

## STUDYING THE PROCESSING OF FOOD DYE FROM BEET JUICE

*Tamila Sheiko, Serhii Tkachenko, Mikhailo Mushtruk, Volodymyr Vasylyv,  
Olena Deviatko, Roman Mukoid, Marina Bilko, Mykola Bondar*

### ABSTRACT

The manuscript describes a new method of red beet processing and the technology of manufacturing food colorant from the juice concentrate, which is natural, safe and useful analogue to existing expensive offers on the market of similar goods that have chemical origin not useful for regular consumption. Nowadays in order to give to food products a colour, close to natural coloring of fruits and vegetables, expensive synthetic dyes are used, which might have cancer-inducing effect when being accumulated by human organism. Therefore improving the technology for producing food grade dye from red beet juice is remarkably important task. Currently, there is a problem for vegetable processors – pectin substances complicate the process, like the illumination of juice and negatively affect its storage capacity. The article below reveals and substantiates the necessity of using a natural carbon-containing adsorbent shungite for the purification of beet juice from pectin substances. On the basis of the study, the authors suggest a more cost-effective way of producing a food dye from juice concentrate, which allows avoiding usage of expensive enzyme processing additives.

**Keywords:** food grade dye; red beet juice; natural adsorbent; shungite; studying the processing

### INTRODUCTION

Red beet juice is very useful food product, because it contains significant amount of sugars, mineral substances and vitamins. It is also valuable because it's used for producing food grade dye (Sigurdson, Tang and Giusti, 2017; Lehto et al., 2017).

Nowadays in order to give to food products a colour, close to natural coloring of fruits and vegetables, expensive synthetic dyes are used, which might have cancer-inducing effect when being accumulated by human organism. Therefore improving the technology for producing food grade dye from red beet juice is remarkably important task (Scotter, 2015; Gras et al., 2017).

Currently for the purpose of obtaining dye from red beet juice, a part of pectin substances, which make the process of juice concentration more difficult, is removed with the help of expensive enzymatic agents (Sheiko, 2015; Sheiko, Tkachenko and Petrenko, 2017; Mehmedli et al., 2015). It is well-known that to clean juice, the following enzyme preparations are used: pectophytoside with a complex of pectolytic enzymes (pectinase) for the cleavage of pectin substances; amylorizine and glucoadamorine with an amylase advantage; protohetoidin with an enzyme complex of proteolytic (protease) and pectolytic activity for juice processing mainly for the purpose of juice illumination and removal of turbidity (Murashev, Zhemchuzhnikova and Verzhuk, 2015; Loretz, Lopaeva and Neverova, 2016; Stich, 2016).

The main disadvantages of the enzymatic methods of juice cleaning are permanent repetition and long process flow (Schweiggert, 2018; Kolyanovska et al. 2019).

The authors have suggested using natural carbon-bearing adsorbent shungite to purify red beet juice from pectin substances (Sheiko, 2015; Sheiko, Tkachenko and Petrenko, 2017; Vetrov, Akishin and Akimov, 2016).

Shungite is a mineral consisting of amorphous carbon and fractured graphite. Its chemical composition is not constant: shungite contains 60 – 70% of carbon and 30 – 40% of other elements.

Shungite is the only known mineral to have fullerenes (recently discovered new globular form of carbon existence). Fullerenes' structure is peculiar because carbon atoms in molecules are situated at the tops of regular pentagons and hexagons, which cover sphere's surface and present themselves as closed polygons composed of paired quantity of coordinated carbon atoms (Rumyantsev and Sizov 2018; Kornen, 2016; Kozelová et al., 2011).

Fullerenes differ from particles with metallic properties due to the location of electron cloud and ability to change the form of carbon structure.

