flax

Owing to the poor dyeability of flax fibres, a method based on microwave treatment of flax fiber with urea to improve its dyeability with reactive dyes was recently developed. It was found that the treated flax fibers had significantly improved dyeability. The causes to the improvement of the dyeability of the flax fiber were found to be the increased absorption of dye on the fiber and the

increased reaction probability between the dye and the fibre [41,42].

2.2 Polyester

2.2.1 Dyeing of Polyester Fabric

The possibility of coloration of polyester fiber using microwave irradiation was studied [43]. An investigation was undertaken to assess the effect of microwave heating on aqueous and solvent-pretreatment (perchloroethylene), as well as dyeing polyester fibers with disperse dyes. Microwave irradiation and solvent-pretreatment allow a high increase in dye uptake and dyeing rate acceleration. Performance of dye leveling and color homogeneity was achieved, which was found to be better than that obtained by conductive heating. The rate of dyeing with microwave heating is much faster than the rate of dyeing with conductive heating. In the case of microwave heating equilibrium can be reached within a few minutes, whereas in the case of conductive heating equilibrium was established after a few hours. Of course, this depends upon conditions of pretreatment (time, temperature and nature of solvent, as well as temperature of the dyeing bath). In other words, the heating technique is the main factor in this respect. Polyester dyeing rates were also evaluated; the result showed that the rate of dyeing with microwave heating is much faster than the rate of dyeing with conductive heating. In the case of microwave heating equilibrium can be reached within a few minutes, while in the case of conductive heating equilibrium was established after a few hours. The half dyeing time ($t_{1/2}$) was reached in a few second when using microwave irradiation versus four hour in case of conductive heating [43].

A study on the effect of microwave irradiation on the extent of aqueous sodium hydroxide hydrolysis of PET fibre and the impact of this treatment on its coloration with disperse dyes was investigated. Comparison of the results obtained from the microwave irradiation and the conventional heating methods showed that the rate of hydrolysis was greater using microwave irradiation. The treated fabric was then dyed using microwave irradiation to heat the dye bath. Increased levels of dye uptake were observed with increasing weight loss of the hydrolyzed polyester fabric [44-46].

The rates of dyeing of polyester fibre with amino – and hydroxy – substituted anthraquinones in aqueous N, N – dimethylformamide (DMF) using microwave and conductive heating have been compared. Structural changes, commonly associated with interactions between DMF and polyester fiber, were limited and minimal shrinkage occurred due to the low DMF to water ratio and short dyeing

times. Tensile properties reflected this minimal shrinkage through increased elongation at break, and differential scanning calorimetry also indicated that structural changes were minimal. Microwave radiation increased dye diffusion, shrinkage, and elongation at break compared to conductive heating. It is believed that molecular oscillation within the microwave–irradiated dyebath increases the rate of migration of dye to the fibre interface and enhances the plasticization of the polymer by DMF, thereby increasing the rate of dye diffusion within the fiber. Localization of energy associated with the use of microwave radiation is believed to be the predominant cause of increase in the rate of dyeing [47].

2.2.2 Effect of Microwave Pretreatment on the Dyeing Behavior of Polyester Fabric

Polyester fabric (filament by filament) was pretreated in a microwave oven in the presence of solvent and subsequently dyed with commercial disperse dyes {Dispersol Red C-B (CI Disperse Red 91) and Dispersol blue B-G (CI Disperse Blue 26} at different temperatures and for different durations of time. It was observed that the solvent interaction with the polyester could be enhanced by using microwave heating. Solvent molecules interact rapidly, not only with the surface of the fiber but also with the interior parts. Scanning electron microscope results showed that structural modifications take place, which produce surface roughness and voids; this enhances the dye uptake by threefold in comparison to conventional methods [48].

2.2.3 The Research Progress of the Physical Technology on the Polyester Fabric Dyeing

Some new physical methods were introduced to solve the problems in dyeing polyester process. Methods such as ultrasonic, low temperature plasma, supercritical carbon dioxide, microwave, microcapsules, radiation and electronic pre-illuminates are introduced on the point of ecology. Dyeing ability of polyester fiber and dyeing system is improved and quality is increased. Waste water discharge is reduced, no water or non water dyeing is realized to save energy and protect environment [49].

2.2.4 The Level Dyeing Technology of Polyester Fabric by Microwave

Microwave fixation method was used with seven solvent systems (i.e., 100% water, 10, 30, 50% urea and 10, 30, 50% DMF) in promotion of dyeing of polyester. In the respective pad-baths, the padded polyester fabrics were

exposed over a boiling water bath to maintain sufficient moisture content during irradiation. The colour strength values were used to determine the depth of shade or concentration of the dye associated with the seven dyeing methods. DMF, as a strong interacting solvent, causes plasticization of polymer chains by weakening interchain cohesive forces in the amorphous and in the ordered non-crystalline regions of the polymer. When the crystallites formed are large, the swollen polymer structure is rigid, causing formation of voids upon removal of the solvent. Dye uptake will then be greater due to the voids within the polymer increasing the internal surface area [50].

2.3 Polyamide

Many authors have investigated the feasibility of using microwaves for a variety of textile processes including heating, dyeing, finishing, fixation, printing, and drying [51-53]. This application allows some possibilities of energy conservation, reduction of processes, time and chemicals used, production increment and minimization of environmental pollution. The effect of microwave irradiation on dyeing of polyamide fibers (nylon-6) with reactive dyes was studied and the influence of microwave irradiation on the polymer characteristics was compared with conventional heating [54]. The result showed that there are increases in rate of dyeing, and dye uptake levels with high decrease in shrinkage value when using microwave if compared with traditional heating, and this decrease increased when adding alcohols. There are increases in tensile strength as well as elongation. The results show also that microwave heating has no solublizing effect on nylon-6 fabric either in presence or absence of alcohols. Influence of microwave irradiation on nylon-6 molecular weight (M.W.), intrinsic viscosity (η), degree of polymerization and end groups content ($-NH_2$, -COOH) was also evaluated. It was observed that there is an increase in terminal end groups ($-NH_2$, -COOH) with increasing boiling time, and there was also an increase when microwave irradiation was used if compared with traditional heating. However the value of intrinsic viscosity (η), molecular weight (MW) and degree of polymerization (DP) were decreased as the boiling time increased [55].

2.4 Cotton

2.4.1 Microwave Dyeing of Cotton

A recent method based on microwave dyeing of cotton and cotton/wool blend fabric with microwave irradiation to improve its dyeability with reactive dyes

was recently reported. A high increase in dye uptake and acceleration in the dye rate was observed. Microwave radiation appears to be an effective technique for dyeing cotton and cotton/wool blend fabrics and is useful in enhancing dye uptake when compared with conventional heating. From the kinetic parameter it was observed that the $t_{1/2}$ value of the dyed samples is shorter when using microwave irradiation than of those dyed by using conventional heating. Additionally, it is shorter when dyed with the two-bath method than the one-bath method; the ΔH for samples dyed with microwave irradiation is less than that of samples dyed with traditional heating. It can be observed also that the $-\Delta\mu^{\circ}$ has a negative value, which indicates that the dyeing process is an exothermic process. The results of the X-ray diffraction pattern as well as those of the scanning electron microscope indicate that the crystallinity of the fibre decreases with increasing exposure time to both types of heat, but with the certainty that the decrease is more significant with the microwave irradiation. This decrease in crystallinity is associated with an increase in fibre diameter. The value of the $t_{1/2}$ is shorter when using microwave irradiation than with conventional heating. The fastness tests for washing, rubbing, perspiration as well as fastness to light are relatively lower than their corresponding samples dyed using microwave heating [56].

The removal of natural impurities from cotton fibres using oxygen microwave low temperature plasma was studied. The influence of treating power, air pressure and time to the removal efficiency, strength and whiteness of bleaching, were evaluated and comparisons were made with conventional pre-treatment. The research results suggested that using oxygen microwave low temperature plasma in place of traditional refining technology to treat cotton knitted fabrics, the capillary effect, bursting strength, whiteness and the K/S value of dyeing are close or better than the conventional pre-treatment [57, 58].

Dyeing and fixation of cotton fabric with reactive dye using microwave was researched and compared with conventional dyeing process. Based on trials, the results showed that the dye uptake and color fastness of microwave dyed fabric were similar or higher than with conventional process. The microwave heating could shorten dye time, and save energy greatly [59].

The cotton reactive dyeing using dip-dyeing method by microwave heating was also studied. The effects of Glauber salt (Na_2SO_4), soda ash (Na_2CO_3) and liquor ratio on microwave dyeing were discussed. The results showed that cotton fabric could be smoothly dyed by reactive dyes using dip-dyeing method and under microwaves. Na_2SO_4, Na_2CO_3 and liquor ratio had effect on dye exhaustion and dye fixation, and the color fastnesses of dyed fabrics were good. The physical properties of microwave dyed fabric were also studied; the results

indicated that the tear strength and tensile strength of the fabric were improved and the flexibility of the fabric was little influenced [60].

