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Ssue 104 S	eptember 30, 20
In this Issue:	Page
Commercial Extraction of Cannabis – Process Intensification through the Applica Microwaves Marilena Radoiu	ation of 1
Microwaves in the Biorefinery Eleanor Binner and John Robinson	8
Future Challenges and Opportunities of Emergent Technologies in Romania Mariana Patrascu	12
Ricky's Afterthought: DATA, DARK DATA and their STORAGE A. C. (Ricky) Metaxas	16
Upcoming Events A workshop on HPM industrial applications at EUMW2020	18
Announcement of Special Issue on Advances in Microwave Processing of Materi	als 19
AMPERE-Newsletter's Editorial Information	20

Commercial Extraction of Cannabis – Process Intensification through the Application of Microwaves

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*This article is a summary of a full paper recently published on this study [1]

1 Introduction

Cannabis is a genus of flowering plants belonging to the cannabaceae family with three main species: Cannabis sativa L, Cannabis indica L, and Cannabis ruderalis L, Figure 1 [2].

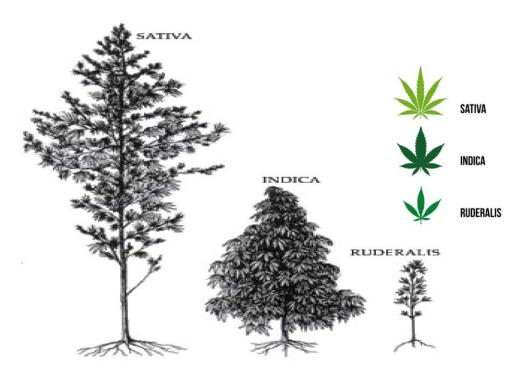


Figure 1: Cannabis plants [2].

Cannabis has a long history of being used for medicinal, therapeutic, and recreational purposes. Cannabis is known, for example, to be capable of relieving nausea (such as that accompanying chemotherapy), pain, vomiting, spasticity in multiple sclerosis, and of increasing appetite. The importance of cannabis in therapeutics is emphasized by the ever-increasing number of research publications related to the use of cannabis and its derived products to treat various indications [3-6].

Cannabis contains more than 500 different compounds, which include terpenes, flavonoids, lipids, sterols, chlorophyll, fatty acids, salts, sugars,

and a unique class of terpeno-phenolic compounds known as cannabinoids or phytocannabinoids. More than 100 cannabinoids have been identified in different cannabis plant strains. Examples include Δ9-tetrahydrocannabinolic acid (THCA), cannabinoldiolic acid (CBDA), cannabinolic acid (CBNA), cannabigerolic acid (CBGA) and cannabichromenic acid (CBCA). In fresh plant material all cannabinoids are present in their acidic form. The acidic cannabinoids can be converted into their decarboxylated (neutral) analogues (CBD, THC) under the influence of light, heat, or prolonged storage, by losing the relatively unstable carboxylic

group in the form of carbon dioxide. THC and CBD are the most widely studied cannabinoids and have been associated with the therapeutic and medicinal properties of the cannabis plant and its associated products and also with its popularity as a recreational drug. THC is mainly recognized for its psychotropic effects when consumed, but lately has also been found to effectively treat pain, muscle spasticity, glaucoma, insomnia, lack of appetite, nausea, and anxiety while CBD is used to treat migraines, inflammation, seizures, irritable bowel syndrome (IBS), depression, insomnia, and anxiety [3,4]. CBD is non-psychoactive and is the major cannabinoid constituent in hemp cannabis.

The terms hemp and marijuana classifications of cannabis adopted into culture even not represent legitimate they do nomenclature for cannabis. Hemp and marijuana are both cannabis; hemp, however, refers to cultivars of cannabis that contain very low concentrations of psychoactive THC (typically less than 0.3% by dry weight). Hemp (sativa) is an industrially grown plant that is cultivated outdoors, better suited for warm climates with a long season. It is mainly used to produce textiles from the fibre, and foods and supplements such as protein and essential fatty acids from the seeds. Hemp seed oil is rich in unsaturated omega-3 and omega-6 fatty acids and is almost entirely devoid of cannabinoids. Marijuana, on the other hand, is often deliberately bred and cultivated in controlled environments in order to optimize the cultivar's characteristics, including the composition of cannabinoids such as THC and CBD. Controlled growing and cultivation is designed to produce female plants that yield budding flowers rich in cannabinoid content. Harvesting of industrial hemp has traditionally avoided collection of flowers to minimize cannabinoid content of industrial products. This practice is however changing as the production of CBD from farmed hemp becomes legalized in more and more jurisdictions world-wide.

North America is experiencing a boom for cannabis-derived products (i.e. packaged foods, edibles and beverages, beauty and personal care, consumer health, pet care, home and garden), made possible by the legalization of recreational cannabis in Canada in 2018 and in 11 U.S. states, two U.S. territories, and the District of Columbia. The global market for cannabis-derived products was ~ 5 trillion

USD in 2018 and is expected to grow 1,200% by 2023 [5].

To this end, there are various conventional biomass extraction methods available for the extraction of cannabis. Given the inherent commercial value of CBD and THC, the applied method to extract them is very important in terms of accomplishing the quantity and quality of the product. Moreover, economics of the processes is a very important parameter in its commercialization.

2 General considerations of Cannabis extraction

In general, the most appropriate methodology to obtain an extract from raw biomass must be selected according to the characteristics of the desired product. There are several important factors to consider when choosing an extraction method for cannabis, the most important being as follows:

- Extraction efficiency, the percentage of bioactive compounds recovered through the entire extraction process;
- Extract quality and consistency, including the purity or "potency" of cannabinoids in the extract and also the relative amounts or "profile" of other potentially synergistic compounds such as terpenes;
- Throughput capacity and scalability, assessment of the extraction method and its efficient implementation at commercial scales vs. market demand;
- Environmental control, e.g., carbon footprint and safety, i.e. minimize risks to the consumers and worker safety.