Sizing of electro-magnetic waves is determined by vibration of electrons which are divided into  $\pi - \sigma$  and  $\pi - \pi$  states. During adsorption on electrically neutral surface the localization of fullerenes,  $\pi - \pi$  states takes place, and a particle loses its metallic properties, and because of that connected electron pair appears in the activated form. Thus mineral shows bipolar properties. Having analyzed the

features of the structure of shungite, one can be concluded that the mechanism of adsorption of pectin substances from red beet juice is explained not only by adsorption in the mineral pores, but also by ion exchange adsorption at the places of occurrence of reactively able centers of fullerenes together with the formation of hydrogen bonds with the pectin molecule. E.g. fullerene which can have properties of metal or semiconductor particles, resulting in compounds with different types of chemical bonding (exhibits bipolar properties) (Sierra-Rosales, Toledo-Neira and Squella, 2017; Vagiri and Jensen 2017; Siva, 2007).

Shungites' important characteristic is the presence of fullerene carbon nanotubes with the diameter of their cylindrical pores constituting 1-6 nanometers and the width – up to several micrometers. The cylindrical surface of tubes is formed by active carbon circles and also has empty pores.

The basis of shungite structure is a globule composed of graphitic networks, formed into packages. Each package has 6 graphitic flat networks with the quantity of carbon atoms attaining to 300 – 600 and one curved network, having 400 carbon atoms.

Colour has been measured in the food industry by subjective visual inspection, including the use of visual colour standards. Colour instruments objectively measures colour, are tools that assist the eye. With proper usage, it can provide repeatable, meaningful colour data that agree with visual measurement (Loughrey, 2000; Nagyová, Berčík and Horská, 2014). The colour of any food product can be represented in terms of 'L' (lightness), 'a' ('+a', redness and '-a', greenness), and 'b' ('+b', yellowness and '-b', blueness) values or combination of these three depending upon the nature of the pigment present in the food material. It is reported that the objective measurement of colour using 'L', 'a' and 'b' system is a good indicator of the total colour change of heat treated peach puree (Ávila and Silva, 1999), broccoli juice (Weemas et al., 1999), mango puree, red chilli puree and paste, papaya puree and plum puree (Ahmed, Shivhare and Kaur, 2002).

Beet root powder or extracted pigments are used industrially for improving the red colour of tomato pastes, sauces, soups, desserts, jams, jellies, ice creams, sweets, and breakfast cereals (Roy et al. 2004; Koul et al., 2002; Sukhenko et. al., 2017). The red beetroot (*Beta vulgaris* L.) is a good source of red and yellow pigments known as betalains. Betalains consist of betacyanins (red) and betaxanthins (yellow). The major betacyanin in beetroot is betanin and accounts for 75 – 95 % of the red pigment. The degradation of betanin was reported to follow first order reaction kinetics. It is reported that in the presence of excess of oxygen, betanin degradation follows pseudo first order reaction (Attoe and Von Elbe 1982; Patkai and Barta 1996; Sukhenko et. al., 2019).

Betanins are reported to have some antioxidant activity and are found to be effective in inhibiting lipid peroxidation. Thus it is suggested that red beet products consumed regularly in the diet may provide protection against certain oxidative stress-related disorders in humans and also improve digestion and blood quality (Kanner, Harel and Granit, 2001; Butera et al., 2002; Herbach, Stintzing and Carle, 2004; Azeredo, 2009). Therefore,

the retention of colour pigments as indicated by its colour it is very important with regard to its health benefits as well as visual appeal. Knowledge of kinetic parameters of colour degradation during thermal processing is very important in optimization of the process parameter. A literature survey indicates that similar work on beet root has not been reported.

Consequently, the use of shungite makes it possible to establish binding to the pectin substances of beet juice, which will significantly improve the process of purifying the juice during the production of the dye.

### Scientific hypothesis

A precedent has been published studies devoted to obtaining dye from beet juice with an adjustable amount of pectin-containing substances.

The purpose of this study was to establish the possibility of replacing expensive enzyme preparations with natural carbon-containing adsorbent shungite for cleaning beet juice.

### MATERIAL AND METHODOLOGY

The method of conducting the experiment was as follows: juice was obtained from table beet and heated to a temperature of 50 – 60 °C (creating conditions as close as possible to the production), mixed with shungite powder TU U 24.6-32177584-003:2011 "Materials are shredded and ground with shungite", industrial fraction 0.001 – 0.002 m.

