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CAETS

EDITOR-IN-CHIEF'S WORD

A few years ago, in line with its dedication to the popularisation of technological and biotechnological sciences, the Croatian Academy of Engineering introduced a new, thematic conception of its Bulletin „Engineering Power", which sought to enhance the public profile and awareness of the importance of these fields of science through presentation of various scientific projects on which the distinguished members of our Academy were working as project leaders and principal investigators, respectively.

The new conception of our Bulletin has proved very successful and well-accepted among our members, and their rising interest often provided the Editorial Board of the „Engineering Power" with several thematic contributions from our members at once, lined up to be published subsequently. This year's volume of „Engineering Power" will not be an exception, as we plan to publish at least three issues of our Bulletin in English as well as at least two issues of our Bulletin in Croatian, the „Tehničke znanosti".

I believe that the following articles will be most interesting especially to our readers from the fields of food technology and biotechnology as well as very edifying to our membership in general, and to all other interested parties.

Editor-in-Chief
Vladimir Andročec, President of the Croatian Academy of Engineering

EDITOR'S WORD

The importance of food industry, which is largely a traditional (low-tech) industry, for quality of life in modern society cannot be overstated. Beside direct impact to standard of living due to the very essence of its products, it is also of high economic importance. According to documents released within FP7-funded Network of Excellence "HighTech Europe – European Network for integrating novel technologies for food processing", the food industry sector employs over 4 million people and is the largest manufacturing sector in Europe. Moreover, the growing human population as well as the ever-rising living standards in increasingly prosperous developing world will intensify demand for food at the global level, emphasising even more the importance of food industry in times to come.

To satisfy this demand, the key-role of innovation for food industries and pertinent organisations is generally acknowledged. Therefore, it is not surprising that one of the first topics that rose as relevant within the framework of EU Scientific Advice Mechanism (that provides independent and transparent advice to European Commission working with High-Level group of Scientific Advisors and European Academies, including Croatian Academy of Engineering via Euro-CASE academy network), is related to the food sector.

To this end, it is my great pleasure to present this issue of Engineering Power, focused on the novel technologies in food industry, to our readers. The issue is guest-edited by Drago Šubarić, Member of the Croatian Academy of Engineering, Department of Bioprocess Engineering, and Dean of the Faculty of Food Technology, Osijek.

Editor
Zdravko Terze, Vice-President of the Croatian Academy of Engineering

THE POSSIBILITY OF UTILISING FOOD INDUSTRY BY-PRODUCTS WITH THE USE OF GREEN TECHNOLOGIES – AN INNOVATIVE APPROACH

GUEST EDITOR'S WORD

Dear Readers,

Food producers are meeting numerous challenges nowadays, starting from increasing world population, eating habits, climatic changes, use of agricultural products in energy production, migration, increasing energy costs etc. Along with all these challenges and limited resources, food producers are obliged to produce sufficient quantities of safe and high quality food for the increasing world population. On the other hand, food industry generates large quantities of by-products that represent a large environmental problem, solved in most cases through landfills, composting, or animal feed.

A large quantity of contemporary research deals with this issue and the top subject of many documents is the utilization of food industry by-products as potential raw materials for food. The reasons for this include the fact that many by-products contain a variety of nutrients, making them valuable as raw materials in the production and development of new products, among other reasons such as increasing food prices, large quantities of generated by-products, increasing cost of waste management, and increasing environmental concerns. Maintaining the quality of a product requires constant generation of certain quantities of by-products. These quantities are constantly growing, as the result of the increasing food production.

The application of food industry by-products in food production results in various changes in products, depending on both the properties of the by-product, which includes the mode of application, and production conditions. To develop a product with desirable organoleptic characteristics, one has to know the properties of the raw materials and processes, and how to adjust recipes and introduce new technologies and/or processes, in order to obtain products as similar to the original as possible.

During the realization of the project *Application of Food Industry By-products in the Development of Functional and Environmentally Friendly Extruded Food Products and Additives* (funded by the Croatian Science Foundation), we used raw materials and technologies that enabled us to develop products with increased nutritional value and desirable organoleptic characteristics. "Green" technologies (supercritical CO_2 extraction, extrusion with supercritical CO_2) were used in by-product preparation and product finalization in order to obtain safe, high quality products and modified half-products that may be used in food production.

I hope you will enjoy reading these articles!

Sincerely

Guest-Editor
Drago Šubarić, Josip Juraj Strossmayer University of Osijek, Faculty of Food Technology Osijek

Antun Jozinović[1], Đurđica Ačkar[1], Jurislav Babić[1**], Borislav Miličević[1], Stela Jokić[1], Drago Šubarić[1**]*

THE APPLICATION OF SOME FOOD INDUSTRY BY-PRODUCTS IN THE PRODUCTION OF EXTRUDED PRODUCTS

Josip Juraj Strossmayer University of Osijek, Faculty of Food Technology Osijek, Franje Kuhača 20, HR-31000 Osijek, Croatia (*ajozinovic@ptfos.hr)
**Croatian Academy of Engineering, Zagreb, Croatia

Abstract

Various by-products of the food industry (fruit and vegetable pulp, oilseed cake, different bark and shells...) are generated in large quantities and represent a huge problem for manufacturers, because they have a significant effect on the environment due to slow biodegradation, water pollution, emissions of methane, and related environmental problems. On the other hand, these by-products, aside from the fact that they are a rich source of polyphenols, dietary fibre, β-glucans, antioxidants, and essential fatty acids, represent cheap and easily available raw materials which could be used for the production of various products consumed by many people (snacks, breakfast cereals, pasta, bread, biscuits...). One of the most important product groups are certainly extruded products, and their production mainly uses various types of grains, such as corn, wheat, rice and barley, which are rich in carbohydrates. Therefore, in recent years there is much more research on the topic of improving the nutritional value of this product group by using various food industry by-products.

CONTENTS

1. INTRODUCTION

The food processing industry generates a huge quantity of by-products, including pomace, peel, husks, pods, stems, bran, washings, press cakes, etc., which have a lower production value and create considerable environmental pollution (Sharma et al., 2016). The use of these by-products has become a growing trend in the food industry in recent years, including the production of extruded products. One of the motives is to increase the nutritional value of new products, since the consumers are aware of the close relationship between nutrition and health, so the demand for food enriched with nutritionally valuable ingredients and functional food is becoming a growing trend. Another motive is certainly the utilization of these nutritionally valuable raw materials, thereby reducing overall waste (Jozinović et al., 2014; Yağcı and Göğüş, 2010).

2. THE UTILISATION OF FOOD INDUSTRY BY-PRODUCTS IN THE PRODUCTION OF EXTRUDED PRODUCTS

Extruded products include a large group of various types of finished products consumed by many people (snacks, breakfast cereals, pasta, confectionery, etc.), as well as modified flours and starches, which are widely used in the food industry as additives. Due to the fact that the extrusion process allows processing of various types of raw materials, numerous by-products, mainly of plant origin, have found their application in the production of various types of extruded products. This paper provides a brief overview of the most commonly used by-products in previous research, as well as some of the possibilities for their application established within the research project "Application of Food Industry By-products in the Development of Functional and Environmentally Friendly Extruded Food Products and Additives" (FUNEXFOOD) (HRZZ-1321).

