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# A study on the effect of coolant parameters to surface roughness in external cylindrical grinding of 90CrSi steel using Taguchi method

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## ABSTRACT

Grinding is one of the last operation performed which machine the work piece to obtain the high quality surface roughness and accurate dimension. It is an complex machining process and there are many processing parameters affect on the quality and productivity. Characteristic of grinding process is the removed material from the work surface in the phase of fine chips by many tiny abrasive particles of grinding wheel. Therefore, it is difficult to evacuating chips and the most of energy converted to heat. To solve these problem require to using the suitable lubrication-coolant and the right lubrication condition. The optimum lubrication-coolant condition will ensure the machine quality and improve productivity. This study to investigate the optimization parameters of lubrication condition in cylindrical grinding 90CrSi harden steel using PV cutting oil.

**Keywords**—Coolant-lubrication; external cylindrical grinding; taguchi method

## I. INTRODUCTION

Grinding process is one of the most important machining method to achieve final surface qualities and precessions. Grinding process directly effect to quality of the products. Therefore, there are numerous researchers interested in improving the performance and quality of grinding process [1]. For example, studies to optimizing grinding process parameters on material removal [2], on surface qualities [3-5], or minimizing grinding time and maximizing the volume of material removed rate [6, 7]. On the other side, the studies about influence of dressing parameters to wheel wear [8], to surface roughness [9] and topography of grinding wheels [10] have conducted. And, there have some studies to optimize the cost of grinding processes which present the cost function of grinding processes [11, 12].

Moreover, studying the effects of lubrication have focused. The lubrication have great effect on the surface qualities of products, the cutting force and temperature damaged to the ground surface. The researchers focus on finding the new lubrication liquid, lubrication method or determining the optimum parameters for lubrication in grinding process. Such as study about using the cryogenic LN<sub>2</sub> (liquid nitrogen) as an alternative coolant when grinding AISI 16 stainless steel by Sol-Gel (SG) alumina grinding wheel [13], or investigate the performance of two specifically designed fluids to reduces the quantities of oil and gas to very low (3ml/min and 0.2 kg/min) [14]. The study of D. Babic indicated that using high speed water mist jets seem to be cheap and effective way of cleaning the reducing specific energies [15].

The new lubrication method which applying the MQL (Minimum Quantity Lubricant) have studied. It have presented in study a system which use of two nozzles: first, oil is supplied as MQL, and then a flow of CO<sub>2</sub> at 238°K is in charge of fixing the frozen oil on the surface of the abrasive grits, protect them from wear and improving sliding conditions where show the better surface quality of the component [16]. But the other study indicated that the effect of compressed cold air (CCA) and vegetable oil on the surface integrity and residual stresses of ground tool steel component show a good cooling efficiency only at small grinding depths [17]. More over, the study of Hadad to compare between MQL grinding performance to dry and fluid grinding process in terms of temperature

distribution on the surface and subsurface of the work-piece, show the MQL despite the good lubrication, it can not meet the grinding cooling requirements compared with fluid grinding [18].

Until now, there haven't got any lubrication method which totally replace the fluid method. Regarding to determine the optimum parameters for lubrication they have got some study in internal grinding [19] or external grinding process [20].

This paper, presents a study to determine the optimum parameters of lubrication when grinding 90CrSi harden steel using PV Cutting oil by applying Taguchi method.

## II. EXPERIMENTATION

In this research, the experiments were carried out by using PV Cutting oil in external cylindrical grinding process where its parameter was described in table 1. This cutting oil is widely used in cutting processes with advantages as: safety, easy dissolve, stable emulsions, low foaming, good in cooling and corrosion protection.

Table 1. Parameters of PV Cutting Oil

Parameter	Method	PV Cutting Oil
Kinematic viscosity at 40°C	ASTM D445	46.5
PH in 5%, min	-	9.0
Density at 15°C	ASTM D1298	0.875
Flash point COC, °C, min	ASTM D92	220

The grinding system and measuring equipment are shown in the table 2.

Table 2. Grinding system and measuring equipment

Grinding machine	CONDO-Hi-45 HTS	Japan
Grinding wheel	Cn80MV1 400x40x203 35m/s	Viet Nam
Dressing tool	3908-0088C type 2	Russian
Surface roughness tester	Mitutoyo 178-923-2A, SJ-201	Japan

Work-piece material is 90CrSi harden steel to 62÷65 HRC with chemical composition in table 3. Work-piece dimension is  $\phi 25 \times 180$  mm.

Table 3. Chemical composition of 90CrSi steel

Steel grade: 9CrSi						
C	Si	Mn	Cr	P	S	Co
0.85-0.95	12-1.6	0.3-0.6	0.95-1.25	≤0.03	≤0.03	≤1

### Experiment design

To evaluate the effect of the coolant on the grinding process and determine the optimum coolant parameters, the grinding and dressing condition had been kept constant during the grinding process. The coolant parameter were chosen as input parameters to investigated are flow rate, coolant pressure and concentration of lubricating fluid with three different level as shown in table 4.

Table 4. Coolant parameters and control levels

Symbols	Controlled parameters	Level 1	Level 2	Level 3
F	Flow rate (l/min)	5	10	15
P	Coolant Pressure (at)	1	1.5	2
C	Concentration (%)	2	3	4

The experiments were designed by Taguchi method for optimizing the input parameters using  $L_9$  orthogonal array. The result of experiment design by Minitab software are described in table 5.

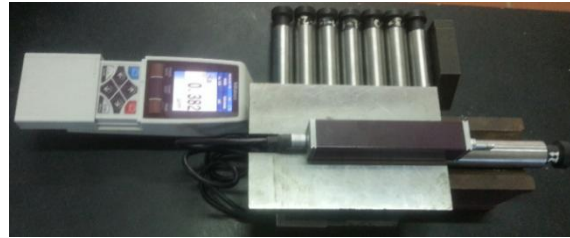


Figure 1. Surface Roughness Test

### III. RESULT AND DISCUSSIONS

In the grinding test, the surface roughness of work-piece are measured by Mitutoyo Surface Roughness tester (SJ-201, Japan) (Figure 1). The experimental plans and the output results and their response are shown in Table 5.

Table 5. Experimental plans and output response

Test	Flow rate (l/min)	Coolant Pressure (at)	Concentration (%)	Surface Roughness (Ra)			SNRA	MEAN ( $\mu\text{m}$ )
				Trail 1	Trial 2	Trial 3		
1	5	1	2	0.491	0.487	0.455	6.4126	0.477667
2	5	1.5	3	0.382	0.388	0.365	8.4396	0.378333
3	5	2	4	0.422	0.415	0.425	7.5208	0.420667
4	10	1	3	0.286	0.275	0.280	11.0454	0.280333
5	10	1.5	4	0.315	0.332	0.325	9.7871	0.324000
6	10	2	2	0.427	0.428	0.431	7.3575	0.428667
7	15	1	4	0.342	0.338	0.325	9.4971	0.335000
8	15	1.5	2	0.410	0.412	0.405	7.7653	0.409000
9	15	2	3	0.315	0.302	0.312	10.1807	0.309667

For achieve the minimum of surface roughness, the testing results were analyzed by the criterion for evaluation "Smaller is better  $SN=10\log[1/n(\sum y_i^2)]$ " has been used. By using ANOVA in Minitab 18, the analysis of Variance (ANOVA) for SN ratios is shown in Figure 2. The specific percent contribution of the input parameters are show clearly in Figure 3. From the figure, the concentration of lubricant fluid is the most effect on surface roughness (about 61%), the flow rate contribute about 36% and the coolant pressure has least contribution with 2%.