Shungite to be used for research was prior washed out with cold water and then thermoactivated at 100 °C during 90 minutes. Cooled an adsorbent in concentrations of % mass: 2.44; 3.23; 4.76; 9.09 was put into fresh red beet juice at temperature of 20, 40, 50, 60 °C, mixed during 10, 20, 30, 60 min., filtrated. The content of pectin substances in filtrate was measured in accordance with calcium pectate method (Sachek, Tile and Sevostyanov, 2017) under formula:

$$PIP = \frac{(g - g_0) \cdot 100 \cdot 0.9235}{V \cdot d}$$

whereas PIP – content of pectin substances in juice, mg.g<sup>-1</sup>; g – weight of weighting cup with precipitate before exsiccation, g;

g<sub>0</sub> – weight of empty weighting cup, g;

V – juice volume, cm<sup>3</sup>;

d – juice density, g.s<sup>-3</sup>;

0.9235 – coefficient for conversion of calcium pectate into pectic acid.

The obtained results were compared by effect of purification (Table 1):

$$E = \frac{100 \cdot (K_1 - K_2)}{K_1}$$

where: K1 and K2 – quantity of target component in juice which was not processed by adsorbent and juice which was processed by adsorbent.

The next phase of research was determining the content of coloring substances of red beet juice, processed by shungite at temperature of 50 °C. Preparation of shungite was performed in the same way as for adsorption of pectin

substances. The content of coloring substances was determined according to standard method of Leventaal-Neubauer (Danilova and Popov, 2004). Estimation of mass content of coloring substances was performed under formula:

$$X = \frac{(M_1 - M_2) \cdot K \cdot 0.004157 \cdot O_1 \cdot 100}{H \cdot O}$$

where: X – mass content of coloring substances, %;  
 $M_1$  – quantity of potassium permanganate ((chemically pure for analyzes) supplier company PrimaRia, Kiev, Ukraine) solution (0.1 mol.L<sup>-1</sup>) used for the first titration, m<sup>3</sup>;  
 $M_2$  – quantity of potassium permanganate solution (0.1 mol.L<sup>-1</sup>) used for the second titration, m<sup>3</sup>;  
 $O_1$  – volume of the primary extraction, m<sup>3</sup>;  
 H – quantity of juice used for experiment, m<sup>3</sup>;  
 $O_2$  – volume of the secondary extraction, used for titration, m<sup>3</sup>;  
 0.004157 – coefficient which takes into account correlation between potassium permanganate and juice coloring substances.

### Statistic analysis

The statistical evaluation of the results was carried out by standard methods using statistical software Statgraphics Centurion XVII (StatPoint, USA) – multifactor analysis of variance (MANOVA), LSD test.

## RESULTS AND DISCUSSION

The obtained data shows that adsorption of pectin substances from red beet juice by shungite takes place already at temperature of 20 °C. If temperature rises the process is somewhat accelerated. The rise of temperature of processing juice over 60 °C is unreasonable because coloring components are destroyed and that causes the changes in juice colour.

Comparing the obtained results and their practical efficiency the authors determined optimal parameters, concluding that the optimal parameters for processing juice by shungite is adsorbent concentration of 4.76% mass, temperature of processing 50, 60 °C, duration 30 min. Under such conditions 36.8 – 42.1% of pectin substances are removed.

The mechanism of adsorbing pectin substances from red beet juice by shungite is explained not only due to the fact that impurities infiltrate the mineral's pores, but also due to ion-exchange adsorption in places where reactive centers of fullerenes are formed and hydrogen bonds with pectin molecule are created.

Shungite's selectivity is explained not only because of the existence of micro-, mezzo- and macropores, but also because nanotubes participate in adsorption processes and there are pores in between them, created when packages are formed, and also because of free non-compensated charges which appear on adsorbent's surface.

Emphasizing on the receipt of natural dyes should be allocated colorant Beet Red – E162. The main colorant is betaine – an alkaloid-like compound obtained from beet juice. The molecular formula of the additive

E162:C24H27N2O13 (Sheiko, 2015; Sheiko, Tkachenko and Petrenko, 2017).

The research was performed and its results are showed in Figure 1.