Using pad-batch method, the effect of batching time on coloration of cotton with monochlorotriazine reactive dyes using microwave irradiation and conventional heating was investigated. The results show that microwave for short time (2 min) was better than 12-h batching time under conventional heating [61, 62].

2.4.2 Microwave Versus Conventional Dyeing of Cotton Fabrics

Microwave irradiation dyeing and conventional dyeing were compared. The results showed that the color depth, color fullness, levelness and crocking fastness of fabrics dyed by microwave irradiation method were comparative to those of conventional dyeing. The effect of microwave irradiation dyeing process on dyeing behavior was investigated through single factor experiment method: the optimal dyeing technique was obtained through orthogonal experiment: the power of microwave irradiation was 595 W, dyeing time and fixation time were 6 min and 5 min, 15g/L of sodium sulfate, 15 g/L of sodium carbonate. The crystallinity of cotton fiber was decreased under microwave irradiation, which was favorable to improve the dyeing behavior of cotton fabric [63].

2.4.3 Application of Microwave Technology in Cotton Fabric Dyeing with Reactive Dyes

The influence of different factors on dyeing depth was explored when the cotton fabric was dyed with K reactive dyes by microwave radiation, and the rubbing fastness and soaping fastness of the fabric by conventional dyeing and microwave dyeing were compared. And the effects of microwave heating on the dyeing properties of different dyes were researched. The results showed that dyeing time of cotton fabric by microwave heating could be greatly shortened, and the dyeing performance was similar to conventional dyeing process [64].

2.4.4 Saturated Steam-Assisted Radio Frequency Fixation of Reactive Printed Cotton Fabrics

Previous study declared that the development of a prototype machine for the processing of fabrics, which can be used as a reactor for the continuous finishing processes. The optimum applicator design for fabrics was developed in accordance with laboratory – scale experimental studies. Reactive printed samples were treated with the combination of radio frequency and steam in

order to take advantage of the steam energy and to improve energy absorption. The colour yields of the samples fixated with the combination of radio frequency and steam energy were compared with conventional steaming (10min) and thermofixation (5min) processes. It was observed that steam – assisted radio frequency fixation ensured similar colour yields in a shorter setting time (3min) compared with conventional methods. It was concluded that the new system could be an alternative method of setting, with the advantages of time savings and lower energy consumption [65].

2.4.5 The Use of Microwave Energy for the Fixating of Reactive Printed Cotton Fabrics

The wastes which are formed by the consumption chemical, water and energy, play an important role on the increase of environment pollution in textile industry, like other industries. Assessment of chemical and energy saving potential are of great importance in terms of costs and environmental issues. On this account in this study, application of microwave energy for the fixation of reactive printings was investigated. It was determined that the fixation of the samples could be obtained with only microwave energy without a drying process. Higher color yields were ensured with microwave fixation compared with conventional methods for the small and medium molecular sized (red and blue) reactive dyestuffs. On the other hand lower color yields occurred at microwave fixation process for the dyestuffs with big molecular size (turquoise), compared with conventional methods. The effects of the concentration of urea in the printing paste were also unvestigated and it was shown that adequate color yields could be obtained without using urea, depending on the dyestuff properties [66].

2.5 Wool

2.5.1 Dyeing of Wool Fabrics

The difficulty in dyeing of wool fibre is due to its scale-like surface structure which contains a hydrophobic lipid barrier [67]. This complex structure makes it difficult for the dye molecules to penetrate the fiber molecules, resulting in low level of the dye exhaustion. Improvement in dyeability of wool fabric by microwave treatment was investigated. The color yield of the fabric treated under various conditions, i.e. the microwave time treatment and power were evaluated. It was observed that the color yield of wool fabric was improved after microwave treatment. The treatment time and irradiation power have a greater impact on the color yield of dyed fabric. The color yield of dyed fabric

increases with increasing treatment time, but decreases with increasing power from 400W to 700 W. The longer the treatment time, the greater is the color yield of dyed fabric, as a result of the grater dyeability of treated fabric. The microwave treatment causes a slight damage on the surface scale of the wool fiber, this promoting the adsorption and penetration of dye molecules into the wool fibers as well as improving the extent of reaction between reactive dye and wool fibers. The microwave irradiation doesn't significantly affect the crystallinity and the chemical structure of wool fibers. The tensile strength increased after microwave modification [68-70].

Wool fabric was treated with microwave irradiation. The surface morphological structures and crystallinity of the untreated and microwave-treated wool were investigated with scanning electron microscopy (SEM) and X-ray diffraction (XRD).Wool was treated with microwave and then dyed with Lanasol reactive dyes and Palatin 1: 1 metal complex dyes. Adsorption behavior and diffusion coefficient were also evaluated. A higher dye uptake rate and increased diffusion coefficient of treated fibers were observed in the dyeing test, which was attributed to the damages of wool surface morphological structure under microwave irradiation [71]. Use of microwave heating for dyeing wool fabrics with palatine acid red means saving energy and time was also investigated. The optimum color strength is obtained in minutes, with good leveling and compatible fastness properties with conventional method. The scanning electron microscope examination revealed that microwave helps in physical change of microstructure of the fibre with higher dye diffusion. A kinetic investigation of the dyeing process revealed that the half dyeing time ($t_{1/2}$) of dyed wool using conventional heating was higher than ($t_{1/2}$) of dyed wool when using microwave heating. The opposite holds true for the specific dyeing rate constant (K') and diffusion coefficient (D). Comparison of the values of dye affinity ($\Delta\mu°$) and dyeing heat (ΔH) shows that the affinity of samples dyed using microwave heating is much higher than of those dyed using conductive heating. The heat of dyeing is lower in case of microwave heating; both are exothermic reaction [72].

2.5.2 Microwave Heating for Fixation of Pad-Dyeing on Wool

Microwaves are high – frequency radiation capable of producing very rapid, uniform and efficient heating in suitable materials. In particular, they can be used for the rapid heating and fixation of pad – dyeing on textile. The feasibility of applying certain reactive dyes to wool in fixation times of 30-60s has been demonstrated. Factors influencing the rates of fixation of the dyes have been investigated in some detail [73].

2.6 Silk

The possibility of dyed silk fabric using microwave irradiation was studied. The measurement results showed that when microwave radiation is applied to the treatment of silk the dye uptake, color fastness and the coloring of silk can be improved. It is very prospective when microwave treatment is combined with the traditiona1 dyeing method [74].

Study about the micropore structure of mulberry silk after being refrigerated at super-low temperature and radiated with microwave was evaluated. Through the analysis and research about the micropore morphological structure of the boiled mulberry silk fiber after being refrigerated at super low temperature and radiated with microwave, cracks in the longitudinal surface and many micropores in the horizontal cross-section due to the obvious thermal dilation during treatment appeared [75].

2.7 Application of Microwave Technology in Textile Modification

The mechanism of microwave including thermal effect and nonthermal effect and drying characteristics of microwave was reviewed. The applications of microwave technology in textile dyeing and finishing was introduced: pretreatment (degumming of refining, retting of silk fabric and bleaching), dyeing (by fabric or two mode, microwave could not be applied in dyeing of hydrophilic fiber but in hydrophobic fiber by adding appropriate auxiliaries), printing (the printed fabric was irradiated by microwave, which could prevent the water in the printing paste to outflow, and these water could be applied in the fixation and tintage on the fabric) and finishing (epoxy resin finishing, formaldehyde – free durable press finish, oil repellent and water repellent finish). It was pointed out that the applications of microwave technology in other fields including measuring humidity, drying latex of the carpet, the drying of polyester, treatment of nylon rope and microwave low temperature plasma finishing. The development trend of microwave technology was also prospected [76].

2.8 Acrylic Fiber

Microwave dyeing was carried out under a variety of conditions in terms of the power and time of a microwave to investigate the effects of microwaving on

the dyeability of acrylic fibers. The results have shown that, at low concentrations of dye, adsorption using the microwave - based procedure is higher and much faster than conventional methods, but K/S is the same around the saturation point**.** The surfaces of microwave-irradiated acrylic fibers are rougher than conventionally dyed fibers, allowing the dye molecules to permeate and adsorb into the acrylic fibers. As a result, a reaction between the dye molecules and the acrylic fibers would be more probable. Differences in tensile strength and thermal gravity parameters for fibers dyed by conventional and microwave methods are not considered to be a significant cause for concern.

Increased dyeability is related to the local overheating due to microwave irradiation [77] and the increased roughness of the fiber surface. A power of 720 W and microwave irradiation time of 14 minutes have been found to be an optimum dyeing condition for acrylic fibers, although 5 minutes using 720 W microwave irradiation is enough to obtain the same dyeability as conventional methods [78].