Analysis of Variance for Means						
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Flow rate (F)	2	0.012170	0.012170	0.006085	55.71	0.018
Coolant Pressure (P)	2	0.000774	0.000774	0.000387	3.54	0.220
Concentration (C)	2	0.020927	0.020927	0.010463	95.79	0.010
Residual Error	2	0.000218	0.000218	0.000109		
Total	8	0.034089				

Figure 2. Analysis of Variance for means of SN ratios for Surface Roughness (Ra)

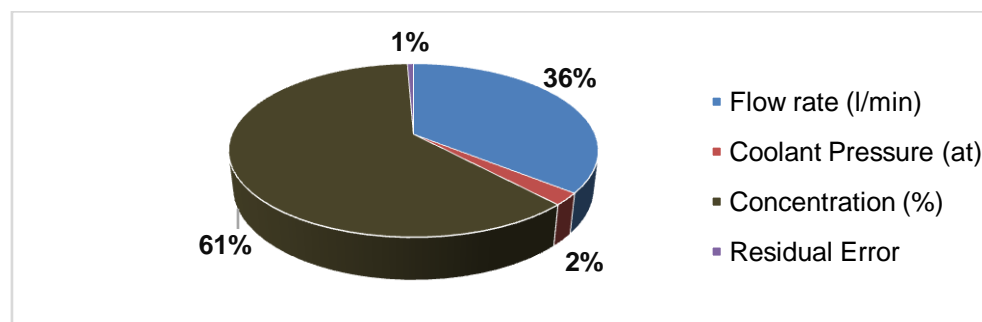


Figure 3. Percentage contribute of parameters to surface roughness (Ra)

Base on the analysis result in Figure 2, the P-value also describes that flowrate (F) and concentration (C) of lubricant fluid have significant effect on the surface roughness and the coolant pressure (P) has not significant effect.

Figure 4 present the respond table for signal to noise ratios for the surface roughness. It indicates the influences of input parameters on the surface roughness by the ranks of its effect in sequence are the concentration, the flow rate and the coolant pressure.

### Response Table for Signal to Noise Ratios

Smaller is better

	Flow	Coolant	
	rate (F)	Pressure	Concentration
Level		(P)	(C)
1	7.458	8.985	7.178
2	9.397	8.664	9.889
3	9.148	8.353	8.935
Delta	1.939	0.632	2.710
Rank	2	3	1

Figure 4. Response table for signal to noise ratios (Smaller is better)

The Main effects plot for SN ratios for surface roughness is presented in figure 5. As in the mention above, the smaller value of surface roughness is better in grinding process. In this figure, the large S/N ratio means a smaller surface roughness. Therefore, as describe by plot of each parameter in the figure, it was found that the optimum values of the input parameters for surface roughness are flowrate 10 l/min (level 2), concentration 3% (level 2) and coolant pressure 1 atm (level 1).

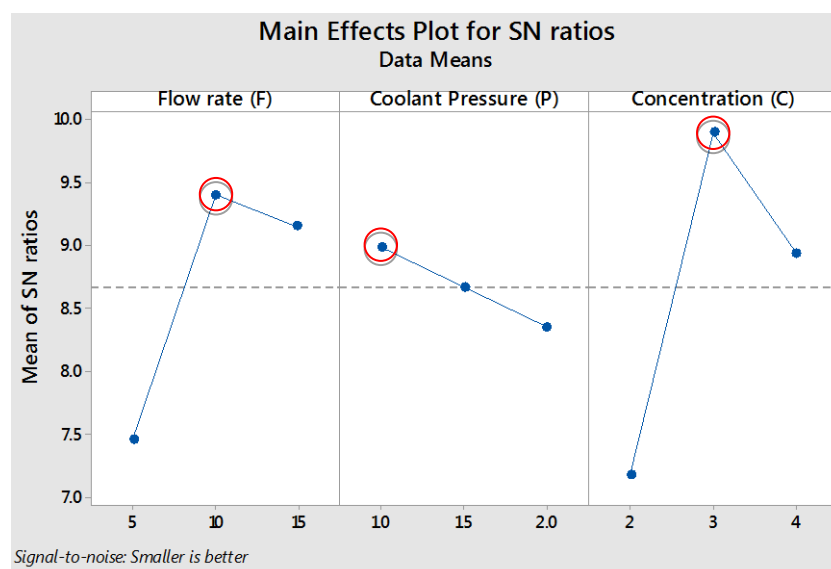


Figure 5. Main effect plot for SN ratios

An analysis using Minitab 18 to predict the surface roughness respective optimum parameters of lubricant has been proceed. As the result which is shown in figure 6, the predict optimum value of the surface roughness is 0.284  $\mu\text{m}$ .

### Predicted values

#### Prediction

S/N Ratio	Mean	StDev	Ln(StDev)
10.9356	0.284037	0.0087846	-4.84671

Figure 6. Predicted values of surface roughness at optimum lubricant parameters

To verify the optimum input parameters, a test using the optimum values was conducted. The surface roughness of testing was 0.295  $\mu\text{m}$ , compare to predict surface roughness, the error was only 3.87%. Its mean that the experiment work is satisfied.

#### IV. CONCLUSIONS

In this study, the optimum lubricant parameters in external cylindrical grinding are determined. The experiments were designed by using Taguchi technique, the result are analysed by Minitab 18 indicated the effect of coolant parameters on the surface roughness. The optimum values of lubricant parameters to get the minimum surface roughness in sequence of its effect are the lubricant concentration of 3%, the flow rate at 10 l/min and the coolant pressure of 1 at. In addition, the optimum values of input parameters with predict value of surface roughness was successful verified by an experiment and the error was 3.87%.

#### ACKNOWLEDGEMENTS

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#### REFERENCES

- [1] Rowe W.B., *Principles of modern grinding technology*. 2013: William Andrew.
- [2] Pandiyan V., et al., *Predictive modelling and analysis of process parameters on material removal characteristics in abrasive belt grinding process*. 2017. **7**(4): p. 363.
- [3] Banh T.L. *A Study on Optimization of Surface Roughness in Surface Grinding 9CrSi Tool Steel by Using Taguchi Method*. in *Advances in Engineering Research and Application: Proceedings of the International Conference, ICERA 2018*. 2018. Springer.
- [4] Li G., L.S. Wang, and L.J.J.o.m.p.t. Yang, *Multi-parameter optimization and control of the cylindrical grinding process*. 2002. **129**(1-3): p. 232-236.
- [5] Tung L.A., et al. *A Study on Optimization of Surface Roughness in Surface Grinding 9CrSi Tool Steel by Using Taguchi Method*. in *International Conference on Engineering Research and Applications*. 2018. Springer.
- [6] Gupta R., K. Shishodia, and G.J.J.o.M.P.T. Sekhon, *Optimization of grinding process parameters using enumeration method*. 2001. **112**(1): p. 63-67.
- [7] Mekala K., et al., *Optimization of cylindrical Grinding parameters of austenitic stainless steel rods (AISI 316) by taguchi method*. 2014. **3**(2): p. 208.
- [8] Vidal G., et al., *An Analysis of Electroplated cBN Grinding Wheel Wear and Conditioning during Creep Feed Grinding of Aeronautical Alloys*. 2018. **8**(5): p. 350.
- [9] Tu H.X., et al. *Influence of dressing parameters on surface roughness of workpiece for grinding hardened 9XC tool steel*. in *IOP Conference Series: Materials Science and Engineering*. 2019. IOP Publishing.
- [10] Palmer J., et al., *An experimental study of the effects of dressing parameters on the topography of grinding wheels during roller dressing*. 2018. **31**: p. 348-355.
- [11] Le Xuan Hung, V.T.L., et al., *A STUDY ON COST OPTIMIZATION OF INTERNAL CYLINDRICAL GRINDING*.
- [12] Pi V.N., et al., *Cost optimization of internal grinding*. 2016. **6**: p. 291-296.
- [13] Manimaran, G., M.P.J.A.o.c. Kumar, and m. engineering, *Effect of cryogenic cooling and sol-gel alumina wheel on grinding performance of AISI 316 stainless steel*. 2013. **13**(3): p. 304-312.
- [14] Wang Y., et al., *Experimental evaluation of the lubrication properties of the wheel/workpiece interface in minimum quantity lubrication (MQL) grinding using different types of vegetable oils*. 2016. **127**: p. 487-499.
- [15] Babic D., et al., *Mist jet cooling of grinding processes*. 2005. **45**(10): p. 1171-1177.
- [16] Sanchez, J., et al., *Machining evaluation of a hybrid MQL-CO<sub>2</sub> grinding technology*. 2010. **18**(18): p. 1840-1849.
- [17] Xiao K. and L.J.J.o.m.p.t. Zhang, *The effect of compressed cold air and vegetable oil on the subsurface residual stress of ground tool steel*. 2006. **178**(1-3): p. 9-13.
- [18] Hadad, M., B.J.I.J.o.M.T. Sadeghi, and Manufacture, *Thermal analysis of minimum quantity lubrication-MQL grinding process*. 2012. **63**: p. 1-15.
- [19] Hung L.X., et al. *A Study on Coolant Parameters in Internal Grinding of 9CrSi Steel*. in *Materials Science Forum*. 2019. Trans Tech Publ.
- [20] Tu H.X., V.N. Pi, and G. Jun. *A study on determination of optimum parameters for lubrication in external cylindrical grinding base on Taguchi method*. in *Key Engineering Materials*. 2019. Trans Tech Publ.