Analysis of data presented in Figure 1 gives grounds to state that content of coloring substances in red beet juice, after it was processed by shungite, practically does not change. This can be explained by the fact that the basis of coloring substances in red beet juice is anthocyanin (Sheiko, 2015; Sheiko, Tkachenko and Petrenko, 2017; Sukhenko et. al., 2020). By their structure they are chains of glycosides, composed of heterocyclic compounds. By their chemical nature they are surface active substances. On interphase border anthocyanin molecules are situated in such a way that hydrophilic group remains in liquid state. Hydrophobic effect takes place and thus coloring substances are not adsorbed by shungite.

The authors improved apparatuses and technological scheme for producing food grade dye from red beet juice by installing two adsorbing devices with shungite which work in regime of sorption-desorption.

Installing adsorbers will remove excess substances and do not have a temperature effect on the juice. The colour of the dye E162 may vary depending on the bright red to the blue-violet (at an elevated pH) and is destroyed by the action of a high temperature (above 65 °C). In the course of studies it was found that the optimum temperature of juice processing in the adsorber is 50 °C. So you can stabilize the colour of juice for further technological operations (Sheiko, 2015; Sheiko, Tkachenko and Petrenko, 2017).

The results of the studies allowed to develop a mathematical-statistical model of the shungite regeneration process, using second-order root-level planning.

$$y = -490.5 + 6.2t_n + 0.1\tau + 0.02 t_n \cdot \tau - 0.02t_{n2} - 0.1\tau_2$$

where:

$t_n$  – temperature of superheated water vapor, °C,

$\tau$  – duration of regeneration, min.

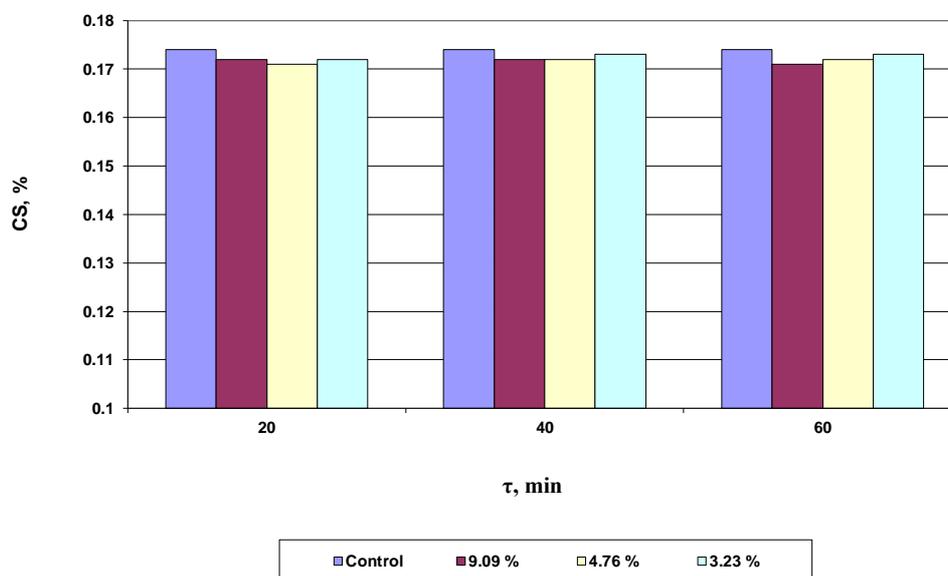
On the basis of the obtained mathematical model, optimization of the process was carried out regeneration of shungite by superheated water vapor Figure 2.

The rational parameters regeneration of shungite should be carried out with superheated water vapor with a temperature of 170 °C, pressure 0.3 MPa. Regenerated shungite is used in the technological process up to 5 times. The duration of regeneration is 15 – 20 minutes; the mass flow rate of steam is about 2.305 10<sup>-3</sup> kg·s<sup>-1</sup>.