2.1. By-products of fruit and vegetable processing

Apple pomace is a major by-product, comprised of the remains left after crushing and pressing apples during the production of juice, vinegar, or cider, and it contains about 30% of the whole fruits. It is a wet by-product, mostly used as animal feed or as fertilizer, also as a source of pectin, dietary fibres, and polyphenols (Jozinović et al., 2014). Along with citrus peel, it represents basic raw material for the production of pectin, but it is important to note that pectin from apples can't be used for the production of very light products, because its colour contains a slight brown hue (Schieber et al., 2001). Furthermore, it is a great source of dietary fibres (51.1% of total dietary fibres), 36.5% insoluble, and 14.6% soluble fibres in dry matter. Several studies about the application of apple pomace in the production of extruded products were carried out so far, such as corn extrudates (Karkle et al., 2012; Jozinović, 2015), extrudates based on whey proteins (Sun et al., 2015), and pregelatinized corn starch (Paraman et al., 2015).

Grape pomace is the main by-product of wine production, and it represents about 20% of processed raw materials. It is a good source of dietary fibres, mainly cellulose, and small proportions of pectin and hemicellulose. Furthermore, it has also been evaluated as a source of antioxidants because of its high polyphenol content (Jozinović et al., 2014). Grape pomace contains pulp, seeds, skins, and stems, and since it is a valuable raw material rich in phenolic compounds, ethanol, tartaric acid, citric acid, grape seed oil, dietary fibres, etc., it could be used as raw material for the isolation of those groups of compounds, with the potential application in functional food production (Teixeira et al., 2014). Therefore, dry pomace as grape flour has found its application in the production of extruded products based on barley (Altan et al., 2008b; 2009) and whey proteins (Sun et al., 2015), as well as in the production of noodles (Rosales Soto et al., 2012).

Citrus pomace is generated in large quantities as a by-product of processing this fruit during the production of juice, and it mainly consists of peel and seeds. Aside from their use in the production of pectin, citrus peel and seeds have significant antioxidant potential, so their application has recently been steadily increasing (Jozinović et al., 2014). Thus, orange peel modified by extrusion was used for the production of cookies based on wheat flour (Larrea et al., 2005). Furthermore, it was found that by extruding lemon pomace, insoluble fibres could be transformed into soluble fibres (García-Méndez et al., 2011), and the successful application of mango peel has been observed in the production of macaroni (Ajila et al., 2010).

Tomato pomace is generated in the amount of 3 – 7% during tomato processing in the production of juice, paste, puree, ketchup, sauce, and other products. It contains a large proportion of dietary fibres, which represents up to 50% d. m. of this by-product, but it is also a good source of high-quality ingredients, such as lycopene. The application of tomato pomace has been recorded in the production of extruded snack products based on corn, rice, and wheat (Dehghan-Shoar et al., 2010), extrudates based on barley (Altan et al., 2008a), and corn extrudates (Da Costa et al., 2010; Huang et al., 2009; Caltinoglu et al., 2014; Obradović et al., 2015).

Carrot pomace is a by-product obtained during carrot juice processing. Considering that a large portion of valuable components, such as carotenoids and dietary fibre, are retained in the pomace, this valuable by-product is a good source of α- and β-carotene. The total dietary fibre content of carrot pomace was found to be 63.6% d. m, with 50.1% d. m. being the insoluble fraction and 13.5% d. m. the soluble fraction. Considering that carrot pomace doesn't contain kernels and seeds, it has a wide application in numerous products such as bread, cakes, dressing, and functional drinks (Schieber et al., 2001), and furthermore, its application in the production of "ready-to-eat" expanded products has been recorded (Kumar et al., 2010), as well as in the production of other extruded products (Upadhyay et al., 2010; Obradović, 2014).

2.2. Oilseed cakes

Oilseed cakes are generated as a by-product in the production of oil. Considering that the annual amount of processing of various types of oilseeds in the EU reaches 30 to 35 million tons, these by-products are created in large amounts, but they are mainly used as animal feed. They represent a rich source of protein and dietary fibres, so their application in the production of various types of food products started in the recent years, mainly in bakery (bread, biscuits, tortillas) and protein supplements (Lai et al., 2017). Some of the most important applications in the production of new products are certainly related to the production of extruded products, so the application of hazelnut cake has been recorded in the production of snack products (Yağcı and Göğüş, 2008; 2010), the application of sesame oil cake (Nascimento et al., 2012) and olive paste in the production of corn extrudates (Bisharat et al., 2013), as well as the application of hemp powder in the production of rice extrudates (Norajit et al., 2011).

2.3. Hulls and bran

Hulls and bran represent the outer shells of different types of grains and oilseeds. They are a rich source of dietary fibres, preferably insoluble, and therefore, they have found application in the production of different extruded products. Thus, the applications of wheat bran (Onwulata et al., 2001), soybean hulls (Duarte et al., 2009), and corn fibres (Wang and Ryu, 2013a; 2013b) have been recorded in the production of corn extrudates.

2.4. Brewer's spent grain

Brewer's spent grain (BSG) is the major by-product of the brewing industry. Although it is a rich source of protein (about 20% d. m.), dietary fibres (17% cellulose, 28% non-cellulosic polysaccharides, chiefly arabinoxylans, and 28% lignin in dry matter), and ß-glucans, it is mostly used as animal feed (Jozinović et al., 2014). Mussatto et al. (2006) observed that this by-product could be used for enrichment in the production of flakes, whole-wheat bread, biscuits, aperitif snacks, and other products, but before its application it should be dried and ground. Its application has been recorded in the production of various types of extruded products, such as baked snacks (Ktenioudaki et al., 2013), corn extrudates (Makowska et al., 2013; Ainsworth et al., 2007; Jozinović, 2015), and "ready-to-eat" expanded products (Stojceska et al., 2008).

The main limitations of its implementation are the significant effects on colour and flavour when it is used in higher amounts.

2.5. Sugar beet pulp

Sugar beet pulp (SBP) represents a major solid by-product of the sugar beet used in the sugar refining industry. The content of this valuable by-product is 20-25% cellulose, 25-36% hemicellulose, 20-25% pectin, 10-15% protein, and 1-2% lignin in dry matter. Although it is high in pectin content, pectins from SBP have poor gelling properties due to their high degree of methylation and low molecular weight, so they are not extensively used in the food industry when compared to citrus and apple pectins (Jozinović et al., 2014). The application of this valuable by-product in the production of extruded products has been recorded for the development of corn extrudates (Lue et al., 1991; Jozinović, 2015), and moreover, it is used in the production of spaghetti (Özboy and Köksel, 2000).