# GAS DIFFUSIONS IN FLEXIBLE PIPELINES.

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## ABSTRACT

*This presentation describes a model used to study gas diffusion through layers of flexible pipes by time. The temperature gradient pipe is considered as temperature dependent permeability rates. This model is coupled with a calculation that indicate changes in pressure and volume of vapors resulting in the annular space. Associated mathematical models and methods for solving the results obtained are presented in Math Soft with a user-friendly interface that helps in data entry and processing results. In this presentation will show the possibilities of this software*

**Keywords**—rehabilitation permeability, diffusion, pipelines, polimer, riser.

## I. INTRODUCTION

The offshore oil industry, hoses and raisereleare made with polymers with internal and external coatings, which provides fluid flow through the inner and outer insulation in relation to the marine environment. These polymers have a certain permeability to gas that can facilitate the reduction of the potential damage mechanisms of the life of the steel layers located between the pipe and the outer shell of polymer. The destruction mechanisms associated with water condensation, therefore, they must be removed, [1].

## II. OPERATING ENVIRONMENT FOR FLEXIBLE PIPE AND RISER

They are considered deepwater activity in terms of oil, waters deeper than 400 m; 1 500 m is considered ultra deep water (over 1 600 m after MMS Mineral Management Service, USA)

Oil industry operators are turning to large water depths, because there are significant resources that ensure high yields. Some oil wells in these areas can produce 8000 m<sup>3</sup> / day crude oil production justifying additional costs and risk.

Projects operating from premises situated in water depths of 2000 m in the Gulf of Mexico, Brazil and West Africa Offshore were unimaginable not long ago. A large number of wells have been drilled at depths of water; record of 10 400 ft (m 3174) was passed in February 2013 in the Indian Ocean. The most important aspects in production wells located in deep water depths are related to high water, but



also on the bottom, the hostile environment in which it operates: waves 30 meters high; Winds exceeding 80 knots (148.2 km / h); low air temperatures: -15 ° C; Sea water temperature: 0 ° C; marine currents 3 knots (5.5 km / h) etc.

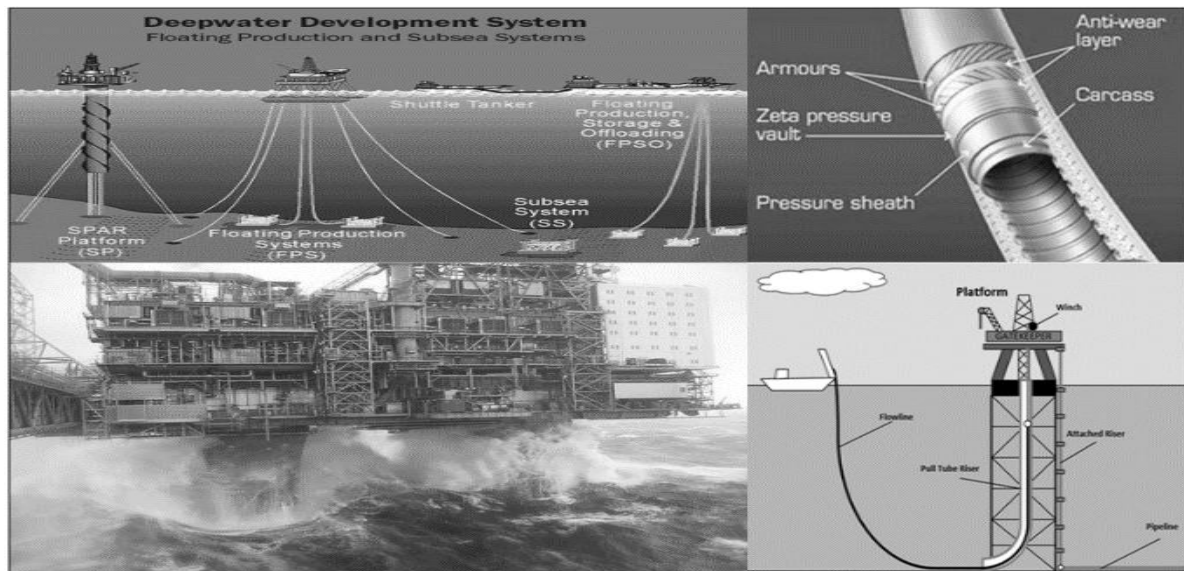


Fig.1 Flexible pipes operating and production risers [3]

Column production or production riser is the portion that lies between the host plant surface and the seabed near the home of an installation depth. Discharge sizes are from 3 to 12 in (76.2 mm to 304.8). in diameter. The length of the riser is dependent on water depth and configuration of the column, which may be vertical or a variation waveform. Derivatives may be flexible or rigid and contained in the operating area of a fixed or floating platform installation type

### III. STRUCTURE FLEXIBLE PIPE. GENERAL ITEMS

Unbounded flexible pipes over the last 30 years were a key component in the production of oil and gas offshore. They represent an alternative to rigid steel pipes where they have the advantage of a quick installation and the potential adaptation de route. These benefits often make unbounded flexible pipes, a more economical solution than rigid steel pipes.

Successful exploitation of the majority of floating production systems depends on good performance systems dynamic flexible riser or jumper. The limits are consistently higher pressures and higher temperatures for deep waters, leading to increasing demands on the performance of pipe components. Layers of steel materials are decisive for their behavior in acidic environments static and dynamic applications.

Unbounded flexible pipe structure requires that the steel is in direct contact with the fluid product. The medium is determined by permeation of small molecules (mainly H<sub>2</sub>O, CO<sub>2</sub>, H<sub>2</sub> and CH<sub>4</sub>) by lining the polymer. Predictions therefore operating environment is a key issue for the prediction, design and service life of flexible pipes.

Unbounded flexible pipes are made of concentric layers of polymer material and steel. In order to preserve the flexibility of the construction of the pipe layers are not bonded together. The following figure shows a typical cross section of a flexible layers depicting typical. Different types of flexible, [4], [5] unbounded pipe may omit some of the layers. It is presented in the most general description of each of the major layers, Figure 2.