In many cases, additional processing steps, both upstream and downstream of the extraction itself, are required to obtain the final cannabis extract product. The incorporation of these steps with the extraction method and their impact on the overall process efficiency and product quality must also be considered. Some common processing steps discussed further below include:

- Decarboxylation, the process of converting nonactive native acidic cannabinoids into their active, neutral forms via a thermal reaction;
- Winterization, the process of removing plant lipids and unwanted waxes by a secondary solvent, freezing and filtration;

- Decolorization, the process of removing chlorophyll and unwanted pigments;
- Secondary purification, the process of further purifying the extract to increase the potency or alter the composition of cannabinoids and other components, via various methods including distillation, chromatography, or crystallization.

There are generally three typical extraction methods currently being used for commercial cannabis extraction, albeit at only modest scale:

- Supercritical CO₂ (SC-CO₂) extraction
- Pressurized gas (hydrocarbon) extraction
- Conventional organic solvent extraction
 These are discussed in more detail below.

In addition to these "big three", there are several non-conventional, alternative extraction methods that are being assessed at laboratory scale, including for example ultrasound-assisted extraction, hydrodynamic extraction, pulsed-electric field extraction. Given that none of these has yet been demonstrated at any reasonable commercial scale, they are not further discussed.

2.1 Supercritical CO₂ (SC-CO₂) extraction

Supercritical fluids are a well-documented alternative to traditional organic solvents suitable for various extractions. Any material in its critical state when it is both heated above its critical temperature (T_c) and pressurized above its critical pressure (P_c) and hence there are no distinct liquid and gas phases. The specificity of this technique relies on solvent's physicochemical properties, which can be 'tuned' by an increase of pressure and/or temperature beyond its critical values [6].

Supercritical CO₂ extraction is a common technique for cannabis extraction-separation, which uses supercritical CO₂ (74 bar, 31°C) in a batch process. Although non-toxic and non-flammable, SC-CO₂ requires very high pressures to be employed. In addition, the method is somehow inefficient and, therefore, not conducive to high throughputs, as well as environmentally damaging (e.g., producing large amounts of the greenhouse gas carbon dioxide as a by-product). The resulting extracts are, however, considered to be solvent-free. The decarboxylation must be carried out on the cannabis biomass upstream the extraction process (acidic cannabinoids are poorly soluble in SC-CO₂).

This potentially increases overall costs (decarboxylation must be performed in advance on what may be large quantities of cannabis biomass) and leads to the loss of some light volatile terpenes. SC-CO₂ also co-extracts heavy fats and waxes which must be subsequently removed in downstream processing steps (winterization), leading to further cannabinoid losses and reduction in overall efficiency or recovery of available cannabinoids. Finally, the scale up of SC-CO₂ is only possible by addition of multiple machines.

2.2 Pressurized gas (hydrocarbon) extraction

Hydrocarbon extraction is the most popular technique that uses liquified gases such as n-propane and n-butane pressurized into liquids (2-10bar) as solvents for extraction of cannabinoids. An advantage of the method is the possibility of these gases to remain in liquid phase at low pressure and the possibility to remove them from the system at the end of the extraction by gentle heating leading to an extract with low traces of residual solvent. Hydrocarbons such as n-butane and n-propane are good solvents for the low-polarity cannabinoids [6]. In this method, butane or propane is pressurized to a liquid state for extraction and then either depressurized or heated for removal from the obtained extracts. This extraction process is carried out in batch and creates what are known as cannabis "concentrates", e.g., shatter, a viscous material with very high concentration of THC and other cannabis compounds like terpenes, which is popular for recreational users. Decarboxylation can be carried out upstream or downstream of the extraction. Although effective, the process is undesirable for medicinal and consumer products, due to the risk of solvent contamination. Safety is also a major concern given the high flammability/explosivity of the hydrocarbon solvents employed. In principle, the scale-up is only possible by the addition of multiple machines.

2.3 Conventional organic solvent extraction

The most traditional and perhaps the simplest method for extracting active compounds from cannabis involves maceration in organic solvents such as ethanol, ether, chloroform, and methanol. When organic solvents are used for the extraction, the obtained product consists of various compounds, including some undesired substances that dissolve together with the cannabinoids. Also, high boiling or extraction temperatures often lead to the degradation of heat sensitive compounds. This extraction method is operated in either batch or continuous flow and can use decarboxylated biomass or decarboxylation can be performed on the extracted product. The main drawbacks of the method are linked to the high input ratios of biomass-solvent and implicitly to the high quantities of solvent to be separated from the extract and recycled and also to the co-extracted molecules, such as fats, waxes, and pigments, which means more complex downstream processing (separation, purification, etc.)

3 Microwave-Assisted Extraction of Cannabis (MAE) in continuous flow, MAP™

Microwave-Assisted Extraction (MAE) is different from the methods presented above because the extraction occurs as a result of the volumetric heating as opposed to transferring heat from the surface inwards, making the process more efficient and more uniform due to the ability to precisely control temperature and contact time. The very nature of heating through the involvement of the raw material under processing (instead of using fossil fuels or less efficient, indirect electrical heating systems) brings about quality consistency as well as positive environmental impacts.

The carefull design and optimization of all MAE parameters (e.g., solvent type, residence time, extraction temperature, microwave power density) and of the reactor (e.g., microwave frequency, number of microwave inputs along the reactor, precise measurement and control of forward and reflected power) can lead to lesser solvent requirement as compared to conventional methods and the biomass can be exhausted with one extraction only.

The basics of MAP^{TM 1} continuous flow extraction of cannabis consists of coupling MAE and continuous flow technology and as such creating a very promising way to produce high value-added extracts since unlike batch processing, the continuous flow has been demonstrated to facilitate

¹ MAPTM is a patented microwave-assisted processing by Radient Technologies Inc. (www.radientinc.com), which has been successfully operating a continuous-flow microwave

process intensification and contributes to a safe, efficient and sustainable production. By employing continuous-flow MAPTM, it is possible to control extraction time and temperature very precisely, both of which can greatly influence extraction efficiency and the composition of the extract.