Table 1. Some examples of the application of food industry by-products in the production of extruded products*

 Apple pomace		• corn extrudates • extrudates based on whey protein • extrudates based on pregelatinised corn starch
 Grape pomace		• extruded products based on barley • extrudates based on whey protein • noodles
 Citrus pomaces		• extruded orange peel in the production of cookies based on wheat flour • extrusion of lemon pomace with the aim of transforming insoluble fibres into soluble fibres • mango peel application in the producti-on of macaroni
 Tomato pomace		• snack products based on corn, rice and wheat • extrudates based on barley • corn extrudates
 Carrot pomace		• "ready-to-eat" expanded products • corn extrudates
 Oilseeds cakes		• snack products with hazelnut cake • corn extrudates with sesame oil cake, and olive paste • rice extrudates with hemp powder
 Hulls and bran		• corn extrudates with the addition of wheat bran, soybean hulls, and corn fibres
 Brewer's spent grain		• baked snacks • corn extrudates • "ready-to-eat" expanded products
 Sugar beet pulp		• corn extrudates • spaghetti

*photos in the table are the property of the authors

3. EXTRUDED PRODUCTS DEVELOPED AS PART OF THE PROJECT FUNEXFOOD

During the investigation for the project "Application of Food Industry By-products in the Development of Functional and Environmentally Friendly Extruded Food Products and Additives", 9 new expanded snack products have been developed so far, with acceptable sensory properties, enriched with three food industry by-products: corn snack products with 5, 10, and 15% of BSG, 5, 10 and 15% of SBP, and 5, 10 and 15% of apple pomace (**Figures 1 and 2**) (Jozinović, 2015). Furthermore, current research on the project is focused on the development of new extruded products based on corn grits, produced by extrusion with supercritical CO_2, enriched with defatted hazelnut cake, pumpkin seed cake, hemp cake, and defatted *Camelina sativa* cake. Moreover, 10 new modified wheat and barley flours were developed as part of the project so far, enriched with various by-products (SBP, BSG, and apple pomace), these flours will be used in further investigations on the project, in order to produce enriched breads and cookies.

Figure 1. Corn snack products developed as part of the project FUNEXFOOD (from left to right: Corn grits (without added by-products); Corn + 10% brewer's spent grain; Corn + 10% sugar beet pulp; Corn + 10% apple pomace

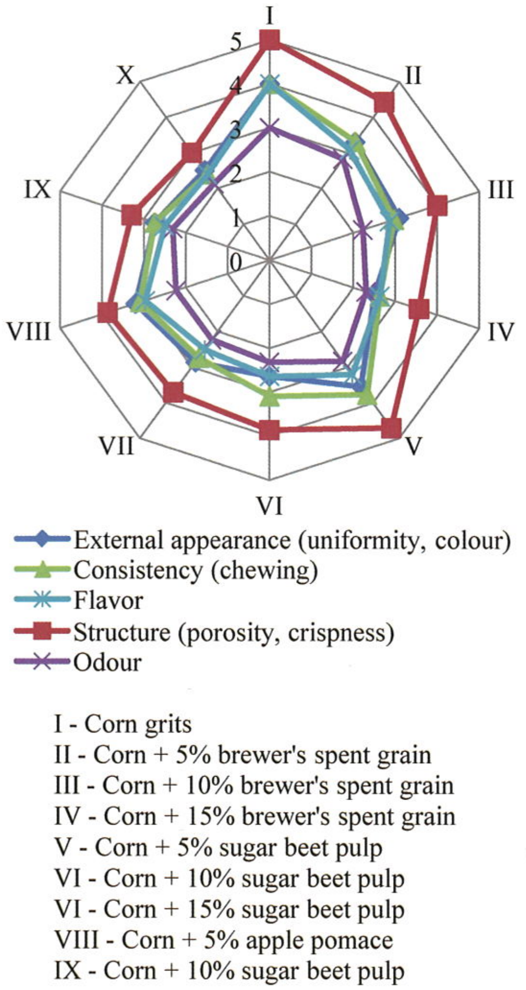

Figure 2. Sensory evaluation results of the corn snack products developed as part of the project FUNEXFOOD (HRZZ-1321) (Jozinović, 2015)

4. CONCLUSION

The trend of improving the nutritional value of various types of food products, including extruded products, is increasing as a result of increased consumer awareness about the importance of healthy diets and the increased demand for so-called functional products. On the other hand, the food industry is faced with the problem of generating large quantities of by-products, which are usually classified as waste, even though they are rich in nutritionally valuable components and can be used in the production of new products. This paper presents a part of the potential utilization of various by-products in the production of extruded products, along with examples provided by other researchers and the examples provided by the research team on the project FUNEXFOOD.

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Stela Jokić[1], Krunoslav Aladić[2], Đurđica Ačkar[1], Antun Jozinović[1], Jurislav Babić[1**], Drago Šubarić[1**]*

SUPERCRITICAL CO_2 EXTRACTION – A NEW PERSPECTIVE IN THE UTILISATION OF FOOD INDUSTRY BY-PRODUCTS

[1]Faculty of Food Technology, Josip Juraj Strossmayer University of Osijek, F. Kuhača 20, 31000 Osijek, Croatia (*stela.jokic@ptfos.hr)
[2]Croatian Veterinary Institute, Branch – Veterinary Institute Vinkovci, J. Kozarca 24, 32100 Vinkovci, Croatia
**Croatian Academy of Engineering, Zagreb, Croatia

Abstract

The food industry, same as any other industry, aims to utilise raw materials as efficiently as possible in the production process, but it also tries to create minimum amounts of waste, where waste is not necessarily "waste", but a by-product or a raw material for some subsequent process. Today there is great interest in utilising food industry by-products for various purposes because they contain many potentially useful substances, and they could become significant raw materials in the production/development of new products due to that fact. This paper places special emphasis on the influence of modern green extraction techniques, like supercritical CO_2 extraction, on the utilisation and extractability of certain biologically active components from selected by-products which have an application in food, cosmetics, and pharmaceutical industries.

1. INTRODUCTION

The human awareness regarding the protection of nature and the environment is growing daily. Sustainable development, one that meets today's requirements and does not jeopardise the needs of future generations, has become an important issue, and all of the research on reducing the use of toxic chemicals, conserving energy, and managing waste and by-products is welcome at the international level. All over the world, there is research dedicated to this area, demanding that each researcher follows the tradition of sustainable development directed at a certain area, which also includes long-term decision making in the agriculture and food production sectors. The high quality of food production and the commitment to environmental protection has contributed to the successful marketing of agriculture and food products (Perretti, 2006). Large quantities of solid (shells, peels, seeds, leaves) and liquid waste (wastewater) are created during the processing of raw materials from plants in the food industry every year, and their storage, processing, or management present a serious ecological and economic problem. The large quantities of by-products created every day are mostly managed through disposal sites or by making cattle fodder. Therefore every industry, including the food industry, has the goal of fully using their raw materials in the production process, while creating as little waste as possible, where waste is not necessarily "waste", but a by-product or a raw material in some future process. Today there is great interest in using by-products from the food industry for various purposes because they contain many potentially useful substances, and they could represent significant raw materials in the production/development of new products (Schieber et al., 2001; O'Shea et al., 2012).

