New applications of microwave energy in textiles

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Received: October 10, 2024
Accepted: November 30, 2024
UDC 677.021.15

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Review

The paper presents the impact of microwaves in textile finishing, focusing on the application for the processing of biomass, which is becoming an increasingly important source of various raw materials, including cellulosic fibres. Various processes utilising microwave energy are used more frequently in different sectors, as they offer significant advantages due to their environmental and economic benefits. One of such succesfull applications is the use of cold argon plasma, which improves the change in the structure of lignocellulose fibres. Recently, microwave energy is starting to be used more often in waste recycling procedures, and examples from practice are given in the paper. In the final part of the paper, guidelines and recommendations are given that can contribute to the faster introduction of this green technology in the extremely important sector of textiles, clothing, leather and footwear.

Keywords: *microwave energy; textile finishing; lignocellulosic biomass; cellulose fiber isolation*

Pregled

Nove primjene mikrovalne energije u tekstilstvu

U radu je prikazan utjecaj mikrovalova u oplemenjivanju tekstila, a naglasak je stavljen na njihovu primjenu za obradu biomase koja postaje sve značajniji izvor različitih sirovina pa tako i celuloznih vlakana. Raznovrsne obrade koje primjenjuju mikrovalnu energiju dobivaju sve veći značaj i počinju se sve više upotrebljavati u vrlo raznovrsnim sektorima jer imaju značajnu ekološku i ekonomsku prednost. Kao jedan od uspješnih primjera prikazana je primjena hladne argonove plazme u svrhu modifikacije površine i strukture lignoceluloznih vlakana. U najnovije vrijeme se mikrovalna energija počinje sve više primjenjivati u postupcima recikliranja otpada, te su u radu navedeni primjeri iz prakse. U zaključnom dijelu rada su date smjernice i preporuke koje mogu pridonijeti bržem uvođenju ove zelene tehnologije u iznimno značajan sektor tekstila, odjeće, kože i obuće.

Ključne riječi: *mikrovalna energija; oplemenjivanje tekstila; lignocelulozna biomasa; izolacija celuloznih vlakana*

1. Introduction

The range of microwave radiation (MW) lies in the electromagnetic spectrum between infrared and high-frequency radiation, and the wavelengths cover the range from 1 mm to 1 metre. Electromagnetic waves that propagate in space represent the oscillation of an interconnected electric and magnetic field that is perpendicular to it, Fig.1. Microwaves are categorised as electromagnetic waves due to their frequency of between 300 MHz and 300 GHz.

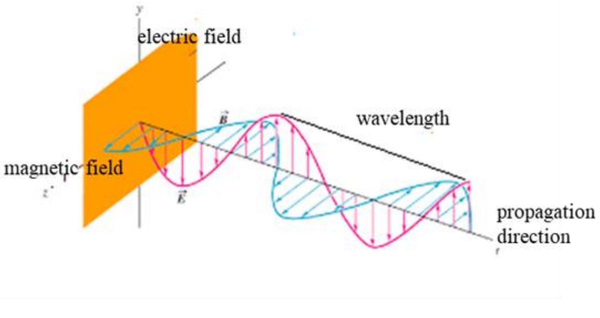

Fig.1 Schematic representation of electromagnetic waves [1]

Due to their extremely low energy, microwaves cannot cause any direct changes in the structure of molecules. The energy of microwaves is 0.125 kJ/mol [2], whereas a classical chemical bond has an energy of 335 to 85 kJ/mol. Electromagnetic waves can be absorbed and become a unit of energy called a photon, and the energy they transmit depends on the wavelength and frequency of the radiation [3]. Their value is very low when you consider the energy required to destroy chemical bonds. The principle of their action is to increase the kinetic energy of the stimulated dipole molecules, making them more mobile. The increased absorption of electromagnetic radiation and the resulting friction cause heating throughout the volume [4].
Polar molecules are required for the effective use of microwave radiation in the system, and the operating frequencies of the devices are set according to the medium they use, usually water. A polar water molecule vibrates at a frequency of 2.45 MHz and the application of microwave technology causes the molecules to rotate 5 million times per second. The energy of the microwaves can be converted into heat by the rotation of ions or dipole molecules in relation to the electric field. Looking at the behaviour of microwaves, materials can be divided into those that absorb microwave energy (they heat up), those that reflect microwave energy (they do not heat up) and those that transmit microwave energy (they do not heat up). The energy supplied by microwaves can be converted into heat: by the rotation of dipole molecules or ions in the direction of the negative/positive pole of the electric field, by the elongation of large molecules and by ionic conductivity. Dipole rotation is defined as an interaction in which a polar molecule changes its position in the direction of the electric field at extremely high speed. This rotation is caused by the molecule's endeavour to align its dipole moment with the electric field. The large oscillations create friction, which manifests itself as heat that heats the material in interaction with the rotation. The main difference between microwave heating and classic drying is the type of drying (Fig.2). In classic drying, the heat is transferred from the ambient air (convection) or the heating element (conduction) to the material, which leads to large heat losses.
Classic drying is primarily characterised by surface drying, in which the heat then penetrates into the interior of the material, which remains cooler as a result. A positive feature of microwave drying is the effect of electromagnetic radiation on the entire surface, including the interior, as well as the spread of heat from the inside to the outside. In microwave drying, the direction of heat and water movement is the same, resulting in more uniform heating. According to the literature, the energy consumption of microwave heating is 60-70% lower than that of conventional heating [6].