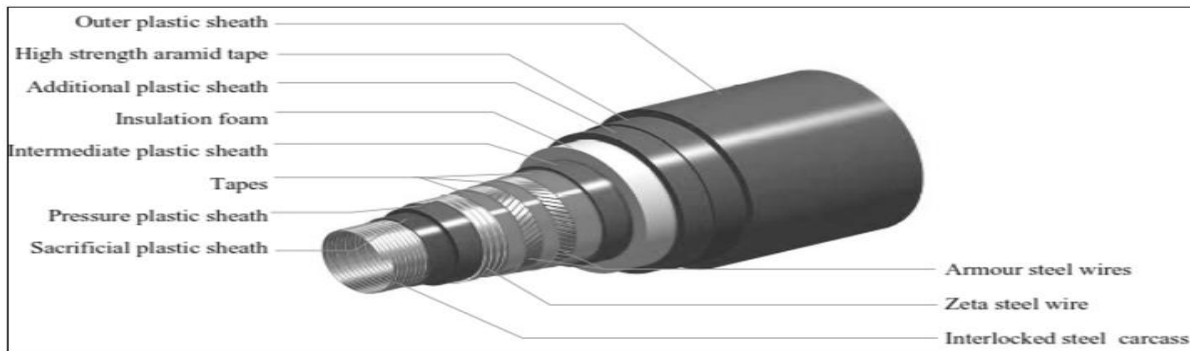


Figure 2 – General description of each of the major layers

**Steel Carcass** An interlocking layer made of a stainless steel strip. The casing prevents the collapse of the inner hull and provides mechanical protection against gear (piggings) and abrasive particles. Quality stainless steel structure is studied in detail, but is outside the scope of this paper research.

**Inner thermoplastic sheath** A polymer layer extruded ensure the integrity of the internal fluid. Common types of polymers are polyethylene (PE), cross-linked polyethylene (XLPE), polyamide 11 (PA11) and Polyvinilien fluoride) PVDF.

**Pressure armor layer** A number of layers composed of helically wound wire form C of steel and / or metal strips. The layers of reinforcement provide resistance to radial loads.

**Fittings traction** A number of structural layers consisting of helically wound flat steel wire. Layers are against and wrapped in pairs. The layers provide resistance to axial loads. External thermoplastic Sheth (A layer of extruded polymer) function is to protect steel components pipe from the outside (often seawater) and to provide mechanical protection.

**Nomenclature:** PA11 polyamide 11:PE polyethylene: PVDF poly(vinylidene fluoride) C concentration ( $\text{cm}^3/\text{cm}^3$ ): D diffusion coefficient ( $\text{cm}^2/\text{s}$ )

#### IV. FACTORS INFLUENCING THE INTEGRITY AND LIFE OF

Interior factors affecting the integrity riser are: fatigue fracture of steel casing, deformation housing, erosion figure 3a, influence of thermal variation, aging in action, chemical factor, temperature, diffusion in the annular space of  $\text{H}_2\text{S}$  /  $\text{CO}_2$  figure 3b, fatigue protective coatings, the formation of hydrates.

External factors affecting the integrity riser are: wear resulting from the interaction with the plant surface and submerged constructive elements, normal wear constructive materials, interacting with other lines submerged, deterioration protective outer covering Corrosion, hydrogen cracks action.

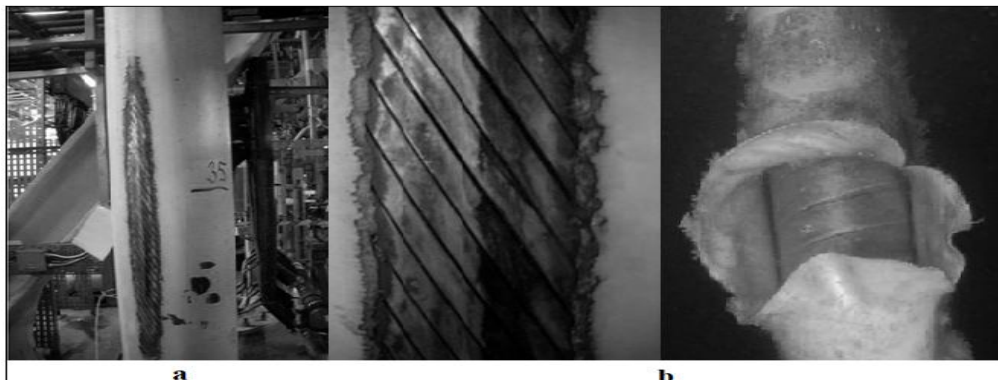


Figure 3 a) Damage to the outer covering contact with the production plant; b) Explosion outer covering due to accumulation of gas in the annular space, [3]

## V. GAS DIFFUSION – PERMEABILITY

It is well known that polymeric materials can be regarded as watertight only to a certain extent. With a difference of partial pressure of a fluid in a polymer membrane (liner) will result in higher penetration fluiduluide pressure to low pressure. The mechanisms of permeation polymers are outside the scope of this work are described extensively in the literature. Flexible pipe, coatings characterized by migration of gas permeability inside the pipe and the outer casing annulus. The offshore oil and gas production of molecules of interest are significant and methane ( $\text{CH}_4$ ), carbon dioxide ( $\text{CO}_2$ ), hydrogen sulfide ( $\text{H}_2\text{S}$ ) and water ( $\text{H}_2\text{O}$ ), [2].

Manufacturing companies are realizing research programs in order to determine the characteristic phenomenon of diffusion constants: permeability diffusion coefficient, solubility, the polymers used as material for deconstruction to flexible pipes. In addition to the main layers, are included more polymer layers to prevent wear between the structural layers. The strips of insulation with a low thermal conductivity may be used, for example, between the main reinforcement and the outer jacket, in order to obtain specific properties of the pipe insulation.

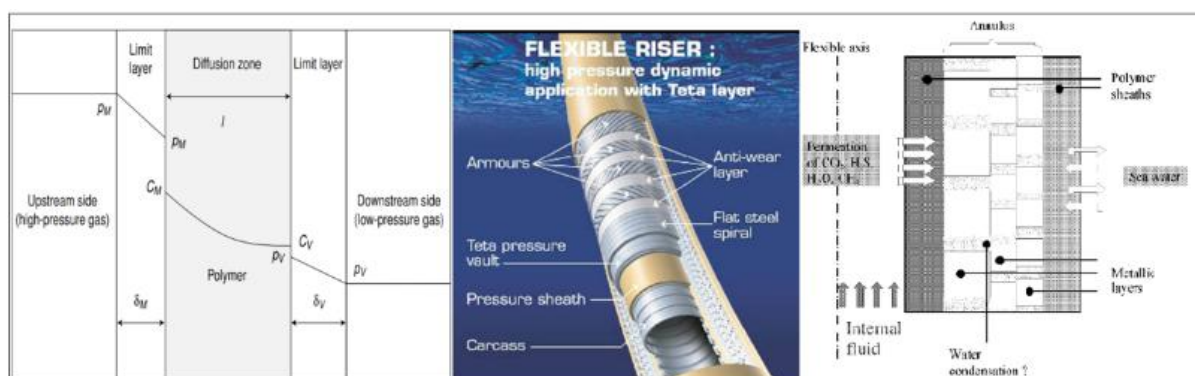


Figure 4 – The model of an element wire, [2]

Understanding the damage mechanisms in polymer coatings during gas decompression goes through the knowledge of gas transport phenomena in polymers, by studying the influence of gas absorption on material properties, and by modeling the behavior of the material during a decompression. The mathematical theory of diffusion (Crank, 1968) in an isotropic system is based on the hypothesis of the proportionality between the scattering flux of the molecules (which is the quantity of species crossing a membrane per unit time and surface) and the concentration gradient between the two faces Of the membrane. It is Fick's first law [2]:

$$J = -D \frac{\partial C}{\partial x} \quad (1)$$

where:

J is the scattering flow of the molecules

D - diffusion coefficient ( $\text{cm}^2 / \text{s}$ );  $D = D(C, T, p)$

C – concentration

The permeation, [4]. [5] Table 1, of methane and carbon dioxide plasticized polyvinylidene fluoride water (PVDF) and plasticized polyarnid 11 (PA11) was measured for a number of temperatures and pressures with testing devices.