The use of food industry by-products has become extremely interesting because they represent cheap and nutritionally valuable raw materials, and that is enough to affect the reduction in waste. From the economic standpoint, large amounts of by-products, especially grape pomace and olive pomace, are still not being used in the best possible way in the Republic of Croatia. Furthermore, there is no competition on the domestic market in the area of highly profitable final products that could be created from those initial raw materials, and they are used for some of the most highly sought after food, cosmetics, and pharmaceutical products in the world.

Modern extraction techniques, like supercritical CO_2 (SC-CO_2) extraction, that represent new, mild, and selective food processing technologies, with the purpose of environmental protection, are becoming more prominent in the world. In recent years, this technique has been applied in the extraction of potentially useful substances from food by-products (**Table 1**).

2. SELECTED FOOD INDUSTRY BY-PRODUCTS

2.1. Grape pomace

Grape pomace created in the process of wine production, as well as olive pomace created in olive oil production, are just some of the potentially valuable by-products that could be successfully used in the production of some functional food products, but also in creating various extracts rich in active substances (Palma and Taylor, 1999; Ibáñez et al., 2000; Marti et al., 2001; Louli et al., 2004). Grape pomace represents a major problem due to its quantity and properties, primarily the leftover acids that make processing, usually composting, more difficult. If the pomace is not treated properly, it can represent a serious risk for the environment, starting with surface and deep pollution, to unpleasant odours created if it is left unprocessed. Na-

Table 1. Selected examples of SC-CO_2 extraction of high-value food industry by-products of plant origin

Sample	Extraction parameters						Analyte	Reference
	Temperature (K)	Pressure (MPa)	Time (h)	Flow rate (g/min)	Co-solvent (%)	Particle size (mm)		
Grape seed	313	16 18 20	---	10.2	---	0.75	Grape seed oil	Passos et al. (2008)
Grape seed	310 353	25	7	---	---	1.125 0.638 0.363	α-tocopherol enriched oil	Bravi et al. (2007)
Grape seed	313 323 333	20 30 40	1.5	32.33	---	0.380	Grape seed oil	Jokić et al. (2016)
Grape pomace	308 328	10 40	3	0.8	Ethanol (5)	0.165 0.261 0.319	Resveratrol	Casas et al. (2010)
Grape pomace	318	10 15 25	---	18.3	Methanol (5)	---	Phenolic compounds	Louli, Ragoussis and Magoulas (2004)
Grape skin	313	15	0.25	2	Ethanol (7.5)	---	Resveratrol	Marti et al. (2001)
Elderberry pomace	313	21	1.6	---	CO_2 (0-90) Ethanol (0.5-100) H_2O (0–95)	---	Anthocyanins	Seabra et al. (2010)
Apricot by-products	316–350	13.3–47.3	1.5	1	Ethanol (2–28)	0.07-0.6	β-carotene	Sanal et al. (2005)
Apricot seed	313-343	30-60	---	1-5	Ethanol (0-3)	0.425-1.5	Apricot seed oil	Özkal, Yener and Bayındırlı (2005)
Peach seed	303 313 323	10 20 30	2.5	8.3	Ethanol (2.5)	0.25-0.35	Peach seed oil	Mezzomo et al. (2010)
Tomato skin	313-373	20-40	1.5	1-2	---	---	Lycopene	Chun et al. (2009)
Tomato skin	313 343	25 45	0.17-0.33	6.38	Ethanol (5.15)	0.5-1	Lycopene	Kassama, Shi and Mittal (2008)
Tomato skin and seed	313 333 353	30 38 46	---	---	---	0.3 0.4 0.6	Carotenoids, tocopherols sitosterols	Vagi et al. (2007)
Apple and peach pomace	313–333	20–60	0.17-0.67	2	Ethanol (14–20)	0.638	Phenolic compounds	Adil et al. (2007)
Cherry seed	313-333	18-22	---	---	---	1.25-2.25	Cherry seed oil	Bernardo-Gil et al. (2001)
Orange peel	293 – 323	8-28	---	8.3-58.3	---	0.1-10	Essential oil	Mira et al. (1999)
Orange peel	313	10	1	29.3	---	0.324	Essential oil	Jerković et al. (2015)
Citrus peel	333	9.5	0.75	---	Ethanol (15)	---	Naringin	Giannuzzo et al. (2003)
Olive pomace	323	35	3	33	Ethanol (10)	---	Tocopherol	Ibáñez et al. (2000)
Olive pomace	313-323	10-30	2.5	1.8-2.7	Ethanol (10)	0.30-0.55	Olive oil	De Lucas, Rincon and Gracia (2003)
Cocoa shell	323 358	15-45	---	---	---	2-4	Theobromine	Rossi (1996)
Hemp pressed cake	313 323 333	20 30 40	1.5-7.5	11.7 29.1 46.7	---	---	Hemp oil Defatted cake	Aladić (2015)
Rice by-products	353	68	---	1.082	---	---	Tocochromanols Oryzanols	Perretti et al. (2003)

mely, large quantities of pomace attract vermin and flies, and can lead to occurrences and spreading of various diseases. Tannin solutions and solutions of other wine pomace components, which are separated during resting, can cause the reduction of oxygen levels in the soil, but they can also penetrate into the soil and groundwater (Voća, 2010). If the pomace is used as fertilizer in vineyards, the soil in the vineyards becomes acidic over time, which is also a problem. Because of that, more and more attention is dedicated to processing waste or by-products from wineries that mostly consist of solid bio products (skins, stems, and seeds), which comprise on average 20-30% of the mass of the processed grapes, and 15% of the solid waste are grape seeds. Its use in the food industry may contribute to the reduction of production costs, as well as to the development of new products. The extraction process using SC-CO_2 can be used, for example, to completely extract oil from grape seeds, which is very significant in industrial processes, because it creates minimal losses in the production process and the full utilization of the initial raw material (Jokić et al., 2016). Oil obtained by using SC-CO_2 extraction is greenish-yellow in colour with a characteristic aroma; it is rich in vitamin E and can be used for cooking, medicine, and in pharmaceutical and cosmetic industry. The proportion of linoleic acid is higher when compared to any other oil, like sunflower or corn oil, which makes grape seed oil suitable for storage because it is highly stable. Defatted grape seed flour, which is created as a by-product of SC-CO_2 extraction, can also be used for other purposes (in making enriched extruded products, bakery industry, etc.). Grape seed pomace is also characterised by high phenolic compounds content due to low extraction during the wine production process. In the total amount of phenolic substances that can be extracted from grapes, 10% is in the pulp, 60-70% in the grape seeds, and 28-35% in the grape skin (Palma and Taylor, 1999; Murga et al., 2000; Louli et al., 2004) so the tablets and capsules based on extracts rich in polyphenolic compounds with antioxidant properties could be developed.