A schematic of one process involved in the cannabis extraction of the biomass decarboxylation of the extracted products is presented in Figure 2. In this method, the raw milled cannabis biomass is mixed with a solvent (e.g., ethanol, IPA, pentane, PEG400) selected based on its dielectric properties vs. type of biomass and its concentration of cannabinoids. The obtained slurry is pumped in the continuous flow microwave-assisted extraction reactor and progressively heated to the desired extraction temperature by using 915 MHz microwaves – Figure 3; the microwave power can be automatically 'tuned' to the process conditions as to reach power densities between 0.1 and 10kW/kg of biomass. Downstream the extractor, the spent biomass and the extract are separated from the slurry. The extract is treated to obtain a final product containing the target compounds in sufficiently high yield and high purity. The spent biomass may be processed to yield less than 0.3% concentration of THC naturally produced by plants and disposed of once this condition has been achieved [1].

The extractor/reactor consists of a food grade stainless steel tube within which a mechanical stirrer is placed. Microwaves are provided from a 75kW (max. power), 915MHz microwave generator consisting of a low ripple switch mode power supply, a magnetron head, a circulator and water cooled load with reflected power meter. The microwave generator can be operated from 10kW up to 75kW in continuous wave (CW) mode or controlled pulse. Due to the possibility of working with flammable solvents, the microwave generator is installed in a different, non-ATEX (directives for explosive atmospheres) room. The microwave transmission line, standard WR975 rectangular waveguide, passes the wall between the ATEX and non-ATEX environments through a separation window and then it splits into two inlets delivering equal microwave

extractor in Canada for over five years at throughputs over 200kg/h of biomass input

power all along the reactor. Within the reactor, the separation between the reaction mixture and the microwave transmission line is done via microwave transparent windows.

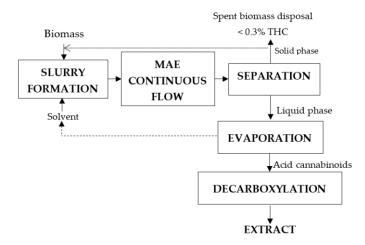


Figure 2: Schematic of the microwave-assisted cannabis extraction and acidic cannabinoids decarboxylation.

Due to the continuous measuring and controlling of the reflected power and the automatic impedance tuner installed immediately after the circulator (in the non-ATEX zone), the microwave forward power is automatically adjusted as to maximize the absorbed energy by the extraction mixture and to minimize energy losses by reflected power. Microwave components located within the ATEX zone are continuously purged with nitrogen; arc detectors are installed within all microwaves components as such as the microwaves are shut down if arcing detected. Wall mounted microwave leakage detectors can shut down the microwaves if leakage levels $> 2.3 \text{mW/cm}^2$ are detected around the reactor. As described in Figures 2 and 3, the main advantages of MAPTM related to cannabis biomass are:

- Continuous-flow method at atmospheric pressure which allows for much higher volumes of cannabis biomass to be processed in much less time than existing extraction methods;
- Higher rates of consistency and quality because the process does not require stopping and restarting material flows;
- Scale-up to industrial scale without the need to purchase an endless supply of new machinery and without the use of pressurized batch vessels;

- Eliminates additional steps required in most extraction methods, such as winterization;
- Ability to achieve high extraction efficiency at industrial scale. Typical recovery of active compounds via MAPTM is up to 95%.From a process intensification view, the continuous flow extraction and its heating via microwaves comes with several additional benefits, including significantly increased flexibility and safety with respect to operation:
- The contact time between the biomass and solvent before, during and after microwave treatment can be adjusted much more easily;
- It is fully ATEX or "Hazardous zone" classified, meaning it can be used with any flammable liquid and be completely safe.
- It is possible to precisely control biomass residence time in the microwave zone and if desired separate the biomass from the solvent very quickly after treatment, or continue contact for any length of time at any temperature, depending on the desired outcome;
- The use of multiple microwave field deposition points through the use of a split waveguide and a "ridge wave deposition" allowing for non-uniform dispersal of the wave from the inlet to the outlet to account for changing dielectric properties as the material is treated;
- It has an automatic impedance matching unit that allows for constant, automatic adjustment of the field strength and microwave energy absorption maximization;
- It has a built-in mechanical agitator with variable speed control to randomize movement of biomass thus making the field uniform for the materials at all times;
- It is fully automated (operators simply input desired MW parameters on an HMI and it runs itself while connected to the plant PLC systems).

The extractor is also easily scalable. The continuous flow approach eliminates the requirement for having geometric similarity between scales, i.e. the equipment shape and dimensions do not have to scale proportionately. Classically, even geometric similarity does not ensure thermal similarity in scaled systems; for example, heat transfer is an interface-controlled process and so the surface area relative to the volume is critical. As the volumetric scale increases, the area relative to the

volume decreases and the overall efficiency of heat transfer can decline considerably. There is no thermal inertia with microwaves, on the other hand. Since penetration depth is not an issue with the continuous flow design, the energy is deposited uniformly throughout the mixture resulting in rapid energy transfer and direct dielectric heating – hence the thermal inertia inherent to classical methods is not an issue.