Table 1

PERMEATION COEFFICIENTS FOR METHANE AND CARBON DIOXIDE THROUGH PLASTICIZED PVDF FOR TWO DIFFERENT GAS MIXTURES AT 120°C.

Gas mixture	Permeation coefficient, P ( $\text{cm}^2/\text{s}\cdot\text{bar}$ )		Pressure
	Methane	Carbon Dioxide	
97% $\text{CH}_4$ - 3% $\text{CO}_2$ - $\text{H}_2\text{O}$	$2.2 \cdot 10^{-7}$	$8.4 \cdot 10^{-7}$	25 bars
75% $\text{CH}_4$ - 25% $\text{CO}_2$ - $\text{H}_2\text{O}$	$2.1 \cdot 10^{-7}$	$8.3 \cdot 10^{-7}$	25 bars

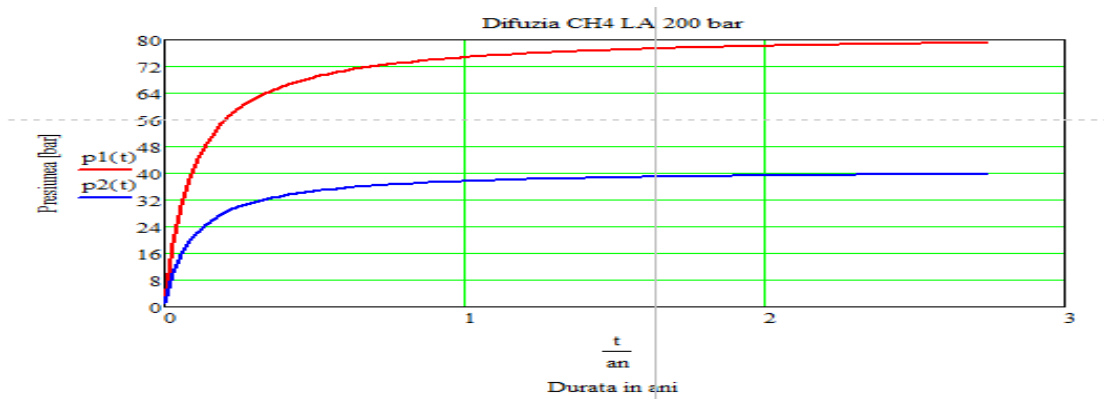


Figure 5 a

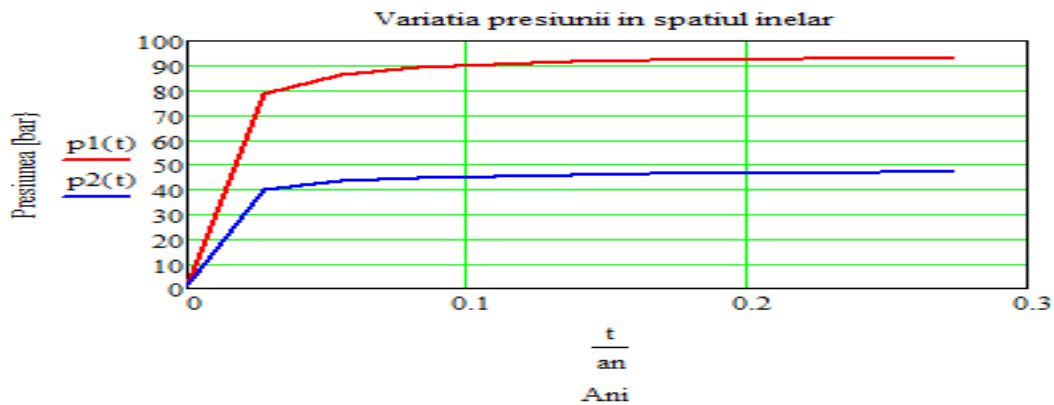


Figure 5 b

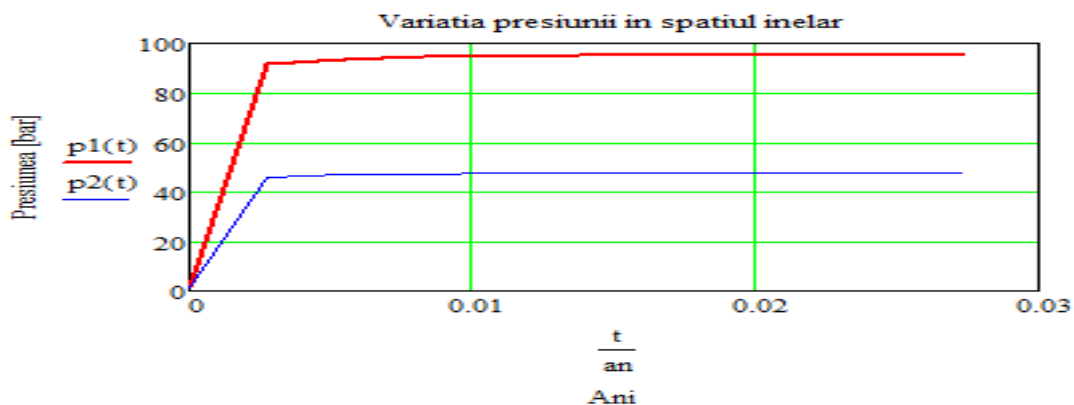


Figure 5 c

Figure 5 – Numerical simulation for a) difussion CH<sub>4</sub>;b) difussion CO<sub>2</sub>;c) difussion H<sub>2</sub>O

## CONCLUSIONS

To avoid gas diffusion effects in terms of maintaining balance in corrosive environment created by their penetration is required ventilation gas in the annulus. Establishing the ventilation status of layer fluids and observing the conditions considered to limit the upper end: continuous ventilation or - Intermittent respectively at atmospheric pressure ventilation or - sub-atmospheric (vacuum). Relationships and mathematical equations of the model proposed for the analysis and design of

ventilation for independent parameters of time (geometric) while those for addicts (hydraulic parameters). works constitute a separate objectives.

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### REFERENCES

- [1] Eugene Sas , Jean Marie Pere, Pierre Savy, Ahmed Omar *Flexible Pipe Design Optimization for West Africa Deepwater: Agbami Production Riser Case Study*, Offshore Technology, 5-8 May, Houston, Texas, 2008.
- [2] Benjelloun Z.-Dabaghi<sup>1</sup>and A. Benali, *Mathematical Modelling of the Permeation of Gases in Polymers*,<sup>1</sup> Institut français du pétrole, 1 et 4, avenue de Bois-Préau, 92852 Rueil-Malmaison Cedex – France, Oil & Gas Science and Technology – Rev. IFP, Vol. 56 (2001).
- [3] Lazăr Avram, Claudiu Tănasă,. N, N. Antonescu, Marius Stan, Valentin Tudorache, *Asupra proiectării coloanelor de raizare și criteriul de selecție a acestora*, cnr-cme.ro, FOREN 2016.
- [4] \*\*\*ANSI/API 17J "Specification for Unbounded Flexible Pipe" 3, Edition, 2008.
- [5] \*\*\*ANSI/API 17B "Recommended Practice for Unbounded Flexible Pipe" 3, Edition, 2009.