2.2. Olive pomace

Another by-product created in the process of olive oil extraction is olive pomace that includes skins and seeds. By-products of olive processing are rich in antioxidants, and various extraction techniques are used to isolate them from the plant material (Ibáñez et al., 2000; De Lucas et al., 2003). The use of the appropriate green technology, such as SC-CO_2 extraction, can result in the production of oil from olive pomace, where the quality and utilization of oil would be significantly higher than with using the standard pressing procedure. The goal is to get completely defatted olive pomace created as a by-product of SC-CO_2 extraction, which could then be used for the development of new functional products, i.e. in the process for the production of corn snack products enriched with defatted pomace.

2.3. Apricot kernels

Apricot kernels as by-products of processing are a valuable source of amygdalin (in literature often referred to as vitamin B17). Aside from that, apricot kernel seeds contain a high proportion of oil rich in mono- and polyunsaturated fatty acids, and a series of ingredients with lower proportions, like tocopherols and phenolic compounds (Turan et al., 2007). The oil extracted from apricot kernels using SC-CO_2 may have significant applications in cosmetics.

2.4. Cherry seeds

Cherry processing generates significant amounts of kernels for which there is no further use. However, since cherry seeds are a valuable source of oil (which can be obtained by SC-CO_2 extraction) rich in essential fatty acids, as well as in compounds like carotenoids, tocopherols, squalene, phytosterols, etc., they can be utilised as a potential natural source of nutraceutical ingredients in the pharmaceutical industry (Bak et al., 2009; Górnas et al., 2015).

2.5. Citrus peel

Three main by-products of citrus are dry pulp, molasses, and citrus oil (from the peel). Essential oils from citrus have many applications as aromas and for aromatising various products (Kesterson and Hendrickson, 1958). Essential oils can be produced from selected varieties of citrus peels by using various extraction techniques, where SC-CO_2 extraction is the preferred technique (Jerković et al., 2015).

2.6. Coffee processing by-products

By-products of coffee processing, such as mucilage and parchment, have been less studied, despite the fact that they contain important bioactive components (Esquivel and Jimenez, 2012). They can be an alternative source of natural antioxidants. Cocoa shells, which are removed during the processing of cocoa beans (*Theobroma cacao* L.), represent at least 10% of the weight of cocoa fruit (Owusu-Domfeh, 1972). In countries that produce cocoa beans, processing of such waste can provide an economic advantage and reduce the extent of environmental problems. The cocoa bean shell is also a potential source of theobromine (Hartati, 2010), which can be extracted using SC-CO_2, since it has a great pharmacological function, it is a powerful diuretic, it promotes diuresis and stimulates circulation to the kidneys, and consequently helps to remove harmful substances from the urinary tract.

2.7. Tomato waste

A significant amount of waste is accumulated during the tomato canning process; it consists of seeds, skins (peel), and a small quantity of pulp (Avelino et al., 1997). Some of the created by-products are mostly used as additives to cattle fodder, and the unused remains are not managed, and as such represent a problem for the environment (collection, disposal, and processing). The oil that can be created from tomato seeds by applying SC-CO_2 is attracting interest because of the abundance of unsaturated fatty acids, especially linolenic acid (Roy et al., 1996), as well as lycopene, a carotenoid responsible for the red colour of the tomato, the highest concentrations of which are in the skin (Favati et al., 1997; Baysal et. al., 2000; Ollanketo et al., 2001; Rozzi et al., 2002).

2.8. Oilseed cakes

During the production of oil from oilseeds, it is of great importance to obtain higher extraction yields and oil quality. After oilseed processing, the remaining cake is commonly used in animal nutrition (Sorin-Stefan et al., 2013). The by-product (cake) from the process of oil pressing has great potential applications in food production due to the high content of residual oil, protein, fibre, minerals, and other substances. By using SC-CO_2, it is possible to get a totally defatted cake (Aladić, 2015) with the potential application for other purposes (i.e. in the enrichment of extruded products, bakery industry, etc.).

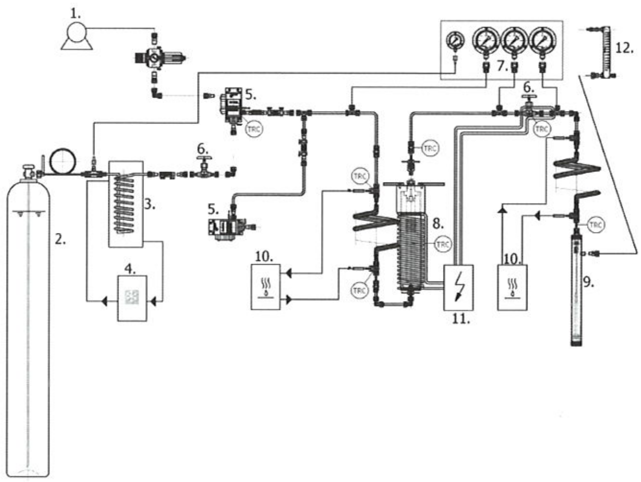
3. SUPERCRITICAL FLUID EXTRACTION

With the discovery of supercritical fluids, toxic and ecologically unacceptable organic solvents are being gradually replaced. Supercritical fluid extraction (SFE) is an innovative technology and it represents an excellent alternative to standard extraction procedures using organic solvents. Additional reasons for that are the numerous advantages of supercritical fluids, like better diffusion, lower viscosity, and lower surface tension, which enables them to better penetrate the material used for extracting the targeted substance. In addition, this procedure enables high selectivity and solvent capacity control of the desired component in the supercritical fluid, by changing the pressure and temperature, and simply removing the solvent from the extracts. Furthermore, SC-CO_2 is considered completely safe for industrial application and food processing. This energy efficient process is considered as "clean technology", because it does not create secondary products that are harmful for the environment and as such is very significant for the food industry (Brunner, 2005; Wang and Weller, 2006; Reverchon and De Marco, 2006; Abbas et al., 2008; Sahena et al., 2009; Temelli, 2009; Cvjetko Bubalo et al., 2015).

In the last few decades, this powerful separation process has drawn an increasing interest in commercial application, particularly due to its technical and environmental advantages compared to the current classical extraction methods using organic solvents. Extracts obtained using SC-CO_2 as the extraction solvent are solvent-free / without any trace of toxic extraction solvents, and are thereby highly valued (Jokić et al., 2014a). CO_2 is an adequate solvent for the extraction of lipophilic non-polar constituents. As such, it has a low affinity to polar compounds. This is one of the main drawbacks of extraction with SC-CO_2. The low polarity of SC-CO_2 can be overcome by employing polar modifiers (co-solvents, mainly ethanol and methanol) to change the polarity of the supercritical fluid and to increase its solvating power towards the analyte of interest. As such, according to Herrero et al. (2010), the addition of a low percentage of ethanol or methanol (1-10%) to CO_2 expands its extraction rates to include more polar analytes.