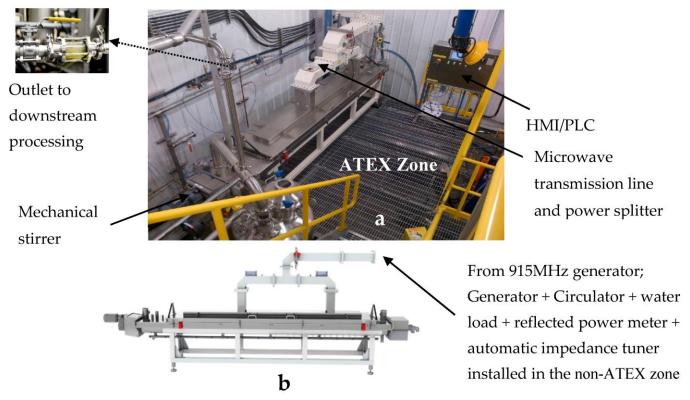


Figure 3: Photo and schematics of the continuous flow microwave extractor in ATEX environment. a) Photo of the continuous flow reactor located in the ATEX production zone; b) schematics of the continuous flow reactor showing the connection with the 915MHz generator installed in a non-ATEX zone

4 Conclusions

While there are various solvent methods for extracting the active compounds out of biomass, e.g., supercritical fluid extraction (SFE), Soxhlet, percolation, agitated tank, countercurrent, when considering cannabis extraction, none of these is optimal in all aspects. Molecules extracted through these processes may differ in the quality (physiochemical properties) and quantity hence altering the chemical composition of the extract; in addition, many of these methods have limitations when it comes to scaling up to suit mass production. In addition, as a result of increased legislation, concerns about the environment and competition within the globalized market, it has become paramount to look for and implement innovative, clean and sustainable ways to obtain natural extracts, i.e. green extraction of natural products. Green extraction refers to looking for, designing and implementing extraction processes that lead to (i) a reduction in energy consumption, (ii) utilization of alternative solvents to obtain products that are natural and renewable, and (iii) extracts that are safe and of high quality.

Microwave continuous flow extraction is a good example of a such technique. In the MAPTM reactor, the process is run in a continuously flowing stream, enabling very tight process control and improved mass heat and mass transfer, consequently achieving higher extraction control and higher product quality. Furthermore, continuous extractors can be easily scaled up by placing multiple cavities in series or in parallel, thereby shortening development time for full-scale production.

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About the author



Dr. Marilena Radoiu is the founder of Microwave Technologies Consulting, France and since January 2018 she has been also acting as the Managing Director of Microwave Innovation at Radient Technologies Inc., Canada. She has more than 15-year experience in the development of microwave-

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Microwaves in the Biorefinery

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1 Introduction

In order to meet the zero carbon circular economy targets that are increasingly being committed to by governments and industry, biorefineries must replace oil refineries as the mainstream source of chemical feedstocks. To become viable, the efficiency and flexibility of biorefineries must be maximised to cater for the inherently wider range of feedstocks and products compared with oil refineries, as well as the geographical distribution wide rapidly deteriorating feedstocks. Microwave technology can play a major role in achieving this due to the potential for process intensification afforded by the microwave heating mechanisms. therefore offer an exciting double opportunity to enhance the performance and flexibility of biorefineries and to replace traditional sources of fossil heat with carbon-free electrical energy. However, step changes in the approach to the design of microwave processes are required to make this a reality. This is due to the fundamental differences in the way microwaves heat materials compared with conventional heating; as many readers well-know, microwaves cannot simply be "dropped in" to replace conventional heat sources. Bespoke systems must be designed based on an understanding of microwave-material interactions and the role they play in processing. For biomass processing this is only beginning to be understood, and the likely reason for the very low conversion of promising labscale microwave technology applications into commercial interests.

This article introduces recent advances in the understanding of microwave-biomass interactions and, more importantly, how this can fundamentally affect mass transfer during microwave processing. We follow with some examples of specific biorefinery applications we're working on, stressing that the fundamental understanding is absolutely key in helping us design the more applied experiments in

a way that will best exploit the microwave heating mechanisms and inform scale-up.

2 Recent advances in fundamental understanding

It is well-known that microwave processing is driven by the direct heat transfer mechanisms of volumetric (instantaneous heating throughout the bulk of the material) and selective heating (variation in heating rates resulting from differing abilities of system components to store and convert microwaves to heat depending on their dielectric properties). These lead to the development of different temperature profiles compared with conventional heating, which is indirect and in the case of biomass processing typically relies on conductive heating from the outside of the material. A key advantage of microwaves in many biomass processes is their ability to disrupt the cellular structure, enhancing the release of cell wall or intracellular chemicals. There are differing theories on the mechanisms by which this disruption is achieved, and understanding which of them are applicable to any given biomass feedstock and under what range of processing conditions is an absolutely essential input to the basis of design of microwave processes. Figure 1 is provided to illustrate this point. In Figure 1a, the selective microwave heating dominates the internal heat and mass transfer, leading to pressure build-up caused by superheated steam [1]. Figure 1b depicts temperature-induced diffusion, in which microwaveinduced temperature differences between the cell, cell wall and solvent drive mass transfer over and above conventional concentration-driven osmotic processes [2]. Both mechanisms could lead to enhanced performance (e.g. faster processing and improved yield/quality), but completely different processing conditions would be required to achieve these: In (a) superheating of steam is required, while in (b) only small degrees of selective heating can effect the pressures required for cell rupture.

b) Steam rupturing

Release of bioactive compound Rupture point Vacuole Ruptured plant cell

a) Temperature-induced diffusion

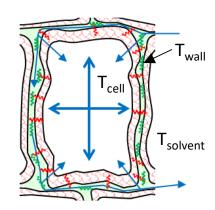


Figure 1: Depictions of possible disruption mechanisms

To better understand how and when these mechanisms may occur, we have recently proposed a cellular scale model of solvent extraction that incorporates conventional and microwave heating, cellular expansion, heat transfer and mass transfer [2]. Using microwave-assisted (solvent) extraction as a case study, we showed that steam-rupture is only possible at the extreme fringes of realistic physical parameters, while temperature-induced diffusion is able to explain cell-rupture across a broad and realistic range of physical parameters and heating conditions. It is interesting to note that, counter to our long-held beliefs, we found that outcomes were far less sensitive to changes in the relative dielectric properties of the solvent and biomass than other system properties such as conductivity. We also note that the study was limited to Microwave-Assisted Extraction (MEA) conditions, and other outcomes may be achievable for other microwave-biomass applications (i.e. steam rupture may be more likely in systems in which the processing conditions achieve significantly higher electric field intensities), and that the model is designed to be expanded and refined to include other processing regimes.