# Bond Performance of Fiber Reinforced Concrete Exposed to Elevated Temperatures

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## ABSTRACT

In structural concrete design, adequate bond between the reinforcing steel and concrete is essential, especially when the reinforced concrete structure is exposed to accidental fire causing elevated temperatures. In this paper, the effect of fiber types and elevated temperatures on compressive strength and the bond characteristics of fiber reinforced concrete (FRC) are discussed and presented. Two types of steel fibers, corrugated and end-hooked, and two types of polypropylene fibers, ordinary and structural, were used. The experimental work includes eight mixes. A plain concrete mix, without fiber, was used as a control mix. Three mixes contained corrugated steel fibers with a various volumetric ratios of 1% , 1.5% and 2% while three mixes contained end-hooked steel fibers with a various volumetric ratios of 1% , 1.5% and 2%. The last two mixes contain a mixture of 1 % end-hooked steel fibers with one type of polypropylene fiber. specimens were produced and exposed to elevated temperatures at 300° and 600 °C for 2 hours. Pull- out tests on cylinders (150\*300 mm) and axial compression tests on cubes (150\*150\*150 mm) were carried out to evaluate the bond performance between 16-mm reinforcing steel rebars and fibrous concrete. The obtained results showed slight reduction in residual compressive and steel–concrete bond after exposure to 300 °C temperatures for 2 hrs. On the other hand, a severe reduction in residual compressive and steel–concrete bond after exposure to 600 °C temperature for 2 hrs was observed. Using fibers minimized the damage in steel–concrete bond under elevated temperature. Hooked steel fibers achieved the highest bond resistance against elevated temperatures followed, in sequence, by those prepared with structural polypropylene fiber .

**Keywords—** *Elevated temperature, steel fiber, polypropylene fiber, , pull- out, bond strength*

## I. INTRODUCTION

The mechanical response of reinforced concrete (RC) structures (crack width, crack spacing, deflection, tension stiffening) is highly dependent on the efficiency of the bond stress transfer evenly distributed all along the rough contact surface between the reinforcing bar (rebar) and the surrounding concrete. According to the state of the art report [1], the description of the rebar-concrete bond is essentially based on an empirical approach. As a consequence, exposure to high temperatures may cause considerable variations in the physical and mechanical properties with irreversible loss of strength and stiffness [2,3]. Concrete is commonly considered to have good fire resistance but chemical and physical reactions occur at elevated temperatures [2,4].

A fresh concrete that is not correctly placed and does not reach adequate hydration, contains free water, when subjected to elevated temperature; this water evaporates at 100°C, causing fragmentation. In addition, this situation accelerates the evaporation of the bound water at 300° C in the hydrated elements. Thus, a rapid degradation of the concrete strength causes the temperature increase in reinforcement bars. On the other hand, calcium hydroxide, which is an important cement



component, shrinks by 33% by losing water and transforms into quicklime at 530° C. During the fire, the water that is squeezed into the structure causes the quicklime changes, resulting in a volume expansion of 44%, and this sudden volume conversion causes cracking of the surrounding concrete [5]. It was also reported that the loss in bond strength could reach as high as 60% when RC is subjected to temperature exceeds excess of 500°C [6]. It was also concluded that, the specimens exposed to 800°C suffer a loss of the ultimate bond strength of 73.6% while the specimens exposed to 600°C suffer a loss of the ultimate bond strength of 67.8% compared with the specimens exposed to ambient temperature [7].

Pull-out test is frequently used to determine the bond between steel reinforcing bars and the surrounding concrete [8,9]. Previous experience showed that exposure of concrete to temperatures in excess of 400o C would have detrimental impact on its strength and integrity [10]. The loss in strength and/or spalling of concrete at high temperature was attributed to three major factors, namely vapor pressure of capillary and gel water, decomposition of cement hydration products, and possible collapse of filling aggregate [11]. It was reported that the loss in bond strength could reach as high as 60% when RC is subjected to temperatures in excess of 500° C [12,13].

Concretes with steel fibers (SF), polypropylene fibers (PPF) and polyvinyl alcohol fibers (PVA) showed good behaviors in fire in the controlling of the spalling [14,15]. In case of fire the PPF and PVA fibers melts around 170 and 230oC, respectively, and will create a network of micro-channels in the concrete which served as a way for the release of water vapor to the outside. Accordingly this will avoid the brittle type of failure in which explosive and the concrete becomes separated from the reinforcing bars.

The concrete specimens without additions suffer heavy loss of the mechanical properties by action of fire to reach a temperature of 650°C, leading to a loss of 73% of this capacity, but a considerable improvement of this loss of capacity by adding short (PP) fibers of 40% took place [16]. From the 200°C, (PP) fibers begin to deteriorate with mass loss. The temperature starting the destruction of the fibers are moved to a temperature of 350°C and the total destruction, 95%, to the temperature of 500°C [16].

Compressive concrete strength has the most important role in RC structural members subjected to elevated temperature. According to the previous test results, it can be observed that a decrease of the concrete compressive strength with the elevated temperatures. Since the cement and aggregate forming the concrete, contain silica and limestone, it is expected that strength loss depends on various parameters. Particularly the quartz in silica-based coarse / fine aggregates, is subjected to polymorphic change at 570 °C temperature. This transformation causes volume increase and damage in concrete. Besides, in dolomitic aggregates, carbonate transforms into CaO or MgO at 800–900 °C. As temperature increases, limestone or dolomite expands; decomposition of CO<sub>2</sub> and formation of CaO or MgO initiate shrinkage. Those volume changes also cause damage in concrete. According to the previous research results, 45% of the concrete compressive strength at 600 °C is preserved, but concrete residual strength is only 18% at 800°C. Other researchers reported that most considerable reduction of compressive strength took place between 400 °C and 800 °C in all cases within different temperature range. The residual compressive strength values at the maximum temperature of 800 °C were between 20 % to 30 % of the strength at 20 °C for concrete with (PP) fibers. [5,17]


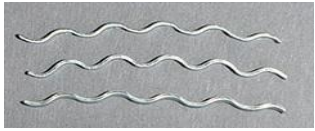


## II. MATERIALS PROPERTIES

### Fibers

Four types of fibers were used in preparing different fibrous concrete mixtures namely: hooked steel fibers (HSF), Corrugated steel fibers CSF, structural polypropylene fibers (SPPF) and traditional polypropylene fibers (TPPF) . The properties of the used fibers are listed in Table 1.



( Table 1) Geometric and mechanical properties of fibers used in the present study

Type	Geometrical configuration	Specific gravity (gm/cm3)	Fibers diameter (mm)	Fibers length (mm)	Aspect ratio
Hocked steel fibers ( HSF )		7.8	0.5	25	50
Corrugated steel fibers (CSF)		7.8	0.5	25	50
Structural polypropylene fibers ( SPPF )		0.91	0.50	50	100
Traditional polypropylene fibers ( TPPF )		0.91	-----	-----	-----

#### **Reinforcing steel**

A ribbed steel rebars B500C-R, with 16 mm in nominal diameter, and complying with ESS 262-2015, were used [18].

#### **Aggregate**

Local sand from natural sources, crushed dolomite size (10 and 20) mm complying with Egyptian standard specification ESS No. 1109- 2001 [19]. were used

#### **Cement**

CEMI 42.5N, cement complying with ESS 4756- 2013 [20] was used.

#### **Concrete Mix proportions and production**

The consistency of concrete mix was measured by slump tests, as a comparison test, and ranged from 80 to 120 mm. The content of cement, water, aggregate, fibres and super plasticizer (Sp) required to produce one cubic meter of concrete are given in Table (2)

Table (2) : Concrete compositions per one m<sup>3</sup>

Mix	Cement (kg)	Fine Aggregate (kg)	Crushed dolomite S1 (kg)	Crushed dolomite S2 (kg)	Water (kg)	Add.	Fibers			
						Sp (Lit)	* HSF (Kg)	** CSF (Kg)	*** T.PP (gm)	**** S.PP (kg)
M - C	350	700	595	595	160	7	----	----	----	----
M - CSF1	350	700	595	595	160	7.25	78	----	----	----
M- HSF1	350	700	595	595	160	7.25	----	78	----	----
M - CSF2	350	700	595	595	160	7.5	117	----	----	----
M - HSF2	350	700	595	595	160	7.5	----	117	----	----
M - CSF3	350	700	595	595	160	7.8	156	----	----	----
M - HSF3	350	700	595	595	160	7.8	----	156	----	----
M - HPF1	350	700	595	595	160	7.25		78	900	----
M - HPF2	350	700	595	595	160	7.25		78	----	2.5

\* end hooked steel fibers

\*\* corrigated steel fibers

\*\*\* Traditional polypropylene fibers

\*\*\*\* Structural polypropylene fibers



The experimental program was designed to measure the bond properties of steel reinforcing bars embedded in steel FRC and a mixture of steel FRC and polypropylene fibers (PPF). One concrete mix was not provided with any steel fibres so as to remain as a plain concrete and serve as the control concrete mix. Three mixes contained corrugated steel fibers with a various volumetric ratios of 1% , 1.5% and 2% while three mixes contained end-hooked steel fibers with a various volumetric ratios of 1% , 1.5% and 2%. The last two mixes contain a mixture of 1 % end-hooked steel fibers with one type of polypropylene fibers.