3.1. HM-SFE System

More than a few hundred commercial plants in the world today are using SFE in different fields (Jokić et al., 2015). The handmade supercritical fluid extraction (HM-SFE) system enables extraction in an inexpensive way. The obtained extraction yields and composition are very similar to those obtained by a commercial SFE system (Jokić et al., 2015; Jokić et al., 2014b). Just like a commercial SFE system, the HM-SFE system is composed of various components that need to be in tune in order to achieve the optimal extraction process. Parameters such as temperature, pressure, and fluid flow rate need to be monitored and controlled in order to enable an efficient and economical extraction process. Furthermore, the system must contain safety features with the possibility of an emergency system shutdown in the event of system failures. When designing the device, it is crucial to pay attention to several factors that make the device safe for work, because supercritical fluids and liquefied gases present a big hazard risk (explosion). Based on our experience in designing the HM-SFE system, we give special importance to the safety of the process. A certificate for working pressure was obtained for each used component, while calculations were carried out for the tube. All parts of the HM-SFE system should be made of stainless steel (AISI 304, AISI 316 Ti) to prevent any corrosion of the material. It is desirable to use certified filters with stainless steel



(1. Compressor; 2. CO_2 tank; 3. Stainless steel coil; 4. Cooling bath; 5. Air driven fluid pump; 6. High pressure needle valves (B-HV); 7. Manometers; 8. Extraction vessel; 9. Separator vessel; 10. Water bath; 11. Centralised system glass fibre heater; 12. Flow meter) (Jokić et al., 2015)

Figure 1. The HM-SFE system at the Faculty of Food Technology Osijek

mesh wire, coated with replaceable fine hard filter paper, in order to prevent plugging and possible hazard events.

When designing the extractor, aside from safety, it is very important to make an extractor which is very easy to maintain regarding loading and unloading, cleaning, connecting, and handling. From our experience in designing the HM extractor, we decided to make an extractor with a screw closure system (Jokić et al., 2015). Greater efficiency and continuous work are accomplished by using systems with the multiple parallel-connected extractors. The best closing system for a separator is also a screw type because of the fast extract collecting during the process at any time after depressurising. Due to the fact that it is necessary to heat and cool the fluid, heat exchangers are required in the HM-SFE system. Heat exchangers have the role of conditioning the fluid stream to required temperatures for pumping, extraction, separation, and holding (process storage) (Del Valle et al., 2014). From our experience, the heat exchanger should be placed just before the entrance of the extractor. This enables preheated CO2 at extraction temperature to enter the extractor. On the HM-SFE system, an air driven non lubricated liquid pump is used to obtain the desirable carbon dioxide pressure. To prevent cavitation and irregularity in the operation of the pump, the head of the pump is additionally cooled through a cooling bath with a stainless steel coil.

The schematic diagram of the designed HM-SFE system (Faculty of Food Technology Osijek, Croatia) is given in **Figure 1** and explained in detail in the previous chapter of our book (Jokić et al., 2015).

4. CONCLUSION

In recent years, SC-CO_2 extraction was demonstrated as a good alternative for processing food industry by-products. There is a great diversity of potential functional products which can be obtained from food by-products. Therefore, the use of food industry by-products has become extremely interesting because they represent cheap and nutritionally valuable raw material, and also have an impact on waste reduction. Modern companies are increasingly investing in research related to the reduction in the amount of waste and in research that would maximise the utilisation of raw materials. By applying SFE technology, it is possible to achieve the "zero waste approach" by ensuring that large amounts of organic waste are not created, which represents a huge environmental and financial problem in almost all branches of the food industry today.

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Đurđica Ačkar[1], Jelena Panak Balentić[1], Antun Jozinović[1], Jurislav Babić[1**], Borislav Miličević[1], Krunoslav Aladić[2], Stela Jokić[1], Drago Šubarić[1**]*

SUPERCRITICAL CO_2 EXTRUSION – NOVEL TECHNOLOGY IN FOOD INDUSTRY

[1]Faculty of Food Technology, Josip Juraj Strossmayer University of Osijek, F. Kuhača 20, 31000 Osijek, Croatia, (*dackar@ptfos.hr)
[2]Croatian Veterinary Institute, Branch – Veterinary Institute Vinkovci, J. Kozarca 24, 32100 Vinkovci, Croatia
**Croatian Academy of Engineering, Zagreb, Croatia

Abstract

Nowadays extrusion is an important process in food industry due to the HTST (High Temperature Short Time) effect that is applied in many processes. It is used to produce a wide variety of products, from modified starch and flours, over pasta and meat products, to snack and confectionary products.

Although it is considered to be an HTST process, it still may result in the formation of acrylamide and other compounds potentially harmful for human health. To overcome this issue, extrusion assisted with supercritical CO_2 has been introduced, opening the possibility to obtain products of retained quality at lower temperatures.

The aim of this paper is to give a short overview of research on extrusion with supercritical CO_2 and address some issues related to it.

1. INTRODUCTION

Compared to other processes, extrusion has been introduced to food industry relatively late. Nevertheless, nowadays it is a very significant and common process applied in the production of snack products, pasta, chewing gums, bonbons, breakfast cereals, modified flours, texturised proteins, etc.

When applied in food industry, extrusion often includes both high pressures and high temperatures. Since high temperatures cause unwanted reactions which result in the formation of compounds that cause adverse health effects (such as acrylamide), contemporary scientific and professional research is oriented towards the production of products without, or with reduced amounts of harmful compounds, but of the same quality. In this manner, CO_2 in supercritical state has been applied in the extrusion process.

2. EXTRUSION WITH SUPERCRITICAL CO_2

The pioneers in this research area, Rizvi and Mulvaney, patented the supercritical CO_2 extrusion process in 1992, claiming it controlled expansion and acted as a plasticiser and a viscosity modifier. In subsequent research (Rizvi, Mulvaney and Sokhey, 1995) they listed its advantages: The formation of composite microstructures at the microcellular level that result in specific morphological and mechanical properties, the potential for application of thermolabile additives (colours, aroma compounds, vitamins…), and viscosity reduction. Other authors (Bilgi Boyaci et al., 2012; Sharif et al., 2014) also recognised these positive aspects. In addition, lower temperature applications require lower energy costs (Bilgi Boyaci et al., 2012).

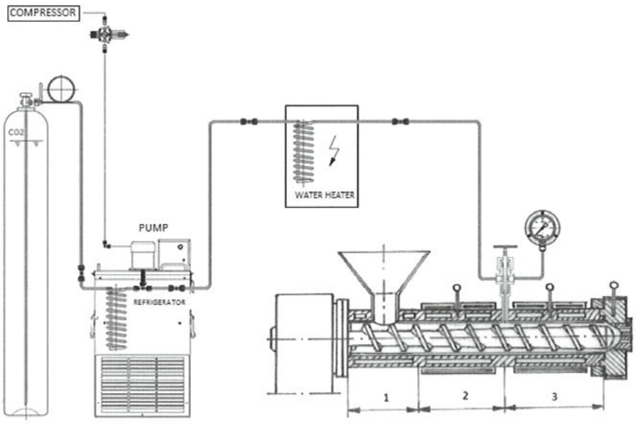

Figure 1. Scheme of an extruder with the addition of supercritical CO_2 at the Faculty of Food Technology Osijek

All these advantages resulted in very extensive research on supercritical CO_2 extrusion in the field of biopolymers and plastics production. Chauvet et al. (2017) made an extensive overview of current research on biopolymer production using this process.