This approach to fundamental understanding can be used to inform the design of processes to exploit the effect of temperature-induced diffusion during microwave heating (i.e. to predict feedstock properties and processing parameters for which microwave heating could enhance process performance).

3 Examples of microwave biorefinery processes

3.1 Green extraction of unconventional polysaccharides

The development of novel products via the green extraction of polysaccharides from co-products and residues of the agri-food industry has received significant attention in recent years. processes such as commercial pectin extraction typically use hot-acid extraction [3]. However, this leads to large volumes of acidic wastes and is not suitable for the extraction of some polysaccharides, limiting the range of novel bio-based products that be produced. For example, can pectin oligosaccharides (POS). derived from rhamnogalacturan I-rich (branched or "hairy") pectin have been shown in-vitro to have a bioactive properties that indicate that they could be developed into novel products, including pharmaceuticals [4, 5] or a new class of prebiotic [6]. However, hot acid extraction destroys the neutral sugar side-chains of interest for these applications, rendering commercial extraction useless for hairy pectin extraction. Existing extraction methods are limited to laboratory scale. Enzymatic processes can produce high yields, but the high cost of the enzymes required has stifled scale-up. Many researchers in the past 20 years have reported that MEA can be used to effectively extract a range of materials, and in fact that it can achieve pectin extraction using only water as the solvent, making it a promising green extraction technique. However, until recently, fundamental understanding of how this process differs fundamentally from conventional solvent extraction has been lacking, and this has led to very low conversion of laboratory-scale experiments into industrial-scale demonstrations. The development of unconventional polysaccharide-based products such as bioactive pectin products has been hampered by the inability to produce them at large enough scale for in-vivo trials.

Many laboratory-scale studies have reported various novel pectin extraction procedures [3, 7, 8]. However, few have considered the effect of extraction method on pectin structure [7], and direct comparison of different technologies and processing parameters using the existing laboratory scale data is challenging due to the difficulty in varying only one process parameter independently. Our recent study [9] systematically investigated the effect of pH, heating type (microwave versus conventional heating), temperature $(90 - 150^{\circ}\text{C})$ and time on the yield and composition of pectin extracted from sugar beet pulp. Contrary to many studies, which report up to double the yield with microwave heating compared with conventional heating and differences in pectin structure [7], the study showed that the yield and composition of the pectin-rich extract was unaffected by heating type when both methods were carried out using the same processing parameters (including heating rate). The yield and composition were most affected by pH and temperature: the highest yields and purity of "hairy" pectin were achieved at high pHs, while strongly acidic conditions favored the extraction of homogalacturan (HG, "smooth" pectin). This is not to say that different feedstock-solvent combinations would yield the same results (i.e. that microwave heating had no effect on the yield or time), and indeed this is a subject of continuing investigation in our lab. The use of hydrothermal processing conditions (110 – 190°C in a sealed vessel) decreased treatment time and increased yield significantly, but pectin degradation was rapid after peak extraction yield was achieved. The results indicated that while strongly alkaline solvents may achieve the highest yields of pectin, hydrothermal processing and "hairy" atmospheric pressure water extraction may be favorable from a perspective of scale-up. In Mao et al., [10] we used a systematic approach to manipulate pectin structure and composition by varying okra

extraction conditions, and correlating this with the extract's functionality as a bioflocculant. While previous papers identified okra as a promising bioflocculant and characterized the pectin content in okra mucilage, Mao et al. demonstrated that the HG/RGI ratio could be used to directly predict the flocculating ability of pectin-rich extracts, which processing conditions would achieve this, thereby informing techno-economic assessment of potential feedstocks and processes [11].

We have utilized these advances in the understanding of the effect of processing parameters on experimental outcomes to scale-up a pectin extraction process [12]. The outcomes of laboratoryscale batch experiments were used as a basis of design for the continuous-flow MEA of pectinderived oligosaccharides from potato pulp in water operating at 85°C. Coupling electromagnetic and process design approaches, the 2kW system developed achieved good temperature control of ± 2.5°C, and a stable target temperature in ≈1 min processing time at a feed flow rate of 250 mL/min. Pectin yields of 40 - 45% were achieved, with a residence time of 0.81 s followed by 20 min coolingdown under stirring, potentially offering a vast improvement on the current batch industrial process, which operates at pH1-3 and a residence time of ≈ 1 hour.

3.2 Microwave pyrolysis

Another area of interest of our research group comes from the unique ability of microwaves to allow materials to be heated whilst maintaining a cold surrounding environment. Microwave transparent liquids are used as the inert media for pyrolysis processes, which regulate the surrounding temperature based on their normal boiling point. The low-temperature environment preserves the quality of primary depolymerisation products, and reduces the risk of thermal runaway that plagues many attempts at microwave pyrolysis [13]. We have also shown that a number of unique products can be obtained in high yield from the pyrolysis of seaweed using microwave heating [14]. The high yields arise from the lack of lignin in the feedstock and the low temperature inert environment, with products that have the potential to form a backbone of a seaweed biorefinery.

4 Outlook

A recent shift in the approach of microwave research and technology is leading to a wider body of evidence and understanding. This is increasingly identifying exactly what advantages microwave can offer in biorefinery processes, and when they can (and can't) be exploited. If embraced by the wider community, microwaves can become an essential tool in the biorefinery, supporting the move from linear petrochemical processes to a sustainable and circular economy.

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wastes to novel products for the food, pharmaceutical and chemical industries.



John Robinson is an Associate Professor in Chemical & Environmental Engineering at the University of Nottingham. He is an expert in microwave processing technologies, with 15 years of experience which spans the scale-up of continuous microwave process that operate in challenging industrial environments

through to establishing a core scientific understanding of microwave-biomass interactions.