2.9 Polypropylene

2.9.1 Acid Dyeing of Polypropylene

A process for making a surface of a non-polar polymeric material receptive to coloration with an acid dye was studied. This study concerns processes comprising treating the non-polar polymeric surface with low temperature microwave plasma wherein a chemical compound has been added, thereby creating receptor sites for acid dye on the surface of the non-polar polymeric material. It was observed that the large volume microwave plasma generator (LMP) treatment enhances acid dyeability of non-polar polymeric material. The result of the evaluations indicates that strongly basic monomers such as amines hexamethylene diamine, acrylamine, hydrazine, acrylic acid and 1, 3-diaminopropane, produce very satisfactory dyeability. Amphoteric, weakly acid or weakly basic monomers, such as the alcohols, and amines formamide, butylamine, ammonia, heptamine, toluidine, acetonitrile and vinylpyrroline are less effective. The amine 1, 3-diaminopropane proved to be the most effective monomer for polypropylene fiber, a woven fabric, or non woven fabric. LMP technology offers a technique for uniformly depositing a layer of plasma product of as yet unspecified chemical nature (depending on the plasma monomer used) onto the surface of a non-polar polymeric material such as polypropylene fiber a woven fabric, or non woven fabric to enhance its

dyeability with acid dyes [79].

2.9.2 Disperse Dyeability of Polypropylene Fibers Via Microwave and Ultrasonic Energy

In this study, the dyeability of polypropylene fibers with a disperse dye via microwave and ultrasonic energy was examined. A dye bath having a liquor-to-goods ratio of 20:1, and including dyestuff 2% owf was prepared and dyeing experiments using microwave and ultrasonic energy were carried out. In the case of microwave dyeing, experiments have been carried out at different energy levels (L, M-L, M, M-H and H) and different time ranges (1 to 5 min) while in the case of ultrasonic energy different temperatures (20, 40, 60 and 80 degrees C) and different time range (1 min to 30 min) were used. Additionally, effect of carrier was also investigated. Dyed samples were examined by determining their K/S values. Diffusion coefficients in all methods were calculated using Shibusawa's approximation of Hill's equation. Washing fastnesses of dyed samples were also examined. This study showed that the dyeability of polypropylene fibers was increased by both microwave and ultrasonic energy [80].

2.10 Use of Microwave Fixation in Printing with Natural Color

Until recently, use of microwave in textiles is restricted to drying process. Recently, some researches have studied the feasibility of using microwaves for variety of textile processes, e.g. drying, dyeing, finishing and printing. Owing to the importance gained by microwave heating and natural dyes, researchers studied the effect of microwave fixation time, additives, and mordant in coloring wool fabric with natural dyes (Lawsone, obtained from *Henna*, *Lawsonia alba* Lam. Leaves). They observed that Lawsone (2-hydroxy-1, 4-naphthoquinone), the dye component of henna, can be employed to print the wool fabric by using microwave fixation technique. Orange color and higher K/S compared to that obtained by conventional methods are obtained without the need of drying step on using this technique. The color strength and overall fastness properties of microwave fixed samples are found to be good and the samples acquire soft handle [81].

2.11 Synthesis

2.11.1 Microwave-Assisted Synthesis of Eco-Friendly Binders from Natural Resources

The most widely used technique for printing textiles is the pigment printing. Aqueous formulations for pigments print pastes typically consist of pigment(s), a suitable thickener, binder and cross linking agents. Because the pigments have almost no affinity for the substrate, the binder and cross linking agents bind the pigments to the surface of the substrate during the heat-curing or fixation step. Binders are also responsible for the hand and many performance properties of the printed textile [82,83]. Synthesis of eco-friendly binders from natural resources using microwave irradiation to improve textile printing was studied. The prepared polymers using microwave heating at reaction time of 45-60 min ensures the formation of alkyd resins *versus* 8-10 hrs when using conventional heating. Modified alkyd resins from sunflower oil have been successfully used as binders in the formulation of pigment printing pastes. They were characterized by non-Newtonian pseudo plastic behavior, weight loss as well as water absorption. Tg of the prepared binders was to be found in the range of (+2.5 - +5.7) which indicate the glassy state of binders is typically formed by cooling to very low temperature. It was observed that the roughness and overall properties depend on the type of fabric and binder used in printing pastes. The rubbing, washing and perspiration fastness ranged from good to excellent in case of using prepared binders, while the ranges were from poor to good in case of commercial binder. The printings have excellent fastness to light. In addition, all samples show soft handle, except in commercial binder it shows harsh handle [84].

2.11.2 Microwave-Assisted Synthesis of New Polyfunctionally Substituted Arylazo-Aminopyrazoles

Synthesis of new poly functionally substituted arylazo - aminopyrazoles for utilization in printing via conventional and microwave heating was investigated. This work has been carried out to investigate the synthetic approaches of new heterocyclic azo-dyes via conventional refluxing and microwave heating. As from a sequence of reactions starting from cyanoacetic acid, 4 – arylazo - 2*H* – pyrazol – 3 - ylamines and 4-arylazo-2-phenyl-2*H*-pyrazol-3-ylamines are obtained. Structures of the obtained compounds were established with certainty via inspection of spectroscopic and analytical data. Evaluation of fastness properties and spectral data of these new disperse dyes in printing polyester fabrics were investigated. From the result we can observe that these dyes give

brown to orange-red shades on polyester fabrics showing poor to very good fastness properties [85].

2.11.3 One-Pot Synthesis of Disperse Dyes under Microwave Irradiation: Dyebath Reuse in Dyeing of Polyester Fabrics

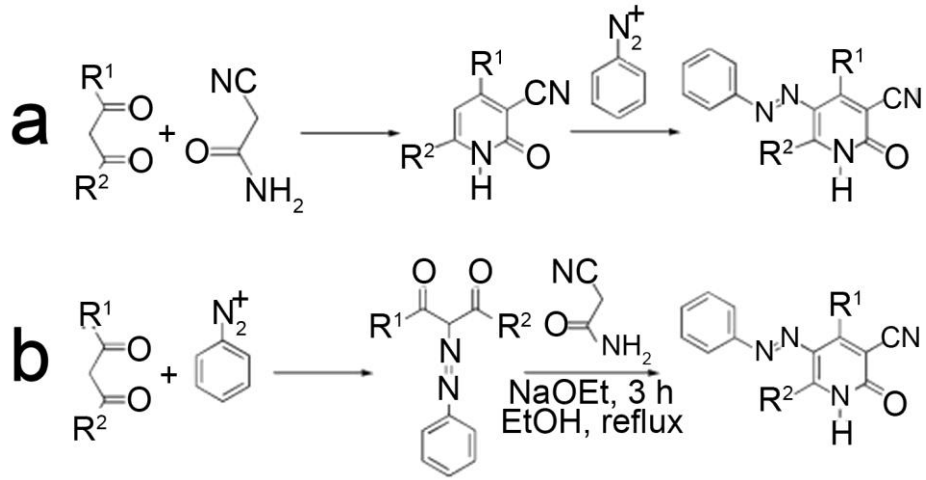
A series of 4-hydroxyphenylazopyrazopyrazolopyrimidine disperse dyes were prepared via one-pot reactions of p-hydroxyphenylhydrazone, hydrazine hydrate, and acetylacetone or anaminones using microwave irradiation as an energy source. Structural assignments of the dyes were confirmed by X-ray crystallographic structure determination. Instead of discharging the dyebath after each dyeing cycle, the residual dyebath was spectrophotometrically analyzed and then pH readjusted for a repeat dyeing with longer time. Fastness of the dyed samples was measured after each recycle. Most of the dyed fabrics tested displayed good light fastness and excellent fastness to washing and perspiration. Finally, the biological activity of the synthesized dyes against gram positive bacteria, gram negative bacteria and yeast were evaluated [86].

2.11.4 Microwave-Assisted Synthesis of 5-Arylazo-4, 6-Disubstituted-3-Cyano-2-Pyridone Dyes

Azo compounds are the largest group of colorants in terms of number and production volume of currently marketed dyes and pigments. The importance of azo compounds as colorants is due to the simplicity of their synthesis by diazotization and azo coupling, and to the almost innumerable possibilities presented by variation on the diazo compounds and coupling components, in conjunction with their generally high molar extinction coefficient and moderate / high fastness properties [87]. Arylazo colorants containing pyridone rings can be prepared from β-diketones and various diazonium salts, followed by condensation with cyanoacetamide as follows: [88-90].

The arylazo dyes obtained in such manner do not contain unreacted pyridone material and are generally obtained in higher yields. Long reaction times, the use of a toxic and strong base for the condensation step, are the other disadvantages of conventional method. The possibility of improved method for synthesizing novel, 5-arylazo-4, 6-disubstituted-3-cyano-2-pyridone dyes from β-diketones and various diazonium salts, followed by high speed microwave-assisted condensation with cyanoacetamide was recently reported. It was observed that the synthesis of an important group of azo-based dyes was rapid and efficient via use of microwave heating. The use of controlled sealed vessel microwave heating allowed the preparation of a variety of pyridone colorants in very short reaction times, and high yields, if compared with

conventional methods [90].