To ensure that a process with supercritical CO_2 is successful, incorporation of CO_2 into the gelatinised mass has to be addressed during the design phase of the extrusion process, because CO_2 "backflow" can occur due to the high pressure within the barrel. It can be prevented by the placement of a ring on the zone transition, or "tandem" extrusion can be applied, where the compression of the material and the incorporation of supercritical CO_2 are performed in the first extruder, and additional homogenisation and partial cooling are performed in the second extruder (Chauvet et al., 2017).

Generally, it is considered that supercritical CO_2 results in a uniform porous structure, but slower expansion than in the classical process has to be taken care of. In addition, the large pressure drop at the exit from the extruder can result in structure collapse and loss of expansion due to gas leakage from the product (Cho and Rizvi, 2008). This problem can be resolved by partial cooling in the twin screw extruders before the introduction of the material to the die or by reducing the die diameter (Alavi and Rizvi, 2005).

Although it is still not extensively researched in food technology, some examples of successful applications of supercritical CO_2 extrusion in food production have been reported in scientific literature. Paraman et el. (2012) reported that supercritical CO_2 application in the production of puffed rice results in the preservation of all essential amino acids and over 50% of vitamins A and C. The results of this research have been confirmed on rice crisps with the addition of soy protein and micronutrients (Sharif et al., 2014), and satisfactory results have been reported for rice crisps with spirulina, minerals, and vitamins A and C (Bashir et al., 2016).

Masatcioglu et al. (2014) applied supercritical CO_2 extrusion in the production of corn extrudates with the addition of hull-less barley flour. Compared to the classical process, extrudates produced with supercritical CO_2 had smoother texture and more uniform expansion, and all other properties were comparable to the classical product. In wheat extrudates, supercritical CO_2 extrusion enhanced the expansion and lightness of the products, without significant influence on the chemical composition (Singkhornart et al., 2014).

Modified flours produced using this process are readily soluble in water (Jeong and Toledo, 2004), and supercritical CO_2 extrusion can replace yeast fermentation and ensure continuity of dough expansion in bread production (Hicsasmaz et al., 2003).

Manoi and Rizvi (2008, 2009) applied supercritical CO_2 extrusion in the production of texturised whey protein. They reported that gel formation in cold water enables the application of this product in modification and texture formation of food, especially when texturisation was performed at low pH, when smooth structure and creamy texture were obtained.

Ruttarattanamongkol et al. (2015) reported that texturised whey proteins produced by supercritical CO_2 extrusion can be used in the stabilisation of oil emulsions with different oil content and consistency – from salad dressings to spreads, and Cho and Rizvi (2008) successfully applied it in corn chips. They reported that supercritical CO_2 enabled extrusion at temperatures below protein denaturation, which in turn prevented hard texture formation.

Paraman et al. (2013) produced fructo-oligosaccharide-enriched whey protein crisps by using supercritical CO_2 extrusion. The sensorial acceptable product contained 8% of prebiotic fibre and 80% whey, and had even porous structure and crispness, with 92% preserved essential amino acids.

Supercritical CO_2 extrusion was a good tool for pore size and water-solubility control in starch modification, and phosphorylated starch produced in this process can be used in the production of biodegradable packaging due to low mass, high water resistance, and characteristic non-porous foamy structure (Patel et al., 2009; Manoi and Rizvi, 2010).

At the Faculty of Food Technology Osijek, as part of the project "Application of Food Industry By-products in the Development of Functional and Environmentally Friendly Extruded Food Products and Additives (FUNEXFOOD)" (HRZZ-1321) financed by the Croatian Research Foundation, a laboratory single-screw extruder has been modified so it can operate both in classical conditions and with supercritical CO_2 (**Figure 1**). Supercritical CO_2 can be added in two places: in front of the die and at the middle section of the compression zone. The addition of supercritical CO_2 before the die only influences the surface – the products are smoother and lighter, while placement in the compression zone also influences the porosity of extrudates.

At present, the research on the influence of supercritical CO_2 extrusion on the retention of phenolics and fibre, the reduction of acrylamide formation and starch damage, and the increase of resistant starch content in snack products and modified flours with the addition of different food industry by-products (defatted cakes of industrial hemp, pumpkin, chestnut, camelina) is in progress.

3. CONCLUSION

Although it is showing great potential for application in the food industry, extrusion with supercritical CO_2 requires additional scientific and professional research before its practical application. Issues regarding expansion still have to be resolved, since expanded products are very easily deflated and the cost-benefit relation should be addressed with regard to the design process with cost-effective functionality.

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HATZ News

REPORT ON THE ACTIVITIES OF THE CROATIAN ACADEMY OF ENGINEERING IN 2016

According to the Statute of the Croatian Academy of Engineering, the Academy, established in 1993, is a scientific organisation of distinguished and prominent scientists in the field of technological and biotechnological sciences with the objective of promoting the sciences, encouraging co-operation of and bringing together the scientists of various technological, biotechnological and other professions in order to support efficient scientific and economic development of Croatia without gaining any profit.

In 2009, the Academy was recognised as a scientific organisation by the Ministry of Science, Education, and Sports of the Republic of Croatia.

Since 2000, the Academy has been a member of the CAETS (International Council of Academies of Engineering and Technological Sciences), and, since 2005, a member of the Euro-CASE (European Council of Academies of Applied Sciences, Technologies and Engineering).

In 2016, the representatives of the Academy, Prof. Vladimir Andročec, PhD, President of the Academy, and Prof. Zdravko Terze, PhD, Vice-President of the Academy, have attended two meetings of the Euro-CASE Board as the Board Members. The first meeting took place in May 2016 in Paris, France, and the second, in November 2016 in Lyngby, Denmark.

The Academy representatives have also participated in the meetings concerning the SAPEA Project (Science Advice for Policy by European Academies), on which our Academy is engaged through its Euro-CASE membership. The Project is very interesting for the academic community as well as for the economy and political life of the EU Member Countries. It has been conceived by the European Commission and serves to represent inter-academy co-operation at the EU level. It encompasses five associations of the European academies (The Five European Academy Networks): AE - Academia Europaea, ALLEA – All European Academies, EASAC – European Academies Science Advisory Council, Euro-CASE – European Council of Academies of Applied Sciences, Technologies and Engineering, and FEAM – Federation of European Academies of Medicine. Aside from the European academies this Project also brings together the academies from Israel, Armenia, Georgia, and Turkey. The goal of this inter-academy co-operation at the EU level is to provide the European Commission with an independent and interdisciplinary science-based policy by virtue of combining resources of 100 individual academies across Europe, with hundreds of members each and covering all scientific disciplines: social sciences, humanities, natural sciences, engineering sciences and biomedical sciences. The SAPEA Project was officially launched on December 13, 2016, in Bruxelles and is funded within the Horizon 2020 Programme.