Future Challenges and Opportunities of Emergent Technologies in Romania

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1 Introduction

Quite often, it is stated that microwave energy is extremely efficient in the selective heating of materials, since it does not heat all the bulk sample and thus less energy is wasted. Microwave heating processes are currently undergoing investigation for application in a number of fields where the advantages of microwave energy may lead to significant savings in energy consumption and process time and thus support environmental remediation. However, despite quite an important number of publications in the field of microwave heating and the AMPERE conference In Oradea, Romania (2007), applications have seen very little commercialisation in Romania. Therefore, in 2012, Primosal/Chemspeed Ltd. decided to organize the Microwave Processing Workshop: from basics to applications in Bucharest, Romania. The workshop had two main objectives: first, to introduce the microwave technology in more detail to researchers and potential (industrial) end users and second, to encourage collaborations among universities, academic organizations, and the industrial sector. The workshop was made possible due to sponsorship of Primosal, Romania and Sairem, France, and the support of the Institute of Physical Chemistry "Ilie Murgulescu" in Bucharest and of the Romanian Academy of Sciences. An important role in the success of the event was also played by the endorsement of AMPERE and the participation of invited speakers and microwave experts from the UK, Italy, and France.

The three-day event focused on many topics, such as microwave fundamentals, microwaves and their interactions with matter, microwave heating, dielectric measurements and modelling. The speakers presented examples from the area of environmental applications, biomass extraction, food processing, synthesis of nanoparticles and nanomaterials in batch and continuous flow system as tools for synthetic and mechanistic chemistry.



Figure 1: The Romanian Academy of Sciences in Bucharest, place of Microwave Processing Workshop in September 2012

Since the event, the interest in the microwave chemistry has been growing in both the business and academic sectors as well in the Romanian Academy of Sciences, see Figure 1. Romania currently has got dedicated small microwave-assisted production facilities for applications such as biomass extraction from plants yielding essential oils, and other active ingredients, for the synthesis of nanomaterials, for the residual biomass pyrolysis and for the remediation and improvement of the reaction yield of biofuels. Results were disseminated via scientific papers, patents, and presentations at national and international events.

2 The development of emergent industries within the Romanian industrial environment

Contemporary emerging technologies in Romania can be distinguished from earlier waves of technological change in three respects:

- They tend to be laboratory-based, requiring major expenditures on research that differs from traditional 'pure' or 'basic' scientific research;
- They are transdisciplinary in their underlying knowledge, therefore requiring knowledge and skills from a wide variety of specialist fields to progress from research to the production of commercially successful products;
- They are generic in scope, meaning they have a wider range of potential functions and applications than earlier waves of technological capabilities and can consequently be viewed as generic "solutions in search of problems or as platform technologies" (Department of Innovation, Industry, Science and Research, 2011).

This latter aspect also has the problematic consequence that emerging technologies tend to be supply driven, not demand-driven.

However, it is clear that Romanian industry must be reactive to the new opportunities in sectors like pharmaceutical or human health and disease prevention. In this context, emerging technologies like microwave- and ultrasound-assisted processing can offer immediate advantages in terms of easiness of automatization/digitalization, safety, and ecofriendliness. The above mentioned advantages and the fact that these technologies can be operated with more automatization, more control and safer than

corresponding conventional ones can be used as a strong argument and as an opportunity to change the mentality of big chemical companies.

In this context, the organizers of the workshop have decided to focus on a extensive educational program for new cohorts of students in the field of microwave-assisted processing.

By the end of 2020 a new pilot company will be established in Romania, focusing on microwaves and ultrasound technologies to develop new products for human health and products for daily life. Equally, Primosal/Chemspeed is going to continue their efforts in view of technology transfer to different customers.

- 3 Examples of Primosal/Chemspeed process development techniques transferred to commercial scale
- 3.1 Extraction of polyphenols from residual grape marc using microwave- and ultrasound- assisted continuous flow processing

Intensification of the extraction process of polyphenols from grape marc was demonstrated at laboratory scale to a wine grower using a simultaneous microwave/ultrasound reactor based on a 2.45 GHz, 2 kW microwave generator and a 25 kHz, 200 W focused ultrasound solid—liquid extraction (FUSLE), Figure 2. At present, Primosal/Chemspeed Ltd. and a customer are looking into the technology transfer and the economics of the process.



Figure 2: Installation for microwave (2.45 GHz, up to 2 kW) and ultrasound (25 kHz, up to 200 W) treatment in continuous flow

3.2 Microwave portable device for woodworm disinfestation

Primosal/Chemspeed Ltd. developed the MICROWOOD 12 portable device, Figure 3, a new generation of microwave-assisted technology for pest control that is expected to play an important role in treatment, preserving, and conservation of heritage objects, unmovable wood structures, cultural and ecclesiastical objects.

The construction and specific design of this device allows for obtaining very short operating time, good yield pest control, and uniform heating of treated area [1].



Figure 3: Microwood 12, 2.45 GHz, 1.2 kW

The Ministry of Culture and National Heritage Institute in Romania decided to evaluate the apparatus for the decontamination of infested wooden objects; the tests highlighted the following advantages:

- Safe to use for the treatment of work art, less expensive than existing methods, less dangerous for the environment and the operator;
- Organisms or microorganisms are heated up above their lethal temperature;
- The temperature reached by the object and its spatial variations does not provoke deformation of the treated object;
- Could be applied to objects of historical-artistic interest as: furniture, frames, musical instruments, paper (books, documents, etc.), cloth (carpets, tapestries, canvas, etc.);
- Can be used for the control of woodworms, funguses, molds;
- Consists of a flexible modular design;

- Reliable with an intelligent service and maintenance concept;
- Environmentally friendly, high control and process speeds, rapid heating to operating temperature, uniform heat distribution;
- Good efficiency of the applied energy.

The drawbacks of this process are, however, because of microwave leakage and related safety issues the microwave treatment has to be controlled remotely, which leads to additional expenditure.