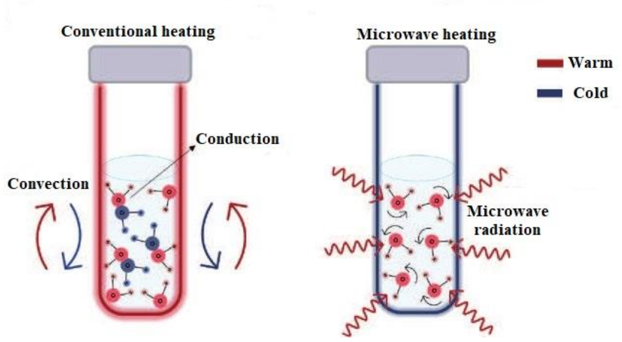

Fig.2 Schematic representation of classical and microwave heating [5]

Research into chemical reactions supported by MW radiation is becoming increasingly important, precisely because of the considerable energy savings that can be achieved. Several thousand scientific papers have been published since 1986 and MW radiation is being used more and more frequently. In the early days of microwave synthesis, there were problems with the experimental part in household microwave ovens, insufficient repeatability, inability to control pressure and temperature, and the high risk of explosion when using organic substances. in 1970, Miller [7] patented the Presset process. In 1947, the first microwave oven appeared in the USA, and in 1949 P. L. Spencer patented the design of a microwave oven. In addition to the patent, he was also involved in the development of magnetrons for radar equipment, which served as the main source of microwave radiation. It is only since 1980 that the use of microwave ovens in households has increased dramatically worldwide. However, chemical reactions in microwave ovens have also led to explosions and it was not until after 1990 that significant research was undertaken. Prototypes of modern systems were developed, the main features of which included pressure and temperature control, which also enabled the use of flammable organic solvents. At the beginning of the 21st century, multifunctional microwave reactors began to be used in which a larger number of chemical reactions could take place. Nowadays, microwave heating is used as a substitute for conventional heating. However, the prerequisite is that the material to be processed absorbs electromagnetic energy, which is then converted into thermal energy.

Microwave heating systems consist of three main units: Magnetron, waveguide and applicator, Fig.3. The magnetron is used as a source of microwave energy in industrial and domestic microwave ovens. It consists of two main parts, the anode and the cathode, which convert electrical energy into microwave energy [8].

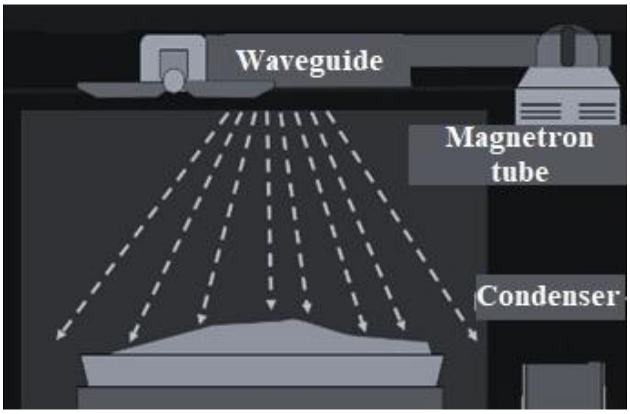

Fig.3 View of the microwave system [9]