Scheme 1. *Synthetic methods for the preparation of arylazopyridones.*

2.12 Colour Removal

2.12.1 Microwave-Assisted Degradation of Remazol Golden Yellow Dye Wastewater as well as Enhanced Chlorine Dioxide CLO_2 Catalytic Oxidation Process

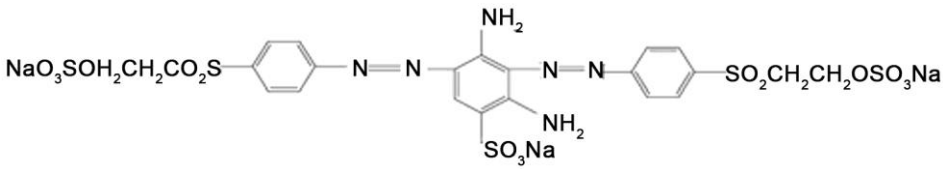


Fig. 11. *Chemical structure of remazol golden yellow RNL.*

The color and high COD of effluents from dye house cause serious environmental contamination problems nowadays. In particular, azo dyes represent about half of the dyes used in the textile industry and, as a consequence, a relevant problem of pollution related to the release of these products in the environment is taking place [91,92]. Although there were several other technologies available for the removal of color and COD from azo dye wastewater such as biodegradation [93], sorption [94-96], electrochemical and oxidative degradation [97-98], and chlorine dioxide (CLO_2) catalytic oxidation was a very attractive and useful technique for treatment of dye house effluents

[99–101]. The removal of remazol golden yellow dye in order to assess the effectiveness and feasibility of microwave enhanced chlorine dioxide (CLO_2) catalytic oxidation process as well as the operating parameters such as the CLO_2 dosage, catalyst dosage, and pH was evaluated [99-101]. The results showed that microwave-enhanced catalytic oxidation process could effectively degrade remazol golden yellow dye with low oxidant dosage in a short reaction time and extensive pH range compared to the conventional wet catalytic oxidation. Under the optimal condition (CLO_2 concentration 80 mg/L, microwave power 400W, contacting time 1.5 min, catalyst dosage 70 g/L, and pH 7), color removal efficiency approached 94.03%, corresponding to 67.92% of total organic carbon removal efficiency. It was found that the fluorescence intensity in microwave-enhanced ClO_2 catalytic oxidation system was about 500 a.u. which verified that there was much hydroxyl radical produced [99].

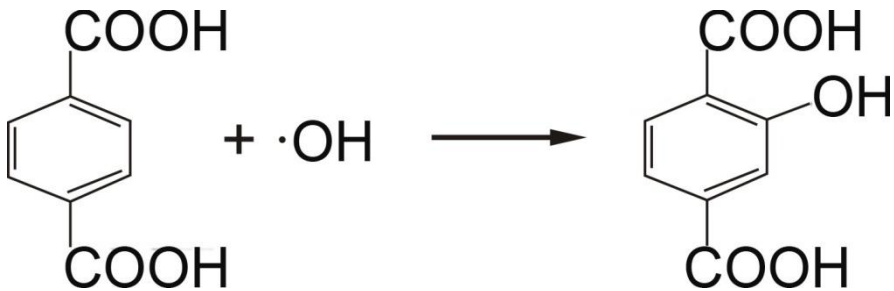


Fig. 12. *Reaction between hydroxyl radical and terephthalic acid.*

Compared with different processes, microwave enhanced ClO_2 catalytic oxidation system could significantly enhance the degradation efficiency. It provides an effective technology for dye wastewater treatment [100-104].

2.12.2 Microwave-Assisted Regeneration Process of Reactive Black 5 Treatments by Combined Electro-Coagulation-Granular Activated Carbon Adsorption

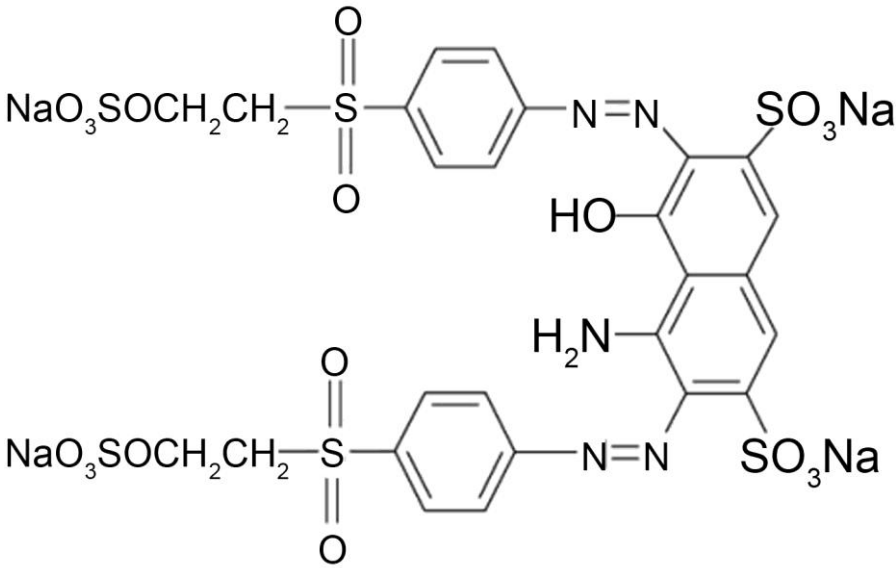


Fig. 13. *Molecular structure of C.I. Reactive Black 5 (RB5, azo dye),* λ_{max} *= 597 nm.*

Textile wastewater is a major water pollution source in developing countries and often contains high concentrations of unfixed dyes (about 20%). Azo dyes are of great concern because of their widespread use, toxic aromatic amine intermediates, and recalcitrance for aerobic wastewater treatment [105]. Several techniques have been applied to remove dyes from wastewater, including adsorption, chemical oxidation, electrochemical degradation, and advanced oxidation processes. However, their low removal abilities or high costs often limit their application [106]. Treatment of an azo dye, Reactive Black 5 (RB5) by combined electrocoagulation-activated carbon adsorption–microwave regeneration process was evaluated. The toxicity was also monitored by the *Vibrio fischeri* light inhibition test. Granular activated carbon GAC of 100 g L^{-1} sorbed 82% of RB5 (100 mgL^{-1}) within 4 h. RB5- loaded GAC was not effectively regenerated by microwave irradiation (800 W, 30 s). Electrocoagulation showed high decolorization of RB5 within 8 min at pH 7, current density of 277Am^{-2}, and NaCl of 1 gL^{-1}. However, 61% COD residue remained after treatment and toxicity was high (100% light inhibition). GAC of 20 g L^{-1} effectively removed COD and toxicity of electro coagulation-treated

solution within 4 h. Microwave irradiation effectively regenerated intermediate-loaded GAC within 30 s at power of 800 W, GAC/water ratios of 20 g L^{-1}, and pH of 7.8. The adsorption capacity of GAC for COD removal from the electro coagulation-treated solution did not significantly decrease at the first 7 cycles of adsorption/regeneration. The adsorption capacity of GAC for removal of both A_{265} (benzene-related groups) and toxicity slightly decreased after the 6^{th} cycle [107].

2.12.3 Regeneration of Acid Orange 7-Exhausted Granular Activated Carbons with Microwave Irradiation

An investigation was performed for the regeneration of three granular activated carbons (GACs) exhausted with acid orange 7 (AO7). The three GACs were made from different materials, i.e. coconut shells, almond nucleus and coal. The AO7 adsorption process was carried out in a continuous-flow adsorption column. After adsorption, the AO7-saturated GAC was dried at 120 ℃, then regenerated in a quartz reactor by 2450 MHz microwave (MW) irradiation at 850 W for 5 min. The efficacy of this procedure was analyzed by determining the rates and amounts of AO7 adsorbed in successive adsorption–MW regeneration cycles. Effects of this regeneration on the structural properties, surface chemistry and the AO7 adsorption capacities of GAC samples were examined. It was found that after several adsorption–MW regeneration cycles, the adsorption rates and capacities of GACs could maintain relatively high levels, even higher than those of virgin GACs, as indicated by AO7 breakthrough curves and adsorption isotherms. The improvement of GAC adsorption properties resulted from the modification of pore size distribution and surface chemistry by MW irradiation [108, 109].

2.12.4 Microwave-Assisted Synthesis of Titania Nanocubes, Nanospheres and Nanorods for Photocatalytic Dye Degradation

Nanomaterials of transition metal oxides have attracted a great deal of attention from researchers in various fields due to their numerous technological applications [110-113]. Among them, nanocrystalline titania has been attracting increasing attention due to its fascinating properties and potential applications. Titanium dioxide is a versatile material which is being investigated extensively due to its unique optoelectronic and photochemical properties such as high refractive index, high dielectric constant, excellent optical transmittance in the visible and near IR regions as well as its high performance as a photo catalyst for water splitting and degradation of organics [114]. With a band gap of

3.0–3.3 eV, titanium dioxide has been photo catalytically active only under ultraviolet light (wavelength k\400 nm) [115]. Titanium dioxide mainly exists in three crystalline phases: anatase, rutile, and brookite [116]. Among the three crystalline forms, anatase titanium dioxide is attracting more attention for its vital use as pigments [117], gas sensors [118], catalysts [119-120], photocatalysts [121-123] in response to its application in environmentally related problems of pollution control and photovoltaic [124]. A simple microwave method to synthesize phase pure anatase and rutile nanotitania with different morphologies *viz*., cubes, spheres, and rods was evaluated. Photocatalytic activity studies of the synthesized samples were carried out using the dye, methylene blue in aqueous solution under ultraviolet light irradiation. The photoluminescence (PL) features of the synthesized titania nanostructures were also compared in the present study. Photocatalytic dye degradation studies were conducted using methylene blue under ultraviolet light irradiation. Dye degradation ability for nanocubes was found to be superior to the spheres and the rods and can be attributed to the observed high surface area of nanocubes. As synthesized titania nanostructures have shown higher photocatalytic activity than the commercial photocatalyst Degussa P25 TiO_2 [125].