A two-steps mixing method was used at first the mortar portion, i.e (cement,sand,and water) with no coarse aggregate and no fibres was mixed in a high mixer and then mixing the mortar portion with coarse aggregate, fibres and SP in a concrete mixer was conducted. After mixing, a sample was taken for slump-flow test. If the measured slump had not reached the required value, a bit more SP was added and the concrete mix was remixed for another 1 min. After achieving the required slump, three 150mm diameter×300mm height cylinder specimens were cast for compression test and three 150 mm cube specimens each with a rebar embedded inside were cast for test. All the specimens were remoulded after casting and then cured in a lime-saturated water tank for 28 days, at a temperature of 27 ± 3 °C.

### III. RESEARCH PROGRAM




The experimental test program was designed to achieve the research objectives of the study. Bond behaviour between concrete and reinforcing bars was studied after exposure to elevated temperatures at 300 and 600°C for 2 hours. Nine different concrete mixes with different fiber

percentages were used as shown in table (1). Eighty one pull-out cylinder specimens ( $\varnothing 150$  mm, 300 mm) were prepared, then, reinforced steel bar of 16mm was embedded in the middle of each cylinder for 200mm, see figure (1-a). The reinforced steel bar's embedded length was controlled by a horizontal steel bar above the cylinders, as shown in figure (1-a). After removing the specimens from the formwork, they were stored in water for seven days then kept at laboratory conditions until testing. Finally, the specimens were tested at room temperature see (1-b) . Standard cubes were cast for each mix, cured in the same condition as the pullout cylinders specimens, then tested to determine the compressive strength.

	
<p>Figure (1-a) : Controlling the bar's embedded length</p>	<p>Figure (1-b) : Pull out Test</p>
<p>Figure (1): Preparing, casting and testing the specimens</p>	

#### IV. HEATING PROCEDURE

All specimens exposed to heat, the outer part of the tested rebar was not covered in order to simulate what may happen in real life applications. The specimens were heated in a gas furnace up to 300 °C and 600 °C. Each temperature was maintained for 2 hours before removing the specimens from the furnace and then cooled at room temperature. The gas furnace, specimens before and after exposure to elevated temprature are shown in Figure (2)

		
<p>The gas furnace</p>	<p>Specimens in the furnace</p>	<p>Specimens after exposure to elevated temperature</p>
<p>Figure (2): The gas furnace, specimens before and after exposure to elevated temprature</p>		

## V. TEST RESULTS AND DISCUSSION

### Failure Mode

Failure modes due to the pullout force changed according to the type and dosage of each fiber used in concrete specimens. The control specimens failed in splitting (the cylinder splitted into two halves), as shown in figure (3a). To avoid this type of failure mode, two types of steel fibers with different dosage were used, they obviously affected the mode of failure of tested specimens as shown in figure (3b), the tested cylinders didn't split into two halves and cracks propagated up to failure. Those cracks became narrower when 1% steel fibers were added to 1% (PP) fiber (hybird mixes), see figure (3c). For all fiber concrete mixes, bond failure partly occurs on the surface of the bar and partly in the concrete by peeling the cortical layer of the bar.







	<div style="display: flex; flex-wrap: wrap;"> <div style="width: 50%;">  <p style="text-align: center;"><b>CSF</b></p> </div> <div style="width: 50%;">  <p style="text-align: center;"><b>HSF</b></p> </div> </div> <div style="display: flex; flex-wrap: wrap;"> <div style="width: 50%;">  <p style="text-align: center;"><b>SF</b></p> </div> <div style="width: 50%;">  <p style="text-align: center;"><b>SF</b></p> </div> </div>	
<p>Figure (3a): failure of control mix</p>	<p>Figure (3b): failure of (SF) fiber mixes</p>	<p>Figure (3b): failure of hybrid fiber mixes</p>

Figure (3): Failure Modes of pullout test specimens of different mixes

## VI. Compressive concrete Strength

The compressive strength of concrete cubes were determined before and after heat-treatment .The test value was taken as the average of three cube specimens.

The reduction of compressive strength for corrugated steel fiber mixes ( M - CSF1, 2 , 3) after exposure to 300 °C for 2 hrs compared with the same mixes at room temperature were 5 % , 5 % and 6 % respectively. But after exposure to 600 °C for 2 hrs the reduction of compressive strength was 55%,53% and 53% respectively compared with the same mixes at room temperature as shown in figure (4a).

For the mixes ( M - HSF1, 2 , 3) recorded an avarege reduction of 7% after exposure to 300 °C for 2 hrs and 55 % after exposure to 600 °C for 2 hrs as shown in figure (4b).

The mixes which provided with steel fibers achieved an improvement of compressive strength with 63% if compared to the compressive strength of control Mix (M-C) at the same exposure conditions.

For the mixes which contain a mixture of steel and structural Polypropylene fibers (M - HPF1) recorded a reduction of 10 % after exposure to 300 °C for 2 hrs and 54 % after exposure to 600 °C for 2 hrs, a shown in figure (3c). On the otherhand mix (M - HPF2) recorded a reduction of 6 % after exposure to 300 °C for 2 hrs and 51 % after exposure to 600 °C for 2 hrs as shown in figure (4c).

So, clarified that steel fiber has a significant effect on concrete compressive strength subjected to elevated tempreature, such behavior may be related to: -



{a} the partial loss in bond between fibers and surrounding matrix due to the difference in their expansion coefficients at high temperatures.

{b} the higher sensitivity of fibrous concrete mixtures to high temperatures as compared to plain concrete, due to the greater strength of the fibrous concrete mixture at room temperature. Heating to 600° C generated extensive cracking and sometimes spalling in the cubic specimens, which was reduced by the use of fibers. The results showed an avarege reduction in compressive strength proportional with increasing the elevated temperature

Table (3) : Compressive strengths results before and after exposure to elevated tempertaure

Mixes	Room Temp.		300 ° C		600° C	
	Compressive strength (kg\cm2)	Residual Strength (%)	Compressive strength (kg\cm2)	Residual Strength (%)	Compressive strength (kg\cm2)	Residual Strength (%)
M - C	376	(100 %)	319	(85 %)	140	(37 %)
M - CSF1	390	(100 %)	370	(95 %)	175	(45 %)
M - HSF1	392	(100 %)	368	(93 %)	186	(47 %)
M - CSF2	401	(100 %)	381	(95%)	188	(47%)
M - HSF2	395	(100 %)	363	(92 %)	165	(41 %)
M - CSF3	412	(100 %)	386	(94 %)	195	(47 %)
M - HSF3	400	(100 %)	376	(94 %)	184	(46 %)
M - HPF1	415	(100 %)	372	(90 %)	192	(46 %)
M - HPF2	401	(100 %)	377	(94 %)	198	(49 %)