2. Application of microwave technology in the isolation of textile fibres from biomass

The term biomass refers to the biodegradable part of residues, products and/or waste from the wood industry, agriculture and forestry and also includes biodegradable parts of municipal and industrial waste that are authorised for energy use [10]. Biomass is of natural origin, degradable and regenerates through natural processes. Fig.4 shows the subdivision of biomass according to its origin:

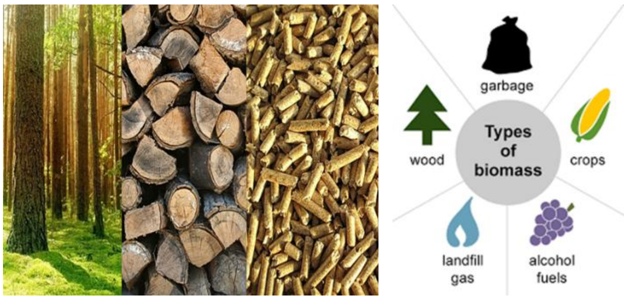

Fig.4 Types of biomass [10]

a) Forest biomass: waste and residues from the wood–processing industry
b) Non–wood biomass: products from targeted cultivation – fast-growing algae and grasses as well as agricultural waste and residues
c) Biomass of animal origin: animal remains and waste.

Depending on the defined form, biomass is divided into liquid biofuels, solid biomass and biogas [11]. From a socio-economic point of view, biomass is considered an ideal resource for the production of renewable and environmentally friendly products. The use of biomass as a raw material therefore minimises waste and pollution and offers a potentially sustainable alternative to petroleum in the production of chemicals, textile fibres and energy.

2.1. Lignocellulosic biomass

Lignocellulosic biomass, which includes agricultural and forestry residues, crops and wood, is a renewable [11], carbon-neutral and environmentally friendly raw material [12]. Lignocellulosic biomass includes agricultural residues such as corn, wheat and rice straw, agricultural by-products such as hulls, bran, bagasse and energy crops such as grass, miscanthus, mixed grasses and hybrid poplar [13]. Lignocellulosic material consists mainly of three biopolymers: Cellulose, hemicellulose and lignin, Fig.5. Normally, most lignocellulosic biomass from agriculture consists of about 40–50% cellulose, 20–30% hemicellulose and 10–25% lignin [14].

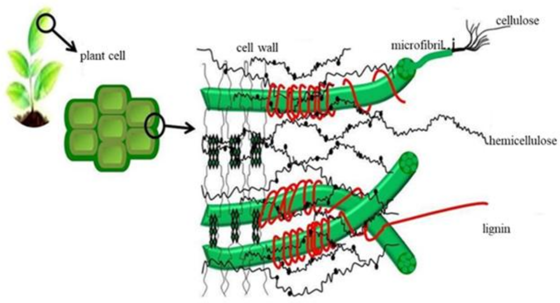

Fig.5 Schematic representation of a plant cell and the composition of its wall [21]

Cellulose is an unbranched linear polymer of β-(1→4)-D-glucopyranose units. Hemicellulose is a branched linear polymer formed from the anhydrides of various sugars. As it is characterised by a low degree of polymerisation, it has low mechanical and chemical resistance. Hemicellulose is always accompanied by cellulose and lignin and does not exist in free form. The lignin molecule is a very complex, cross-linked, 3-dimensional polymer that consists of phenolic units and gives the plant cells strength and structure. Lignocellulose biomass is characterised by its strength, reactivity and biodegradability. It consists of 75% carbohydrates and will be an indispensable source of carbohydrates for fermentation in the future. Lignocellulosic biomass is an environmentally friendly energy source and, in addition to the production of biofuels, electrical and thermal energy [15], is also frequently used for the production of textile fibres [16-20].