2.12.5 Microwave Enhanced-Sorption of Dyestuffs to Dual-Cation Organobentonites from Water

The microwave enhanced-sorption of dyestuffs such as Neutral Red S-BR, Neutral Dark Yellow GL and Acid Blue B onto organobentonites from water was investigated. The decolorization rates of various dyestuffs by organobentonites were increased from 18.0% to 71.8%, the saturate desorption capacity of Neutral Red S-BR and Acid Blue B were increased 83.9% and 76.3 % by microwave irradiation, respectively. The value of the microwave enhanced-sorption parameter R_m increased in the following order: Neutral Red S-BR > Acid Blue B > Neutral Dark Yellow GL, which corresponded with their aqueous solubility. The zeta potentials of particles were decreased greatly by microwave, which is very significant for improving both sorption of dyestuffs to organobentonites from water and the separation of the adsorbents from treated water [126].

Chapter 3

Microwave Pretreatment and Finishing

3.1 Analytical Solutions to the Drying of Textile Materials with Microwave Heating

An analytical model of internal transport characteristics of a textile material during drying using microwave heating was developed. The model is simplified for each period of drying (initial adjustment, liquid movement, constant rate, and falling rate) using existing experimental data and certain known characteristics of microwave heating. Solutions to these reduced modeling equations are obtained for the initial adjustment period and the constant rate period, which are the drying periods comprising the majority of the drying processes of most textile materials of interest. The analytical results are compared to the available experimental data, and a correlation is obtained in the prediction of drying rates, internal temperature profiles, and internal pressure distribution [127, 128].

3.2 Degradation of Pentachlorophenol with Zero-Valence Iron Coupled with Microwave Energy

The objective of this research was to study the degradation of pentachlorophenol with zero-valence iron (Fe^0) coupled with the use of microwave energy. The sample containing 1000 mg/L PCP solution was dosed with 0.5 g Fe^0 and then subjected to 700 W microwave energy for 10 s 85% pentachlorophenol was noted to be removed. If the microwave treatment time was increased to 30 s, the pentachlorophenol removal efficiency exceeded 99% with end products including H_2O, CO_2, HCl, etc. Using Fe^0 as a medium, the microwave treatment is made an efficient method for degrading pentachlorophenol. The time needed to achieve a satisfactory treatment is also reduced leading to significant savings of energy consumption to make this method cost-effective. Since this technology applies Fe^0, which is amenable to natural environment, to speed up the decomposition of an industrial solvent, it is not only cost-effective but also environmentally friendly for the industry to pursuit sustainable development [129].

3.3 Degumming of Silk Using Microwave-Assisted Treatments

A comparative study was carried out between degumming of raw silk yarns

by the conventional methods of degumming and those assisted by microwave radiation. Different reagents are used for this study; *viz*., mineral acid, alkaline substances, domestic soap, and commercial protease. The proteolytic enzyme savinase 16L type EX was found to be effective in degumming of silk either by conventional method or in combination with microwave. The microwave irradiation reduces, to a great extent, the time needed to reach the same degree of degumming by the conventional method. The effect of the aforementioned treatments on some of the inherent properties of silk was monitored. Scanning electron microscopic investigation was carried out to clarify the effect of these treatments on the morphological structure of silk yarns [130].

3.4 Development of Catalyst Materials being Effective for Microwave Sterilization

Recently, airborne virus infections have emerged as some of the most challenging medical problems. To prevent the threat of infection, the processes of sterilization have been studied widely. Microwave sterilization has many advantages in comparison with conventional methods. It is able to raise the temperature of a material in a short time and selectively heat the material. This results in the reduction of usage and the rapid completion of sterilization. We developed a novel microwave sterilization system that can raise the temperature in quite a short time using a lower microwave power (100 W). Filters made with Kao wool (Al_2O_3) were coated with TiO_2 (anatase) by sol–gel method and used to trap microorganisms. In addition, these filters were coated with Pt or Ag by impregnated method. We also prepared a Tyranno-fiber textile filter and a honeycomb SiC filter. Two microorganisms, *Bacillus subtilis* ATCC 9372 and *Bacillus stearothermophilus* ATCC 7953, were used in this experiment, where either microorganism was loaded onto a filter. After irradiation, filters loaded with *B. subtilis* and *B. stearothermophilus* were incubated for 48 h in a TSB medium (BactoeTryptic Soy Broth, Becton-Dickinson, MD) at 37 and 56 ℃, respectively. *B. subtilis* and *B. stearothermophilus* loaded on to Ag-impregnated filters were sterilized in 30 and 15 s, respectively. The Tyranno-fiber textile filter and the honeycomb SiC filter also showed effective microwave sterilization. These results showed that this system could sterilize *B. subtilis* and *B. stearothermophilus* in quite a short time and that microwave-absorbable materials are effective as microwave sterilization filters [131, 132].

3.5 Dissolution of Cellulose with NMMO by Microwave Heating

Environmentally-friendly microwave heating process was applied to the dissolution of cellulose in N-methylmorpholine- N-oxide (NMMO) with 105–490 W and 2450 MHz microwave energy until the dissolution completed. Microwave heating caused a decrease in the dissolution time and energy consumption. Cellulose/NMMO/water solutions with different cellulose concentrations were converted to the membrane to measure the crystallinity and degree of polymerization. It was shown that microwave heating with the power of 210 W is an alternative heating system for dissolution of cellulose in NMMO. The membranes obtained with two different heating methods showed the same crystallinity and degree of polymerization. As a result, microwave heating has an advantage in shortening reaction times, compared to conventional heating [133].

3.6 Effect of Microwave Irradiation on the Physical Properties and Morphological Structures of Cotton Cellulose

Microwave heating has been proved to be more rapid, uniform and efficient, and easily penetrate to particle inside. To investigate the effect of microwave irradiation on the physical property and morphological structure of cotton cellulose, cellulose fabric was treated with microwave irradiation at different conditions. The physical properties of the treated cellulose fabric were investigated. The morphological structures and thermal stabilities of the untreated and treated cellulose were investigated with differential scanning calorimetry (DSC) and X-ray diffraction. The results show that the physical properties of the treated cellulose fabrics were improved and the recoverability had not significant changed. The thermal stability of the treated cellulose was changed. The crystallinity and preferred orientation of the treated cotton cellulose increased [134].

3.7 Functionalization of Cotton Fabric with Vinyltrimethoxysilane

The surface of cotton fabric was successfully functionalized with vinyltrimethoxysilane in order to impart water repellency and wrinkle recovery

and to introduce surface vinyl groups (–CH=CH_2) to the fabric, which could then be initiated for copolymerization reactions with various monomers. The introduction of active groups onto the fabric surface was evidenced from the universal attenuated total reflectance Fourier transform infrared (UATR-FTIR) spectrum of the treated fabric. The spectrum shows two peaks located at 1410 and 1600 cm^{-1} (C=C stretch). An additional peak located at 756 cm^{-1} attributed to Si–O–Si symmetric stretch was also observed. Excellent water contact angle and wrinkle recovery angle values were obtained [135].

3.8 ICP-OES Determination of Metals Present in Textile Materials

The content of elements present in textile materials was assessed since it is known that textiles containing metals may represent a health hazard to consumers. Determination of metal content can be also useful to the textile industry since some metals present in textiles may contribute to problems during textile production. Extraction of metals from different textile materials was performed in an artificial acidic sweat solution according to the Öko Tex standard for materials coming into direct contact with the skin. After extraction from textile products made of cotton, flax, wool, silk, viscose, and polyester materials, all elements were determined by means of inductively coupled plasmaoptical emission spectrometry (ICP-OES). Results in the sweat extracts (minimum–maximum in μg/mL) were: Al 0.11–1.58, Cd 0.02–0.05, Cr 0.01–0.32, Cu 0.05–1.95, Mn 0.01–2.17, and Ni 0.05–0.10. Concentrations of other elements were below detection limits. The total amount of metals present was determined after microwave-assisted acidic digestion of textile materials with 7 M nitric acid. According to the results, the majority of the detected elements were below the concentration limits given by the Öko Tex, and for this reason the textile materials investigated do not represent a health hazard to consumers [136].

3.9 Influence of Microwaves on Nonformaldehyde DP Finished Dyed Cotton Fabrics

An alternative approach to formaldehyde-releasing conventional N-methylol compounds is based on the use of non-formaldehyde durable press polycarboxlic acid (PCA) finishing agents. Another alternative approach, investigated here, is using microwave energy to impart durable crease resistance to dyed cotton fabric. The bifunctional dyes C.I. Reactive Red 195, C.I.