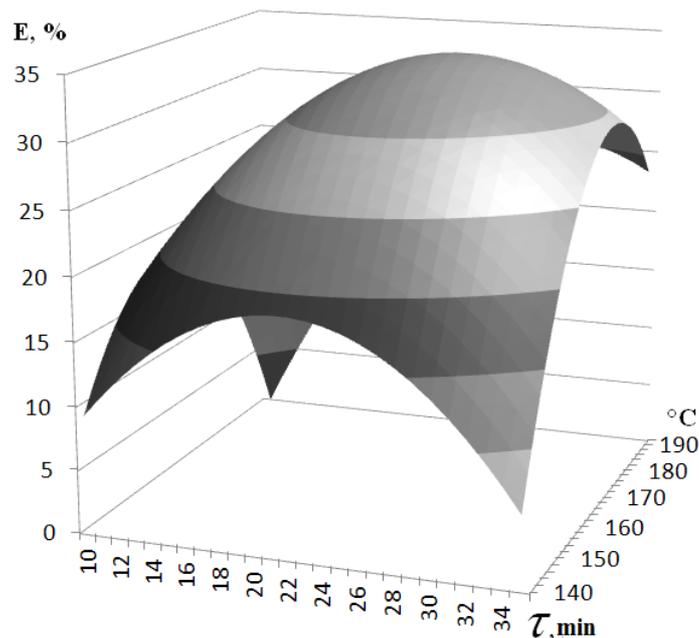
As the result of regeneration, adsorption capacity of the adsorbent may recover completely or partially, depending on adsorption capacity of the desorbed components, chosen method of desorption and operating parameters of the process. In some cases it is justified to have incomplete recovery of the adsorbent activity, as this reduces operating costs. Subsequently spent shungite can be recycled by burning (Sheiko, 2015; Sheiko, Tkachenko and Petrenko, 2017; Dhar et al., 2015).

**Table 1** Effect of purification (E,%) from pectin substances in red beet juice by shungite at different adsorbent concentrations, temperature of mixtures, duration of interactions between adsorbent and juice, initial content of pectin substances is 1.9 mg.g<sup>-1</sup>.

Effect of purification	Adsorbent concentration in juice, % mass															
	2.44				3.23				4.76				9.09			
	Temperature, °C															
	20	40	50	60	20	40	50	60	20	40	50	60	20	40	50	60
	Duration of juice processing, 10 min															
Content of pectin substances, mg.g <sup>-1</sup>																
Processed juice	1.8	1.8	1.7	1.7	1.8	1.7	1.7	1.6	1.7	1.6	1.5	1.5	1.6	1.6	1.5	1.4
E, %	5.3	5.3	10.5	10.5	5.3	10.5	10.5	15.8	10.5	15.8	21.0	21.0	15.8	15.8	21.0	26.3
Duration of juice processing, 20 min																
Processed juice	1.7	1.7	1.6	1.6	1.5	1.5	1.4	1.3	1.5	1.4	1.3	1.2	1.4	1.3	1.2	1.2
E, %	10.5	10.5	15.8	15.8	21.1	21.1	26.3	31.6	21.1	26.3	31.6	36.8	26.3	31.6	36.8	36.8
Duration of juice processing, 30 min																
Processed juice	1.6	1.6	1.5	1.5	1.4	1.4	1.3	1.2	1.5	1.3	1.2	1.1	1.4	1.3	1.1	1.1
E, %	15.8	15.8	21.1	21.1	26.3	26.3	31.6	36.8	21.1	31.6	36.8	42.1	26.3	31.6	42.1	42.1
Duration of juice processing, 60 min																
Processed juice	1.6	1.6	1.5	1.5	1.4	1.3	1.2	1.2	1.5	1.3	1.2	1.1	1.3	1.2	1.1	1.1
E, %	21.1	21.1	26.3	26.3	26.3	31.6	36.8	42.1	21.1	31.6	36.8	42.1	31.6	36.8	42.1	42.1



**Figure 1** Quantity of coloring substances (CS) in juice processed with shungite depending on the duration of its processing at temperature of 50 °C.



**Figure 2** Optimal parameters of the shungite regeneration process.

After part of pectin substances are removed in adsorbing device, red beet juice is placed in vacuum evaporator where it is concentrated around 6 hours. Concentration of juice by its evaporation takes place with discharge of 0.055 – 0.060 MPa and temperature of 55 – 60 °C, concentrated red beet juice, already as food grade dye, is packed in the sealed container, made of dark glass. The level of pH in the obtained food grade dye does not exceed 4.5. The used adsorbent is recyclable or can be recovered.