2.1.1. Isolation of fibres from biomass

The research and investigation of new natural "green" resources that are characterised by environmental friendliness and sustainability is becoming increasingly present. One such example is the isolation of fibres from lotus roots and stems left behind in ponds after lotus harvesting or flowering. Such and similar bioresidues can provide abundant cellulose that can be used in the textile, construction, medical or paper industries. Nowadays, lotus fibres are used for the production of luxury clothing, as the garments are environmentally friendly and comfortable. In addition to the production of luxury clothing, they can also be used for the production of composite materials [22]. Currently, manual extraction is the most common method for obtaining lotus fibres [23]. One of the most efficient methods for obtaining natural fibres is treatment with sodium hydroxide (NaOH), which is successfully used for the pretreatment of cotton, flax, hemp and ramie as well as the above-mentioned lotus fibre [24]. In order to obtain cellulose-rich natural fibres, sodium hypochlorite (NaClO), sodium chlorite ($NaClO_2$), hydrogen peroxide (H_2O_2) and benzoate have been used so far [24]. An environmentally friendly reagent, hydrogen peroxide, whose decomposition products are also environmentally friendly, continues to be used. An example of its application is the isolation of lotus fibres from lotus stems using hydrogen peroxide with prior treatment with sodium hydroxide under the influence of microwave radiation [22]. Cheng *et al.* found that the rate of removal of impurities and contaminants increases from 53% to 73% when the treatment time with hydrogen peroxide under the influence of microwave radiation is increased from 0 to 10 minutes, reaching 77% after 25 minutes of treatment. There are numerous examples of research using biomass as a raw material for fibre insulation, a significant proportion of which is due to the use of microwave energy in this process. Miscanthus (*Miscanthus x giganteus*), Fig.6, is well known as a high quality lignocellulosic biomass used to produce biofuels, but recent research shows that Giant Miscanthus is also an excellent source of cellulosic fibres [25,26].

Fig.6 Giant Miscanthus - *Miscanthus x giganteus* [27]

For the past 20 years, more and more attention has been paid to research on the pretreatment and enzymatic hydrolysis of Miscanthus. Unprocessed Miscanthus has 42% hemicellulose, which contains arabinose, galactose, glucose, xylose and mannose. The use of microwave energy at 130 ºC with H_2SO_4 reduces the hemicellulose content in the biomass by 21%. Increasing the temperature further reduces the amount of hemicellulose. Sulphuric acid removes the hemicellulose in the biomass more efficiently than caustic fizzy drink and water. In addition to the removal of hemicellulose, the removal of lignin is also improved by the effect of microwave energy. Under microwave conditions, a temperature of 180 ºC is decisive for the decomposition process of the biomass [28].

A research group from the University of Zagreb Faculty of Textile Technology has successfully isolated fibres from the biomass of wheat, barley and maize [29,30] as well as from various wild plants (Spanish broom and giant reed) and energy crops (miscanthus and Virginia mallow) [31,32].

Spanish broom, as one of the most widespread Mediterranean wild plants, is discovered to be an excellent source of fibre. In the past, the fibres were extracted from broom by soaking the stems in the sea for 20-40 days [33]. The use of low concentration sodium hydroxide and microwave energy in the treatment of broom stalks significantly shortened the process of fibre isolation, while maintaining the same or even better fibre properties compared to the historically used methods. Dominić *et al*. used microwave energy in fibre isolation from the Miscanthus plant [34]. Exposure to hydrogen peroxide and citric acid, followed by a short treatment with low-concentration sodium hydroxide, isolated cellulose fibres with a fineness of 10 to 15 µm.

2.1.2. Application of microwave plasma in the processing of biomass

The term plasma comes from the Greek word "plasso", which means self-forming matter [35]. It is defined as an ionised gas consisting of charged particles such as photons, ions, electrons and radicals. Its physical and chemical properties are due to the interactions of charged particles in combination with a neutral gas in contact with a surface. For this reason, it is also known as the fourth state of matter. This state is much more excited than the other states of matter. It is quasi-neutral, i.e. it has an equal number of negatively and positively charged ions.
A schematic representation of atmospheric plasma with a microwave generator operating at 2.45 GHz and used for processing cellulose material is shown in Fig.7.

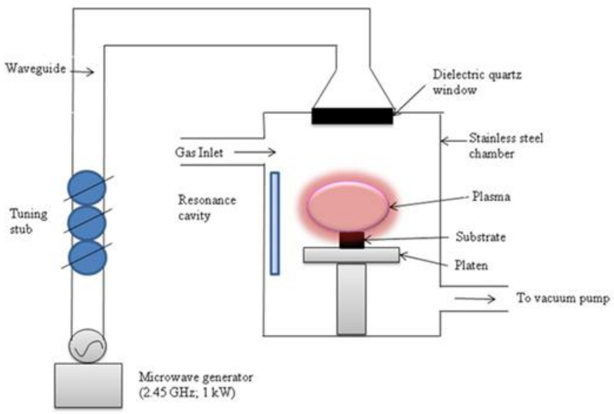

Fig.7 Illustration of the atmospheric microwave plasma [36]