Reactive Yellow 145, and C.I. Reactive Blue 221 were used in the study, and the isocratic HPLC method was employed to quantify the PCA reacted with the cellulosic material for two different curing procedures. Shade change evaluation revealed that microwave curing has a greater influence on the d*E* values than conventional curing. In all other aspects, primarily wrinkle recovery and deformation resistance, microwave curing offers much better results [137].

3.10 Microwave Curing for Producing Cotton Fabrics with Easy Care and Antibacterial Properties

A new microwave curing system was used to affect crosslinking of cotton fabric with non-formaldehyde finishes, namely, glyoxal, glutaraldehyde and 1,2,3,4 butanetetracarboxylic acid (BTCA). Water soluble chitosan was incorporated in the finishing bath in order to impart antibacterial activity to the fabric in addition to the ease of care characteristics. Glyoxal proved to be the best finish and, hence, it was studied along with the chitosan under a variety of conditions including chitosan concentrations, power and time of microwave curing. Besides the crease recovery and strength properties of the finished fabrics, the latter were also monitored for N%, antibacterial activity and characterized using scanning electron microscope (SEM) and FTIR spectra when compared. With conventional curing system, the microwave curing system was found advantageous in production of cotton fabrics with easy care antibacterial properties without high losses in strength properties [138].

3.11 Microwave Properties of Conductive Polymers

Conductive polymers are a new class of microwave absorbing materials which show a number of advantages over traditional granular materials. Polypyrrole, Polyaniline, and Polyalkylthiophenes can be applied in specific fields where the conductive inclusion is directly integrated in the matrix or on the substrate (honeycomb, textile) during synthesis, instead of being mechanically dispersed as in the case of extrinsic conductive materials. This method can be used to produce materials with specific properties, whose performances are equivalent to those of magnetic materials but with lower surface mass. The properties of these materials can be easily modified by chemical means and by tailoring the structural properties. We show that dielectric properties strongly depend on the microstructure of conductive polymer. For that purpose, the influence of the molecular weight, density of defects, size of the alkyl chain on the substituted monomer and nature of

counter anion have been explored. Theoretical models using physicochemical properties of polymer have been developed in order to calculate the frequency dependence of (E', E'') for a chain of Polyaniline [139].

3.12 Microwave Sanitization of Polyester and Cotton

The potential of using 2450-MHz microwave radiation to dry and sterilize polyester and cotton fabrics was evaluated against *Staphylococcus aureus*, *Escherichia coli* and *Bacillus cereus*. *B. cereus* were the most tolerant microoganisms to microwave radiation, and *E. coli* were the most senstive. All *S. aureus* and *E. coli* organisms were killed within seven minutes of exposure to microwave drying, which was substantially more effective than convection oven drying. Microwave exposure had no effect on the elongation of polyester and cotton, but cottons strength decreased 10% after five minutes of microwave exposure [140].

3.13 Microwave Synthesized Chitosan-Graft-Poly (Methylmethacrylate): An Efficient Zn^{+2} Ion Binder

Microwave-promoted grafting of methyl methacrylate onto the chitosan has been optimized. Chitosan-graft-poly(methylmethacrylate) (Ch-g-PMMA) could be synthesized with 160% grafting using 80% MW power in 2 min at (MMA) 0.17 M, (Chitosan) 0.1 g/25 ml. The representative graft copolymer was characterized by Fourier transform-infrared, thermo gravimetric analysis and X-ray diffraction measurement, taking chitosan as reference. The effect of reaction variables as monomer/chitosan concentration, microwave power and exposure time on the graft co-polymerization was studied. A probable mechanism for grafting under microwave heating has been proposed. Viscosity of the grafted chitosan solutions and water/saline retention for the grafted chitosans were determined and compared with that of the chitosan. The microwave synthesized graft copolymer was found to have efficient adsorption ability for Zn_2C ions in aqueous solution. Effect of pH and Zn_2C concentration on adsorption was also studied [141].

3.14 Modern Applications of Nanotechnology in Textiles

Nanotechnology (NT) deals with materials 1 to 100 nm in length. At the National Nanotechnology Initiative (NNI), NT is defined as the understanding,

manipulation, and control of matter at the above-stated length, such that the physical, chemical, and biological properties of the materials (individual atoms, molecules, and bulk matter) can be engineered, synthesized, and altered to develop the next generation of improved materials, devices, structures, and systems. NT at the molecular level can be used to develop desired textile characteristics, such as high tensile strength, unique surface structure, soft hand, durability, water repellency, fire retardancy, antimicrobial properties, and the like. Indeed, advances in NT have created enormous opportunities and challenges for the textile industry, including the cotton industry. The focus of this paper is to summarize recent applications of NT as they relate to textile fibers, yarns, and fabrics [142].

3.15 Optimization of Ultrasonic Extraction of 23 Elements from Cotton

Optimization of ultrasonic extraction of 23 elements from cotton was performed with different solvent volume ratios. For this purpose nitric acid, hydrochloric acid and water were mixed and applied in a mixture for the extraction of elements adsorbed on cotton material. The elements chosen for the extraction procedure (Al, As, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Si, Sn, Tl and Zn) were those that are important in textile processing. Some of them cause problems during fiber processing, dyeing or bleaching. The removal of elements from the processed fabric can be successfully done with ultrasonic extraction in the ultrasonic bath. Extraction procedure was optimized by software package Design Expert 6 (DX6) and the optimum of ultrasonic extraction was achieved with the mixture of 1 M HCl – 1 M HNO_3 – H_2O = 3.32/2.83/93.85 (v/v). Ultrasonic extraction was a fast and efficient extraction procedure easily applied on cotton textile material [143].

3.16 Oxidative Decomposition of Azo Dye C.I. Acid Orange 7 (AO7) under Microwave Electrodeless Lamp Irradiation in the Presence of H_2O_2

A novel microwave electrodeless lamp (MWL) rather than traditional electrode lamp (TEL) was used in a H_2O_2/MWL system as light source. This technique provided a new way to study the simultaneous effect of both UV–vis light and microwave irradiations. This study showed that H_2O_2/MWL process was 32% more effective than H_2O_2/TEL process in degrading azo dye Acid

Orange 7 (AO7). Further study found that the degradation of AO7 by the H_2O_2/MWL process was initiated by the attack of HO• radicals generated by the photolysis of H_2O_2. However, the direct photolysis of AO7 by MWL irradiation was not negligible. Effect of operation parameters, such as the initial concentrations of AO7 and H_2O_2 and pH, were investigated. A kinetic model of degradation of AO7 by H_2O_2/MWL process was found, in which not only the HO• oxidation but also direct photolysis were considered. The kinetic model was consistent with the experiment results. The degradation of AO7 by H_2O_2/MWL corresponded to a pseudo-first order reaction. The apparent reaction constant (k_{ap}) was a function of initial concentrations of H_2O_2 and AO7 and pH of the solution [144].

3.17 Preparation of TiO_2-Pillared Montmorillonite as Photocatalyst Part II Photocatalytic Degradation of a Textile Azo Dye

Use of a photocatalyst based on TiO_2-pillared montmorillonite prepared by microwave has been studied with respect to photodegradation of Solophenyl red 3BL, an azo dye produced from textile plants. Experiments were carried out with an aqueous dye concentration of 100 mg L^{-1}, at different pH and photocatalyst contents (400–5000 mg L^{-1}). The lower the pH, the higher the dye photodegradation rate constants: 0.0966 and 0.0006 min^{-1}, obtained at pH=2.5 and pH=11 respectively. This is in agreement with a higher adsorption of the dye on the catalyst at acidic rather than at basic pH. At pH=5.8 and 2.5 g L^{-1} of TiO_2- pillared montmorillonite, initial degradation rate constant of the dye (0.0096 min^{-1}) was 1.4-fold lower with the synthesized photocatalyst than with commercial Degussa P25 (1 g L^{-1}) for about the same molar amount of TiO_2 in dispersion. According to XRD patterns, rate constants were related to the rate of TiO_2 crystallization within the catalyst. A photosensitized oxidation accounted for about 25±5%, and a direct excitation of the photocatalyst accounted for 80±10% of the global photo degradation process, leading mainly to oxidation of the dye by HO °[145].

3.18 Properties and Applications of Conductive Textiles

A novel technique for the chemical analysis of polypyrrole on textiles, particularly the degree of doping, is described. In addition, we discuss characterization of the electrical properties, including microwave properties,

and the environmental stability of these composite structures as they relate to different types of doping agents. Applications of these useful composite structures will also be described [146].

3.19 Single-End Sizing of Yarn using a Slot Applicator

A single-end sizing process was developed to eliminate the problems associated with the traditional sizing method. By keeping the yarns separated in slots in the single-end sizing apparatus and drying them individually, less damage to the yarns occurred. In this paper, the process and device developed for single-end sizing are described and the properties of single-end sized yarn are compared with unsized yarn and commercially sized yarn [147].