## CONCLUSION

It was established that shungite effectively adsorbs pectin substances from red beet juice and does not adsorb coloring substances. The technology of producing food grade dye was improved by additional purification of red beet juice from pectin substances by shungite. The obtained optimal technological parameters for purifying juice with shungite are as follows: adsorbent's concentration constituting 4.76% mass., temperature is 50 – 60 °C, duration of processing is 30 min. Apparatuses and technological scheme of producing food grade dye is supplemented with two adsorbing devices with shungite, which work in regime of sorption-desorption. The used adsorbent is recyclable or can be recovered.

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**Contact address:**

Tamila Sheiko, Institute of Food Resources of the National Academy of Agrarian Sciences of Ukraine, st. E. Sverstiuk 4a, Kyiv, Ukraine, 02000, Tel.: 044-517-06-92, E-mail: [sheiko\\_tamila@ukr.net](mailto:sheiko_tamila@ukr.net)  
 ORCID: <https://orcid.org/0000-0002-0559-1335>

Serhii Tkachenko, Institute of Food Resources of the National Academy of Agrarian Sciences of Ukraine, st. E. Sverstiuk 4a, Kyiv, Ukraine, 02002, Tel.: 044-517-06-92, E-mail: [sergi-tkachenko@ukr.net](mailto:sergi-tkachenko@ukr.net)

ORCID: <https://orcid.org/0000-0001-6400-6426>

\*Mikhailo Mushtruk, National University of Life and Environmental Sciences of Ukraine, Faculty of Food Technology and Quality Control of Agricultural Products, Department of Processes and Equipment for Processing of Agricultural Production, Heroev Oborony Str., 12 B, Kyiv, 03040, Ukraine, Tel.: +38(098)941-26-06,

E-mail: [mixej.1984@ukr.net](mailto:mixej.1984@ukr.net)

ORCID: <https://orcid.org/0000-0002-3646-1226>

Volodymyr Vasylyv, National University of Life and Environmental Sciences of Ukraine, Faculty of Food Technology and Quality Control of Agricultural Products, Department of Processes and Equipment for Processing of Agricultural Production, Heroev Oborony Str., 12 B, Kyiv, 03040, Ukraine, Tel.: +38(097)465-49-75,

E-mail: [vasiliv-vp@ukr.net](mailto:vasiliv-vp@ukr.net)

ORCID: <https://orcid.org/0000-0002-8325-3331>

Olena Deviatko, National University of Life and Environmental Sciences of Ukraine, Mechanical and Technological Faculty, Department of Technical Service and Engineering Management th. M.P. Momotenka, Heroev Oborony Str., 12 B, Kyiv, 03040, Ukraine, Tel.: +38(066)205-43-01,

E-mail: [helene06@ukr.net](mailto:helene06@ukr.net)

ORCID: <https://orcid.org/0000-0002-4743-6931>

Roman Mukoid, National university of food technology, Educational and Scientific Institute of Food Technology, Department of biotechnology of fermentation and winemaking products, Volodymyrska Str. 68, 01601 Kyiv, Ukraine, Tel.: +38(097)394-49-89,

E-mail: [mukoid\\_roman@ukr.net](mailto:mukoid_roman@ukr.net)

ORCID: <https://orcid.org/0000-0002-3454-1484>

Marina Bilko, National university of food technology, Educational and Scientific Institute of Food Technology, Department of biotechnology of fermentation and winemaking products, Volodymyrska Str. 68, 01601 Kyiv, Ukraine, Tel.: +38(067)702-02-68,

E-mail: [aromat@ukr.net](mailto:aromat@ukr.net)

ORCID: <https://orcid.org/0000-0002-1122-4937>

Mykola Bondar, National university of food technology, Educational and Scientific Institute of Food Technology, Department of biotechnology of fermentation and winemaking products, Volodymyrska Str. 68, 01601 Kyiv, Ukraine, Tel.: +38(093)709-42-84,

E-mail: [bondnik@i.ua](mailto:bondnik@i.ua)

ORCID: <https://orcid.org/0000-0002-5775-006X>

Corresponding author: \*