The use of plasma as a tool for biomass conversion has been widely researched. Atmospheric plasma is used to treat different types of biomass to change their surface and structural properties [37]. The aim is to separate the cellulose from the lignin-hemicellulose complex in the biomass using a green process. One of the examples of the successful application of MW plasma is the pretreatment of sugar cane bagasse [38]. In addition, Zanini *et al*. achieved significant surface modification and degradation of the lignin layer by plasma etching [39]. They applied a cold argan plasma treatment to change the structure of ligno-cellulosic fibres and found that the chemical structure of lignin can be modified very well in this way. Plasma generated by microwaves at atmospheric pressure has several advantages: Microwave discharges allow better energy transfer into the plasma itself, the efficiency is almost 100 % and the energy consumption is lower compared to conventional methods [40].

3. Microwaves in textile finishing

The use of microwaves is of great importance because they are used in mobile phone technology, for wireless computers and television and even for launching satellites into space. They are used in numerous industries, including the textile industry. In addition to the already mentioned processes of isolating textile fibers of plant origin [41-43], microwave technology is also used in the textile finishing process. The main purpose of using microwave radiation is to reduce processing times, as conventional heating agents used in textile finishing processes result in large losses of time and energy [44,45].
Initially, the possibility of using microwaves for pre-processing processes such as boiling or bleaching of cotton fabrics [46] and for the pre-processing of silk yarns was investigated, with microwave radiation reducing the time required to achieve the same degree of degumming as with the conventional method [47].
An example of the successful application of microwave energy is the dyeing and fixing of various types of textile materials [44,45]. Microwave energy has been used to dye cellulose [48], protein and synthetic fibres [49], acrylic fibres [50], polyamide 6.6 [51] and polyamide 6 [52], accelerating the dyeing process. The dyeing process accelerates the diffusion of dye molecules as polar dye molecules and fibres begin to absorb microwaves [51]. The application of microwave energy in the dyeing process of polyester fibres [53-56] increases the dye extraction rate and shortens the dyeing time [57-59]. The use of microwave energy in the dyeing process of polypropylene improves the fastness and strength properties of the dyed material [60-62].
Microwave treatment can also be used in textile finishing, e.g. for treatment against moths [60]. Studies show that treatment with microwave energy not only improves the moth protection effect, but also reduces the amount of formaldehyde released by 50% compared to conventional heat treatment [63]. Another example of application of MW energy in finishing is investigation of effectiveness of cross-linking between reactive polycarboxylic acid and cellulose reactive groups. Improvements in crease-resistance [64], water and oil repellency and flame retardance were achieved while at the same time reducing the strength loss of cotton fabrics [65]. In

the case of the application of MW in the antibacterial treatment of cotton fabrics, the reduction in tear strength was less pronounced [66]. Furthermore, MW has been shown to be more effective in protein-based fibre modification processes, as it ensures higher productivity and better quality of the textile material [67]. In addition to these studies, microwave radiation has also been used in drying, fixing [68-72] and disinfection processes [73,74] to shorten process times and achieve more efficient results.

3.1. The effect of microwaves on the properties of cotton material

Cotton is one of the most widely used natural polymers with a wide range of applications, from clothing to home textiles and industrial products. Conventional cross-linking systems used to finish cotton fabrics transfer heat to the fabric by convection, conduction or radiation. Microwave irradiation is a non-contact heating process in which vibrations and rotations of permanent dipoles are generated in a microwave field. With this method, the heat can be evenly distributed over the entire volume of the textile substrate [75,76]. Very few studies have investigated the use of microwaves for the finishing of cotton [66,77-79]. It has been shown that microwave heating is faster, more uniform and more efficient than other heating methods. However, microwave irradiation can affect the chemical structure, physical properties and morphological surface structure of cotton materials. Studies on the effects of microwave energy on the physical properties and structure of treated textile materials are very rare. Cotton fabric in a wet state contains free water molecules and the fibres partially absorb microwave energy. The energy of the electric field is converted into heat by the dielectric losses of the water when the cotton fabric is wet. Water molecules oscillate in the presence of a high-frequency electromagnetic field [80]. Research into the changes in the structure and properties of cotton fabrics in the wet state after microwave irradiation forms the basis for the further application of microwave technology in the wet treatment of cellulose materials.