Through its memberships both in CAETS and Euro-CASE our Academy is also engaged at the "EU–US Frontiers of Engineering" Platform. The Platform is organised by the US National Academy of Engineering, the largest and the most influential CAETS Member Academy. Following the extensive preparations during 2016, the 2017 Symposium "EU-US Frontiers of Engineering" will take place on November 16-18, 2017 at the University of California in Davis, CA, US. The Symposium is organised jointly by the CAETS (NAE) and Euro-CASE Member Academies (TAF – Technology Academy Finland) and will be dedicated to the development, challenges, and co-operation between the EU and the US in the field of engineering.

The representatives of the Academy Prof. Vladimir Andročec, PhD and Prof. Zdravko Terze, PhD, have participated at the 2016 Euro-CASE Annual Conference „Big Data – Smarter Products, Better Societies", which was held on November 13-15, 2016 in Lyngby, Denmark.

The Croatian Academy of Engineering, through its Euro-CASE membership, is also engaged in the Engineering Education Platform, dedicated to a stronger positioning and visibility of the engineering education within a wider corpus of the European Commission's policies, which are dedicated to higher education in Europe and directly connected to research and development as the preconditions for the creation of new jobs, economic growth and prosperity of the EU.

In 2016 the Academy has been patron and co-organiser of 14 distinguished international and domestic scientific and professional meetings as well as organiser of 15 meetings, and it participated in 50 meetings of public interest. Among the most important meetings is the 2nd Croatian Engineers' Day, organised by the Croatian Engineering Association (HIS), Croatian Academy of Engineering and Faculty of Electrical Engineering and Computing in Zagreb (FER), was held on March 2, 2016, at FER in Zagreb.

On October 17, 2016, the Academy has organised the Ceremony of Unveiling of the Bust of Prof. Vatroslav Lopašić, PhD, distinguished Croatian physicist and late Honorary Member of the Academy. The bust is the work of art by a distinguished Croatian academic sculptor Boris Leiner.

The publishing activity of the Academy in 2016 has been abundant. The Academy has published one new issue of its Bulletin in Croatian, the "Tehničke znanosti" Vol. 20(1) 2016 as well as two new issues of its Bulletin in English, the "Engineering Power" Vol. 11(1) 2016 and Vol. 11(2) 2016, the later issue of which has been dedicated to the first Croatian Nobel Prize Winner Prof. Leopold Ružička, PhD, following the 40th Anniversary of his death.

The Academy has also published the "Annual 2015 of the Croatian Academy of Engineering", encompassing the reprints of the best scientific papers by its members, which had been previously published in the most prominent international scientific journals. The reprints were published in the "Annual" with permissions of both the original publishers and authors of the papers.

In co-operation with the Public Open University Zagreb, the Academy was the patron and co-publisher of the monograph by Prof. Gojko Nikolić, PhD, "The Life and Inventions of Faust Vrančić" – The Second Extended and Updated Edition.

In 2016 the Academy had held numerous meetings of its bodies: 20 Governing Board meetings, 3 Presidency meetings, 1 Assembly meeting and many meetings of Departments, Standing Committees and Centres.

In 2016 the Academy had issued and conducted the Internal Call for the Election of New Members of the Academy and New Emeriti of the Academy. Eight new Emeriti of the Academy have been elected as well as thirteen new Members of the Academy. Seven scientific and higher-education institutions, organisations, and companies have been elected to Supporting Members of the Academy.

In the same year, the Academy had issued and conducted the Call for Applications for the Awards of the Academy for the year 2015. The 2015 Award for Lifetime Achievement and the Medal of the Academy has been granted to Prof. Emer. Zlatko Kniewald, PhD, retired Professor Emeritus and Full Professor with Tenure of the University of Zagreb at the Faculty of Food Technology and Biotechnology, Emeritus of the Academy in the Department of Bioprocess Engineering, Secretary of the Department and Member of the Presidency of the Academy, who had also served as the President of the Academy for two terms: 2003-2005 and 2005-2009. The 2015 Rikard Podhorsky Annual Awards have been granted to Prof. Bojan Jerbić, PhD, Full Professor of the University of Zagreb at the Faculty of Mechanical Engineering and Naval Architecture, Member of the Academy in the Department of Systems and Cybernetics, Secretary of the Department and Member of the Presidency of the Academy, and to Prof. Damir Ježek, PhD, Full Professor of the University of Zagreb at the Faculty of Food Technology and Biotechnology, Dean of the Faculty, Member of the Academy in the Department of Bioprocess Engineering. The 2015 Vera Johanides Awards for Young Scientists have been granted to: Tomislav Capuder, PhD, University of Zagreb, Faculty of Electrical Engineering and Computing, Tomislav Pukšec, PhD, University of Zagreb, Faculty of Mechanical Engineering and Naval Architecture, Ana Belščak-Cvitanović, PhD, University of Zagreb, Faculty of Food Technology and Biotechnology, and Ivan Marović, PhD, University of Rijeka, Faculty of Civil Engineering.

Our Academy has a particularly successful co-operation with the Croatian Academy of Sciences and Arts, the leading Croatian scientific and cultural institution. The two Academies have signed the Agreement on Co-operation in 2014. A very successful co-operation has also been realised with both the Croatian Academy of Sciences and Arts and the Miroslav Krleža Institute of Lexicography in the joint project of Croatian Technical and Technological Encyclopaedia. The tri-partite Protocol on Co-operation on the Project of Croatian Technical and Technological Encyclopaedia has been signed in 2014. The intensive work of all parties on this project during the previous years, and especially in 2016, will result in the publishing of the first volume of the Encyclopaedia in 2017. On our Academy's part, members of the Academy nominated by the Science Council of the Academy as authors and editors, have been particularly engaged on this very important and valuable project.

In the previous year, the Academy has also been engaged in an excellent co-operation with its sister academies in the Republic of Croatia: Croatian Academy of Medical Sciences, Croatian Academy of Legal Sciences and Croatian Academy of Forestry Sciences. The quadrilateral Agreement on Co-operation between our four Academies has been signed in 2012 and ever since the Academies co-operate and engage on a number of successful joint activities and projects, meetings, lectures and fora. We would like to especially highlight the joint Scientific Symposium of the four Academies, "Modern Technologies: The Ethics of Utilisation and the Legal Regulation", initiated by the Croatian Academy of Medical Sciences, which was held on March 17, 2017, at the Croatian Physicians' Assembly in Zagreb, Croatia, and vas very successful. The four Academies co-operate through the Council and Co-ordination of the Academies, which serve as their organisational and co-ordinative bodies.

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Melanija Strika, Business Secretary of the Croatian Academy of Engineering

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Editor-in-Chief: Vladimir Andročec
Editor: Zdravko Terze
Guest Editor: Drago Šubarić
News Editor: Melanija Strika
Phone: + 385 1 4922 559
Tehnički urednik/Technical Editor: Vladimir Pavlić, GRAPA, Zagreb, Croatia
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