3.20 Spectroscopic Characterization of Zinc Oxide Nanorods Synthesized by Solid-State Reaction

Well-crystallized zinc oxide nanorods have been fabricated by single step solid-state reaction using zinc acetate and sodium hydroxide, at room temperature. The sodium lauryl sulfate (SLS) stabilized zinc oxide nanorods were characterized by using X-ray diffraction, Fourier transform infrared spectroscopy, transmission electron microscopy and photoluminescence spectroscopy. The X-ray diffraction revealed the wurtzite structure of zinc oxide. The size estimation by XRD and TEM confirmed that the ZnO nanorods are made of single crystals. The growth of zinc oxide crystals into rod shape was found to be closely related to its hexagonal nature. The mass ratio of SLS:ZnO in the nanorods was found to be 1:10 based on the thermogravimetric analysis. Blue shift of photoluminescence emission was noticed in the ZnO nanorods when compared to that of ZnO bulk. FT-IR analysis confirmed the binding of SLS with ZnO nanorods. Apart from ease of preparation, this method has the advantage of eco-friendliness since the solvent and other harmful chemicals were eliminated in the synthesis protocol [148].

3.21 The Effect of Microwave Drying on Warp Sizing

A paper describes microwave usage in drying the size pick-up was presented. An analysis of three different drying processes in relation to physical-mechanical properties of sized yarn has been carried out. Under the same sizing conditions, but different drying methods, the following parameters

were determined: breaking force, elongation at break, abrasion resistance and yarn hairiness. The application of microwave drying method for warp sizing showed to be equally good, or even better in some cases, compared to the conduct and convection drying methods [149].

3.22 The Effect of Microwave Radiation on the Cell Genome

Cultured V79 Chinese hamster cells were exposed to continuous radiation, frequency 7.7 GHz, power density 30 mW/cm^2 for 15, 30, and 60 min. The parameters investigated were the incorporation of [^{3}H] thymidine and the frequency of chromosome aberrations. Data obtained by 2 methods (the incorporation of [^{3}H] thymidine into DNA and autoradiography) showed that the inhibition of [^{3}H] thymidine incorporation took place by complete prevention of DNA from entering into the S phase. The normal rate of incorporation of [^{3}H] thymidine was recovered within 1 generation cycle of V79 cells. Mutagenic tests performed concurrently showed that even DNA macromolecules were involved in the process. In comparison with the control samples there was a higher frequency of specific chromosome lesions in cells that had been irradiated. Results discussed in this study suggest that microwave radiation causes changes in the synthesis as well as in the structure of DNA molecules [150].

3.23 Thermal Analysis in the Cellulose, Paper and Textile Industry

The present paper reports the main results presented at the 8th ICTA Conference on the thermal analysis of the cellulose, paper and textile industry. An attempt was made to classify the presentations. Thermo-destruction studies on cellulose allow the thermo-oxidative effect of various ions to be classified. Experimental results give evidence that boron-containing cellulose fibres present a different combustibility mechanism than phosphorus-containing antipyrenes. The relaxation and phase transition of cellulose are evidenced by DTA and calorimetry and a classification of plasticizers is made. An original clamping technique for TMA is presented to study very thin paper samples. Interactions between dyes and fibers are analyzed and a kinetic model, expressing sorbed dye rate, is proposed. The use of microwaves in the textile industry is described for polymerization, thermal treatment and dyeing. It appears that the use of microwaves gives a very uniform dye migration with a

50 times higher kinetic rate for acrylic compounds and 200 times higher for viscose [151].

3.24 Technique to Reduce Water and Energy Consumption in Polyester Dyeing

Polyester fibers require high amounts of water and energy for dyeing. An overview of techniques developed to reduce water and energy consumption addresses dyeing with microwave heating, rapid dyeing systems, solvent dyeing systems, foam dyeing, pad-store-wash methods, microencapsulate dyeing, electrostatic powder spraying, microemulsion dyeing, ultrasonic dyeing, alkaline dyeing systems, and supercritical fluid dyeing. Although many techniques have lower water and energy requirements, further research is necessary to render them commercially successful [152].

3.25 Microwave Effect

Researchers studied the effect of microwaves on the thermal and molecular stabilization of thermoplastic fibers. Microwaves without a Lossy (material that absorbs microwaves) acted on polyethylene terephthalate partially oriented yarn (PET-POY), resulting in higher orientation angles and a marginal improvement in the crystalline index. The presence of Lossy resulted in a substantial increase in the X-ray order factor and a decrease in the orientation angle. Even in the presence of Lossy, the birefringence level never reached the desired level of orientation expected from drawn and heat set yarns. Higher exposure times did not significantly affect fiber morphology. The cold crystallization phenomena was conspicuously absent in samples subjected to microwave radiation in the presence of Lossy [153].

3.26 Molecular Magic with Microwaves

Although microwave heating is used in such processes as tempering meat, cooking bacon, drying potato chips, vulcanizing rubber, and drying pharmaceutical compounds, the technology is used much less frequently than radio frequency and conventional gas and electric heating. At the First World Congress on Microwave Processing, held in January 1997, 330 scientists, engineers, and executives exchanged information on potential new uses for microwave technology in such areas as organic synthesis, materials processing,

and waste remediation. Ajay Bose of the Stevens Institute of Technology said that microwave-assisted chemistry could revolutionize the pharmaceutical and biotechnology industries by reducing the use of solvents and chromatographic materials, and by increasing the precision of heating processes. Rajender S. Varma of Sam Houston State University in Texas showed how microwaves make organic synthesis simpler and more environmentally friendly. IBM uses microwave technology to impregnate glass fabric for circuit boards preparation [154].

3.27 Wrinkle-Resistant Advances

Wrinkle-resistant, 100 percent cotton fabrics are expanding into new markets. Sales for these fabrics, which were originally used in men's slacks and shirts, have grown at a phenomenal pace. Sales of 100 percent wrinkle-resistant slacks, for example, grew by 15 million pairs in 1995. Major players in the men's slacks category now include Haggar, Levi Strauss, Farah, Lee Casuals, Thomson, and Duck Head Apparel. Product development soon extended the wrinkle-resistant label to shirtings and, more recently, to women's wear, sheets, and wool suits. Wrinkle-resistant technology involves a precure and a postcure process, but each manufacturer uses a proprietary process, and changes and modifications to the basic technology are frequent. Presset of Marietta, Georgia, is developing a microwave curing process that is promising because it causes less damage than conventional methods [155].

3.28 Microwave Processing of Nonwovens: An Introduction

An overview of the use of microwaves for heating and drying focuses on the interactions of microwaves with materials, applicator designs, and potential applications of microwaves in the nonwoven fabric industry. Currently, the industry does not use microwaves for any type of processing. In their interactions with materials, microwaves are novel in that heat is generated within the material itself and in that different materials heat to different extents (i.e. microwaves are selective). These properties necessitate significant modifications in both processing and equipment. The design of the applicator, which is the component in which the powers of the microwaves are applied to the material, is critical to microwave power conversion efficiency. Microwaves have potential applications in nonwoven fabric manufacture, particularly where the advantages of faster processing, reduced power consumption, selective

heating, reduced space or cooling requirements, and more uniform heating can be exploited [156].

3.29 Microwave Absorption by Textiles

Courtaulds is researching microwave absorption by textiles with the aim of reducing their reflectance of radiation to between 94 and 600 GHz. These textiles could be used in the manufacture of protective apparel or covers for vehicles and weapons that would be difficult to detect. Polyolefins, viscose, or acrylic containing carbon, iron, and/or ferrite particles are woven into fabrics with a rough surface and with a thickness of greater than 0.5 millimeters. The wavelength of the surface roughness must be between 1 and 30 millimeters and must be comparable to the wavelength of microwave radiation likely to be encountered. An organic liquid finish is capable of contributing to the reduction of microwave reflection from the surface [157].

3.30 Microwave Processes for the Combined Desizing, Scouring, and Bleaching of Grey Cotton Fabrics

The article focuses on the study to shorten the time of the combined pretreatment process on cotton fabrics by using microwave energy. The use of microwave energy led to rapid drying and resulted in a loss of strength and whiteness. Another problem observed in the microwave processes was the degree of desizing. A prepared peroxidisulfate salt and Leonil EB was used to increase the degree of desizing. A pre-steaming process was applied before the microwave period [158].

3.31 Usage of Microwave Energy in Textile Production Sector

Application areas of microwave energy, which has been used for many years in science and technology, are increasing. The studies about the usage in textile industry are recent and open to improvements. It is known that usage of microwave energy provides advantages in whole processes in textile industry like pre-treating, dyeing, bare finishing and drying. In this study microwave energy in Turkish textile finishing applications are researched and its advantages, process efficiencies and contributions to energy gain are given. Microwave energy has not been efficiently used in Turkish textile sector.

Promotion of microwave technology is expected to contribute to Turkey's economy especially in terms of energy and time conservation.

The use of MW energy is more efficient than conventional method. Extending the use of microwave technology in textile sector will benefit the industry in terms of time and energy and hence the production cost. Studies addressing the use of MW in textile sector show an increasing trend. Various institutions, organizations and associations can help reduce energy consumption and the corresponding costs in Turkish economy and hence increase its competitiveness, through increasing the awareness of their members and investors concerning the advantages of microwave technology [159].