4. Application of MW energy in recycling

One example of microwave energy usage in recycling is the processing of polyethylene terephthalate (PET). Nowadays, PET-based waste is mainly processed by mechanical methods with the aim of recovering solid plastic waste for reuse. Due to the heterogeneity and degradation of plastics, only single-pole plastics can be produced in order to avoid more complex and contaminated waste. The only questionable aspect is the quality of the mechanically recycled products, which could ultimately only be disposed of in a landfill or incinerated. As part of the DEMETO project [81], a patented technology was developed for the application of chemical recycling of PET waste. Thank you to this recycling process, it is possible to extend the service life of PET. Alkaline hydrolysis was used for the depolymerisation reaction in a microwave reactor. This project is an example of a good realisation of the idea, which provided guidelines for the further implementation of the recycling process using microwave energy.

In the past, biotechnologies dominated commercial processes, but today microwave and thermochemical processes are more widely researched and investigated. Based on the above facts, lignocellulosic biomass represents a renewable source for the production of value-added chemicals that could replace petroleum resources and make an important contribution to sustainable development. Research should focus on the efficient separation of lignocellulosic biomass and the development of environmentally friendly catalysts that contribute to the reduction of reaction by-products and thus product selectivity [82].

Over the last decade, there has been increasing interest in the production of bio-based chemicals from organic waste and lignocellulosic biomass as more environmentally friendly energy sources. Conventional thermochemical and biochemical methods using microwave technology are used to convert biomass into biochemicals through various processes such as depolymerisation, fermentation or hydrothermal liquefaction.

In this case, the more mobile and polar components of the biomass absorb the microwaves more efficiently, but it should be taken into account that the crystalline region of the cellulose absorbs less than the amorphous region. The water in the biomass absorbs the microwaves most strongly. As the temperature increases, the water content decreases, but in the course of the reaction products are also formed, such as the coal obtained by pyrolysis. The synergy between microwaves and biomass components can accelerate the efficiency of biomass conversion into chemicals. To summarise, microwave technology is not only a source of heat, but also plays an important role in the reaction mechanism itself. Microwave radiation enables more efficient internal heating by transferring energy directly to molecules, with the aim of increasing the temperature through ion conduction and dipole rotation. Processing time is significantly shortened, costs are reduced and the quality of the resulting value-added product is improved [83].

5. Conclusion

Nowadays, microwave heating is increasingly being used as a substitute for conventional processes. However, the prerequisite is that the material to be processed can absorb electromagnetic energy, which is then converted into thermal energy. The use of microwave energy delivers more efficient results than conventional heating by conduction or convection. The main advantage of using microwave radiation is the reduction in processing time, as conventional heating agents used in textile finishing or in the pre-processing of biomass lead to large losses of time and energy. Usage of microwave energy in the dyeing process of various fibres, starting with cellulose, proteins, but also synthetic fibres (acrylic fibres, polyamide 6.6 and polyamide 6) has considerably accelerated the dyeing process itself.

Research shows that microwave energy improves the efficiency of finishing treatments such as anti-crease (Durable Press, Easy Iron) treatment, with the benefit of additional reduction of formaldehyde released amount by 50%, compared to conventional thermal treatment.

In addition to the processing of textiles, microwave technology is increasingly being used for the pre-treatment of biomass. The use of biomass has great potential as it is the most widely used form of renewable energy. Numerous studies have been conducted on the application of various technologies for the efficient conversion of lignocellulosic biomass into biofuels (e.g. ethanol) and other bio-products (e.g. cellulosic fibres). In view of current research, a further increase in the application of microwave energy in various stages of biomass or textile material processing is expected.

Despite some limitations, such as the slow introduction of new technologies and the lack of professional personnel who can quickly put advanced technologies into practise, the use of microwave energy in the textile sector is on the rise at a global level. As various institutions, organisations and associations raise awareness among their members and explain the benefits of microwave technology to investors, the use of this green energy is expected to be increased. This will contribute directly to achieving the goals of the European Green Agenda. At the same time, it will also help to increase the competitiveness of those industries that utilise advanced processes and materials while reducing waste as much as possible.

The research presented in this article originates from the bibliographic report prepared for the course "Methods of Scientific Work" in the doctoral program "Textile Science and Technology" at the University of Zagreb Faculty of Textile Technology.

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