3.3 Extraction of rose essential oil from fresh rose petals using microwaves

Essential oils are interesting natural plant products. In addition to many valuable qualities, they possess various biological properties such as antibacterial, antifungal, antimicrobial and antiviral properties. The uses of the essential oils for industry, health, food and fashion are vast and diverse, but usually they are divided in two large categories: general and special. At the intersection of cutting-edge research and traditional wisdom, the new era of technology opens many other applications of essential oils in the nanomaterial field with diverse uses like smart textiles, nutraceuticals and many others.

To support the development of such applications, we demonstrated the extraction of rose essential oil from fresh Rosa x damascena Mill. petals by four methods, hydrodistillation, steam distillation, organic solvent extraction, and ultrasounds followed by microwave hydrodistillation. The chemical composition of the extracts was analyzed by GC-MS, and the antioxidant capacity by DPPH. It was found that both chemical composition and the antioxidant activity of the extracts depend on the extraction method.

Overall it was found that microwaves coupled with ultrasonic treatment can be used effectively for the intensification of the extraction of monoterpenes and sesquiterpenes—fragrance bearing molecules—and equally, for increased antioxidant activity while using about extraction times 4 time shorter than the methods used for comparison and listed above. The scale-up of the method was also evaluated [2]. The results obtained in this research support the possible use of the US/MW method for the extraction of rose

essential oil for the pharmaceutical and fragrance industry, see Figure 4.

The technology was transferred to Chemarkrom, Brasov, Romania who had positive results such as:

- Increase of extraction rates;
- Improved selectivity;
- Lower extraction temperature;
- Shorter extraction time (sometimes minutes versus hours compared to classical steam distillation process);
- 100 % eco-friendly;
- Higher antioxidant activity;
- Mathematical models show a good correlation between experimental and theoretical results meaning easier and faster extrapolation at larger scales.

The drawbacks of this process are, however, its relatively high cost and the difficulty of training specialized personnel to run it.



Figure 4: Pilot for the extraction of rose essential oil from fresh petals, 2.45 GHz, up to 6 kW, 20 L reactor

4 Conclusion

The successful implementation of the microwave technology in the Romanian industry is quite complex today. Primosal/Chemspeed is determined to be the link between the researchers, the microwave equipment manufacturers, and the end users. Our main role consists of sustaining the technology transfer and its implementation by being the main contact between the researchers,

microwave manufacturers and end users, by making available enough in-depth and hands-on explanation about the equipment and training to allow for correct and safe operation.

For further reading

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Ricky's Afterthought:

DATA, DARK DATA and their STORAGE

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Picture yourself in your laboratory conducting the next set of experiments following a particular line of results. After a while, you have obtained a series of results of two interdependent parameters y and x. You start plotting y versus x as you go along in order to get a feel of the trend of the relationship. For small values of x in the abscissa, the ordinate seems to be very high and as x increases y gets progressively smaller. Well, you reflect this is a classic exponential decay so you continue to take additional readings and after a while, you take one or two reading with very large x to confirm that y gets extremely small and indeed this is exactly what you find. However, you left a gap where you deemed it unnecessary to fill with more points as it appears obvious what the relationship is. However, are you sure that such a rash decision is a prudent one? What if between two points, say x_1 and x_2 , there is a peak which you have missed as shown in Figure 1 and you have assumed that the dashed line represents the true measurement.

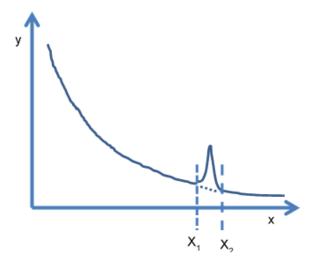


Figure 1: Scheme of an exponential relationship between y and x with significant local deviation

Something akin to this example happened to me many years ago as a Post-Doctoral Fellow when I was carrying out mass-spectrometric analysis of species emerging from a special ion source I had constructed. The range of masses I was observing in the quadrupole mass spectrometer were up to 40. Following graphene's discovery decades later, I revisited my set of results to see if a peak around mass number 12.1 had been observed. Alas it was not in the traces but I daresay had I found, at the time of my experiments, a small peak around that mass number due to tiny fragments of graphene that had been generated in the ion source, I would have dismissed it as spurious. What a mistake that would have been!

In a recent book by Professor David Hand¹ he states that dark data may come in a variety of forms from such as blanks in a data set, data one did not think necessary to collect as in the first example, data that occur by hastily rounding off a set or data that could be deliberately hidden for a variety of reasons.

Professor Hand points to some striking examples of dark data. One involved the calculation of the problem of the seals between the segments of the booster rockets in the Challenger Space Shuttle. Such calculation omitted the data when there was no problem. Had they included such data when there was no problem that would have resulted in the delay of launching the Shuttle and thus averting the disaster that occurred with the loss of the astronauts' lives. To bring the argument closer to engineers and physicists, the latter use the term "cosmic variance" to indicate that often they make assessments about the whole universe when only a fraction of it can be seen and only at one time.

Professor Hand asserts that the fifteen types of dark data are as follows:

- Data we know are missing
- Data we do not know are missing
- When one chooses just some cases
- Self-selection data
- Missing what matters
- Data which might have been
- Data which changes with time
- Definitions of data
- Summaries of data
- Measurement of error and uncertainty
- Feedback and gaming of data
- Information symmetry
- Intentionally darkened data
- Fabricated and synthetic data
- Extrapolating beyond the data one has

We use the term dark data to mean data that have not yet been analysed, or data kept in filing cabinets for a very long time. One can term such dark data as benign. However, what is more worrying is when the data are being generated so fast that one has to make a selection on the spot of which to keep and disastrous ignore possibly with which to consequences. Ethan Siegel in an article in Forbes in Sept 2018, points to the case of data being lost from the Large Hydron Collider (LHC) at CERN. Protons whizzing around at opposite directions in the LHC at practically the speed of light collide at points where detectors are situated to detect the ensuing new particles. Out of every one million collisions that occur at the LHC, only about 30 of them have all of their data written down and recorded, meaning a massive 99.997% of the data were lost for ever. Although nuclear physicists point out that what is monitored and stored are the more important of the collisions, one has to ask whether the vast data that are not recorded contain some important information but we will never know.