3.32 Microwave-Promoted Rapid Curing Reaction of Phenolic Fibers

The reaction of the as-spun fiber derived from melt spinning of a novolac resin with a solution of formaldehyde and hydrochloric acid was carried out under microwave irradiation by controlling the heating rate from room temperature up to the boiling point (103 ℃). The homogeneous highly crosslinked phenolic fiber with the maximum tensile strength and the maximum dynamic modulus of 139 MPa and 2970 MPa, respectively, was obtained at 1.2 ℃ min^{-1} in 86 min. The fiber has a similar tensile strength, dynamic modulus and crosslinking degree with the fiber derived from the conventional reflux method at heating rate of 0.20 ℃ min^{-1} in 8 h. The results suggest that the microwave irradiation promotes not only the diffusion of CH_2OH from the skin into the inner layer of the fiber but also the reaction of CH_2OH with the phenolic ring in a suitable range. During pyrolysis the increase of crosslinking degree in the phenolic fibers diminishes the formation of low molecular weight compounds generated from the unstable terminal groups and inner units, while it promotes the formation of graphite layers [160].

3.33 Polypyrrole Coated Textiles

Using an infrared camera, thermal mapping of microwave irradiated conducting polymer textiles showed a detectable temperature increase in all PPy-pTSA coated nylon–lycra textiles, irrespective of polymerization time, dopant concentration, and choice of dopant or irradiation frequency. We observed a wide-ranging modulation of microwave reflection, transmission and absorption with dopant concentration and polymerization time. As expected,

uncoated samples were transparent to microwave radiation and showed no increase in temperature upon irradiation. Lightly doped samples had high transmission whereas highly doped samples were highly reflective. Polymerization time affected the variation of R, T, A in the same manner. The increase in temperature upon microwave irradiation demonstrated that the conducting polymers absorbed the microwave radiation and it was possible to produce temperature maps of this absorption. For polymerization times up to around 15 min microwave transmission was high. The absorption of incident radiation, and subsequent temperature increase in the sample was pronounced at polymerization times between 30 and 120 min.

For longer polymerization times of 180 and 300 min the absorption levels were still relatively high, though the increased levels of reflection allowed less temperature increase in the sample. On the other hand, even very small amounts of dopant increased the maximum temperature achieved on irradiation significantly. This may be attributed to increased levels of interaction of microwaves with the charged dopant anions. The maximum temperature difference of around 4℃ in the conducting fabrics relative to ambient temperature was observed in samples having 48% absorption and 26.5±4% reflection. Samples polymerized for 60 or 120 min with a dopant concentration of 0.018 mol/l or polymerized for 180 min with a dopant concentration of 0.009 mol/l yielded optimum absorption [161].

3.34 Curing of Polymers and Composites by Microwave Energy

Studies on application of microwaves to cure polymers and composites and the hitherto were presented. It is generally agreed upon that microwaves as an alternative energy source offer various advantages over thermal cure. These include a more rapid curing time, higher efficiency and production rate, and more uniform heating. However, the reported studies are largely qualitative or semi-quantitative and focused primarily on changes in temperature and absorbed power in the sample during cure. No fundamental study of the interaction between the electromagnetic waves and the organic material during cure on the molecular level has been reported. It is clear from this review that we need an understanding of how the polymer network forms in the microwave field, how it differs from the thermally cured network and what are its mechanical/physical properties. At the Polytechnic University we have embarked upon a comprehensive program aimed at modeling the formation of thermoset networks in the electromagnetic field from first principles. Our study will focus upon

experimental and theoretical aspects of chemorheology of cure by microwaves and its subsequent effect on physical/mechanical properties [162].

3.35 Antimicrobial Properties of Cotton Medical Textiles

The antibacterial and antifungal activity of antimicrobial finishes based on citric acid on cotton medical textiles was examined. The ability to effectively reduce the number of gram-negative, gram-positive bacteria and yeast was evaluated, specifically comparing the antibacterial activity after two different drying/curing methods. Citric acid (CA) and diethyl–tetradecyl–[3–(trimethoxysilyl)-propyl] ammonium chloride (Quat) were used for hygiene and disinfection purposes of medical textiles in this study. It was applied by pad-dry process and its fixation to cellulose hydroxyls was enhanced either by high curing temperatures or microwaves (MW). Determination of antibacterial activity of finished products was performed according to ISO 20743:2007 standard before the washing and after the 10 washing cycles. Antibacterial activity was tested on gram-negative bacteria *Escherichia coli*, gram-positive *Staphylococcus aureus* and yeast *Candida albicans*. Obtained results are confirming the possibility of eco-friendly CA application, for the purpose of antimicrobial finishing of cotton medical textiles. Prevention of nosocomial infections with the citric acid is possible using both curing methods (convection and microwave) and furthermore, the treatment is durable up to 10 washing cycles. Citric acid, as one of the suitable active substances, is crosslinked to the cellulose hydroxyls by the formation of ester linkages. Its antimicrobial effectiveness against the chosen microorganisms proved to be the best against *S. aureus*. Applied finish bath has additional crease proof effectiveness, providing sufficient both antimicrobial and crease proof effectiveness, so as the durability against 10 washing cycles [163].

3.36 Eradication of Insects from Wool Textile

Museum personnel, private collectors, and university staff responsible for maintaining textile collections frequently are required to make decisions or are asked questions pertaining to insect control on wool textile. Decision making in this area becomes increasingly difficult with new developments in chemical pesticides and alternative methods of eradication. It is the intent of this paper to present an overview of chemical and nonchemical methods of pest control applicable to museum and home use. The discussion of chemical insecticides is not limited to those EPA-approved for museums, institutions, or public

buildings because many questions concerning fabric pest control come from private collectors. Furthermore, some of the less toxic insecticides approved for home use may eventually be approved for use in museums and other public buildings.

In addition to common methods of controlling insect growth on wool textiles, several unusual and alternative chemical, biological, and nonchemical methods of pest control are discussed below. These may serve as an inspiration for future research. Even though this paper focuses on elimination methods for controlling fabric pests, preventive measures (i.e., good housekeeping practices and periodic inspection of wool, feathers, furs, etc.) can reduce the risk of introducing insect populations onto dwellings and storage areas or providing conditions that favor insect development [164, 165].

3.37 Microwave Radiations for Heat-Setting of Polyester Fibers

The use of radio and microwave frequency is gaining importance for industrial applications such as heating, drying, and other processing. The most important advantage of using microwave is that it is non-contact or localized heating and the heat is produced within the material. This can be much more effective than indirect heating where the heat propagation is by heat conduction through the material. We have been investigating the influence of microwave radiation on different fibers for the last few years. In the present investigation we used microwave frequency of 2450 MHz to investigate its effect on polyester fibers. The polyester fibers were heat set in air as well as a liquid, which acted as lossy substances. The liquid was chosen on the basis of earlier experiments, which showed the maximum effect. A comparative study was also carried out rushing conventional heating in silicone oil. Using the method of X-ray diffraction (XRD) we calculated the changes in % crystallinity and orientation. It was found that as the time of treatment under microwave radiation increased from 15 sec. to 120 sec. the order factor was found to increase from 0.32 to 0.71. The crystalline orientation as determined from the azimuthal scan was also found to increase. Such structural changes can be highly beneficial for the processing of fabric in industry. The microwave radiation process is fast, reliable and energy saving [166].

3.38 Microwave Irradiation Technique to Enhance Protein Fiber Properties

Microwave irradiation technique was used for the chemical modification and grafting of protein fibrous materials, such as domestic silk (*Bombyx mori*), tussah silk (*Antheraea pernyi*), and wool fibers. Epoxide compounds denacol EX810 and EX313 reacted effectively with the protein substrates. As alkali catalysts, sodium hydroxide was more effective than sodium thiocyanate. The optimum concentration was 0.25 w%. Weight gain values up to 8% were attained with 10-15 min irradiation time at 200 W power. Graftcoppymerisation of vinyl monomers onto protein fiber resulted in variable weight gains, depending on the kind of fiber, the grafting monomer used, and the concentration of the padding solution. For example, after grafting with iso – propyl methacrylate (IPMA), the weight gain of fibrous proteins took place in the following order: *Bombyx mori* silk > tussah silk > wool. *Bombyx mori* silk gained more weight with IPMA than with 2- hydroxyehtyl methacrylate (HEMA) or methacrylamide (MAA). The weight gain of *Bombyx mori* silk with HEMA significantly increased when the initial monomer concentration was raised to 400% owf, reaching a maximum value of 40%. The tensile properties of the protein fibres grafted with IPMA, MAA, and HEMA remaind unchanged or slightly improved compared to the reference fibres. Fibers modified with epoxides showed a drop in tensile performance. The surface morphology of fibres treated with Epoxide compounds or graft – copolymerized with vinyl monomers was almost unaffected, with the exception of HEMA – grafted fibers, which showed the presence of homopolymer deposited onto the surface at a weight gain exceeding 20 % [167].

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