Professor Hand points out that dark data derives from the term dark matter which is used by

astrophysicists. Dark matter is the most mysterious, non-interacting substance in the universe. Its gravitational effects are necessary to explain the rotation of galaxies and the motions of clusters. It is said that without dark matter, the universe would likely have no signs of life at all.

Dark data arise in a number of ways. There may be a limitation of the recording instrument. Or they may come too fast for recording or indeed they may be hidden from medical records because of the sensitivity and people are reluctant to divulge any relevant information. People react differently when political volunteers canvass for some data and often it is on the understanding that such data may or may be not used. This could lead to overall distortion of the data set. How about using the data that one has, to find out about data that one does not have. Statisticians use this concept to delve into this matter.

Finally, let me state some mind-boggling statistics regarding data in general. IBM reckons that around 90% of data from sensors and analogue to digital conversions are never used. Most industrial companies collect data for a variety of reasons and only analyse 1%. Often the data collected do not come into distinct categories so that adds to the complication of examining some of these so they are left in storage with the intention of someday being analysed but that day never comes. What is more, storing data can be very energy consuming. It is estimated that in all the countries in Europe, Middle East and Africa the cost of storage and its management could cost nearly \$891 billion.

So next time you are in the laboratory ready to collect some experimental data do reflect on the notion of data acquisition, dark data and the long-term storage of data.

For further reading:

1. "Dark Data: Why What You Don't Know Matters", by David J Hand, Princeton University Press, 22 January 2020.

Upcoming Events



Workshop on High-Power Microwave Industrial Applications at the European Microwave Week 2020.

10th -15th January 2021, Utrecht, Netherlands

This workshop addresses the increasing industrial application s of high-power microwaves. In 2009, the IEEE MTT-S IMS Workshop "Recent Advances in Microwave Power Applications and Techniques (RAM-PaNT)" received significant interest among IMS attendees and won the "Best Quality Workshop" Award. The RAMPAnT Workshop was designed as an inauguration forum that introduced the MTT community to microwave energy applications in science and industry. For the past 10 years, topics related to this Workshop have gradually increased in scope. This Workshop proposed for the EuMW2020 will review the recent advancements in industrial and scientific applications of high-power microwave technology. The discussions will cover well-established systems and processes as well as new trends and emerging applications: beyond well-known microwave heating of food products, they include powder metallurgy (include sintering of particulate materials), microwave-assisted chemistry, microwave plasma generation, manufacturing of nanomaterials and composites (including microwave-assisted 3D printing), waste-to-fuel conversion, etc. Topics that support many of the applications, such as advanced multiphysics modeling and accurate characterization of material parameters, will also be discussed. The Workshop includes ongoing developments of solid-state technology and prospects of the use of solid-state generation in high-power applications for more flexibility and control.

Website: http://eumw2020.org/

Call for Papers: Special Issue on



Advances in Microwave Processing of Materials

The open-access Journal materials published by MDPI has launched a new Special Issue, titled:

Advances in Microwave Processing of Materials

Over the last several decades, high-power microwave technology for materials processing has been an emerging topic in research as well as in industrial applications. Microwave heating enables time and energy savings in numerous industrial applications, particularly when large sample volumes and/or materials with low thermal conductivity have to be heated. About 50% of global energy consumption is used for heating, resulting in 40% of the global carbon dioxide (CO₂) emissions. Thermal processes in industry are responsible for about 50% of that. Efficient use of microwave technology in thermal processes can therefore have a significant impact on the remediation of climate change, particularly if renewable energies are used for microwave generation. This drives research and development in the field of microwave processing, which is attracting growing political and industrial interest.

Nevertheless, the potential benefits of microwave applications, which have been intensively demonstrated in numerous lab-scale experiments, are accompanied by significant challenges (e.g., temperature uniformity and process control) when

upscaled. Successful upscale typically requires detailed knowledge of material behavior during the process, which requires in situ dielectric characterization under process-relevant conditions, process simulation, and experimental validation.

This Special Issue will survey recent progress in microwave processing of materials. The articles in this Special Issue will cover topics such as:

- dielectric characterization
- process simulation
- design of industrial microwave applicators
- process control methods.

Processes may include (but are not limited to):

- high-temperature processing of inorganic materials such as ceramics, glasses, or metals
- processing of organic materials
- microwave chemistry.

This Special Issue will offer a unique glimpse of what has been achieved and what remains to be explored.

For further information:

This Special Issue is now open for submission at: https://www.mdpi.com/journal/materials/special_iss ues/microwaves_process_mat

or directly contact the Guest Editor, Dr. Guido Link, guido.link@kit.edu

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AMPERE Newsletter welcomes submissions of articles, briefs and news on topics of interest for the RF-and-microwave heating community worldwide, including:

- Research briefs and discovery reports.
- Review articles on R&D trends and thematic issues.
- Technology-transfer and commercialization.
- Safety, RFI, and regulatory aspects.
- Technological and market forecasts.
- Comments, views, and visions.
- Interviews with leading innovators and experts.
- New projects, openings and hiring opportunities.
- Tutorials and technical notes.
- Social, cultural and historical aspects.
- Economical and practical considerations.
- Upcoming events, new books and papers.

AMPERE Newsletter is an ISSN registered periodical publication hence its articles are citable as references. However, the Newsletter's publication criteria may differ from that of common scientific Journals by its acceptance (and even encouragement) of news in more premature stages of on-going efforts.

We believe that this seemingly less-rigorous editorial approach is essential in order to accelerate the circulation of ideas, discoveries, and contemporary studies among the AMPERE community worldwide. It may hopefully enrich our common knowledge and hence exciting new ideas, findings and developments.

Please send your submission (or any question, comment or suggestion in this regard) to the Editor in the e-mail address below.

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AMPERE Newsletter

ISSN 1361-8598

https://www.ampereeurope.org/newsletter/