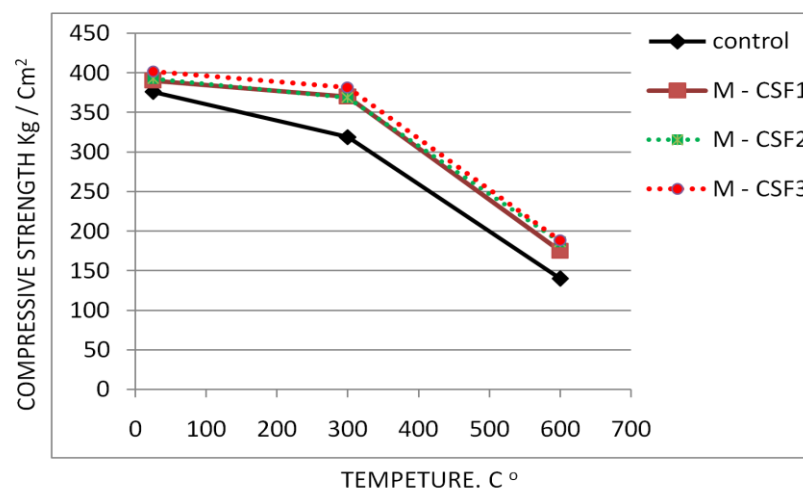


Figure (4a): Compressive strength of ( CSF) fiber mixes at different temperatures

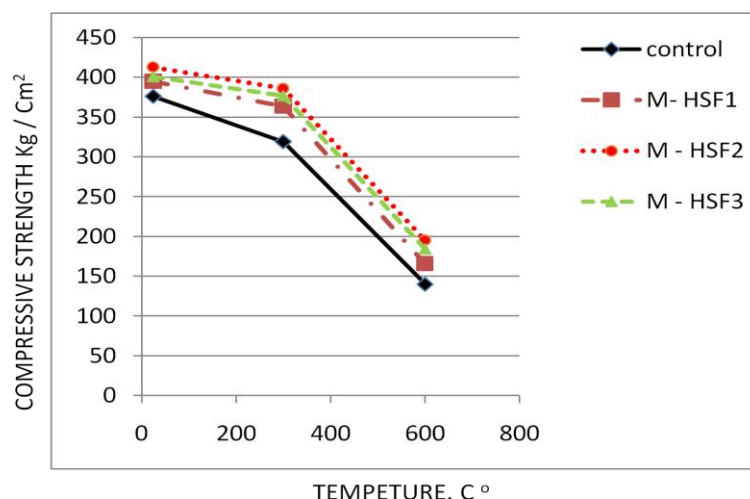


Figure (4b): Compressive strengths of ( HSF) fibers mixes at different temperatures

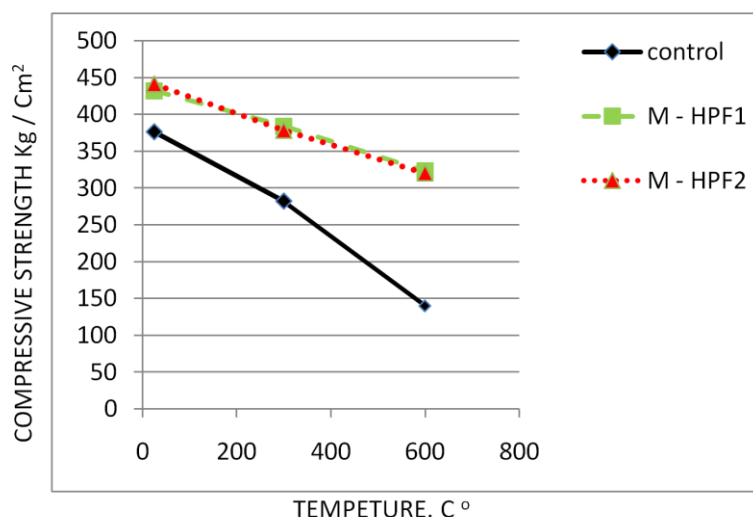


Figure (4b): Compressive strengths of ( HPF) fiber mixes (hybird mixes) at different temperatures

### Bond Strength

Bond strength between reinforced steel bars and concrete was evaluated by pull-out test after exposure to elevated temperatures (from room temperature up to ( 600 °C) according to ASTM C234-91[21]. The results state that the relative residual bond strength for all tested specimens decreased significantly with increasing the temperature . table (4) summarize the bond strength results.

#### At room temperature

For mixes ( M - CSF1, 2 , 3) the bond strength increased by the average value of 43 % compared with the control mix (M-C) . For mixes ( M - HSF1, 2 , 3) the bond strength increased by the average value of 47 %. Mix (M - HPF1) and mix ( M - HPF2 ) increase in the bond strength by 74 % and 62 % respectively. Mix ( M - CSF3 ) with volumetric ratio of 2 % gives the reasonable improvement in bond strength, and so it can be conclude that the percentage of fibers in the range of 2 % is considered an appropriate proportion to increase the bond strength. on the other hand, mix (M - HPF1) gives the best improvement in bond strength as shown in figure (5).

#### At ( 300 °C) temperature

The average residual bond strength for the control mix (M-C) recorded the worst value of 25 %, while the average residual bond strength for mixes ( M - CSF1,2,3) was 37 %. For mixes ( M - HSF1,2,3) The average residual bond strength was 40 %. On the other hand the average residual bond strength



for mixes (M - HPF1) and ( M - HPF2 ) was 46 % and 49 %, respectively. it can be seen that mix (M - HPF2) gives the best improvement in residual bond strength as shown in figure (5).

#### At ( 600 °C) temperture

The average residual bond strength for the control mix (M-C) recorded the worst value of 10 %, while the average residual bond strength for mixes ( M - CSF1,2,3) was 21 % . For mixes ( M - HSF1,2,3) the average residual bond strength was 24 %. On the other hand the average residual bond strength for mixes (M - HPF1) and ( M - HPF2 ) was 26 % and 27 %, respectively. it is obvious that, Mix (M - HPF2) gives the best improvement in residual bond strength as shown in figure (5).

From the previous results it can be seen that the mixes ( M - CSF1,2,3) and ( M - HSF1,2,3) achieve an improvement in the residual bond strength of 48% and 60% ,respectively at 300°C, and 110% and 140% ,respectively at 600°C compared with the control Mix (M-C) at the same exposure conditions because of the use of fibers reduced crack sizes on the surfaces of modified pullout specimens and limited or prevented crack propagation throughout the modified pullout sides.

The mixes (M - HPF1) and ( M - HPF2 ) achieve an extra improvement in the residual bond strength of 160% and 170% ,respectively at 600°C compared with the control Mix (M-C) at the same exposure because of the escape channels formed by melting of the polypropylene fibres, impart limited improvement in bond resistance against heating because of the reduction in both splitting and compressive strengths as a result of the increase in the overall porosity.

Table (4) : bond strength results before and after exposure to elevated tempertaure

Mixes	Room Temp.		300 ° C		600° C	
	Bond strength (kg\cm <sup>2</sup> )	Residual Strength (%)	Bond strength (kg\cm <sup>2</sup> )	Residual Strength (%)	Bond strength (kg\cm <sup>2</sup> )	Residual Strength (%)
M - C	74.00	(100 %)	18.50	(25 %)	7.50	(10 %)
M - CSF1	91.00	(100 %)	29.00	(32 %)	17.00	(19 %)
M - HSF1	96.00	(100 %)	34.00	(35 %)	20.00	(21 %)
M - CSF2	103.00	(100 %)	37.00	(36%)	22.60	(22%)
M - HSF2	110.00	(100 %)	44.00	(40 %)	27.50	(25 %)
M - CSF3	125.00	(100 %)	52.50	(42 %)	26.00	(21 %)
M - HSF3	120.00	(100 %)	54.00	(45 %)	29.00	(24 %)
M - HPF1	129.00	(100 %)	59.00	(46 %)	33.00	(26 %)
M - HPF2	122.00	(100 %)	60.00	(49 %)	33.00	(27 %)

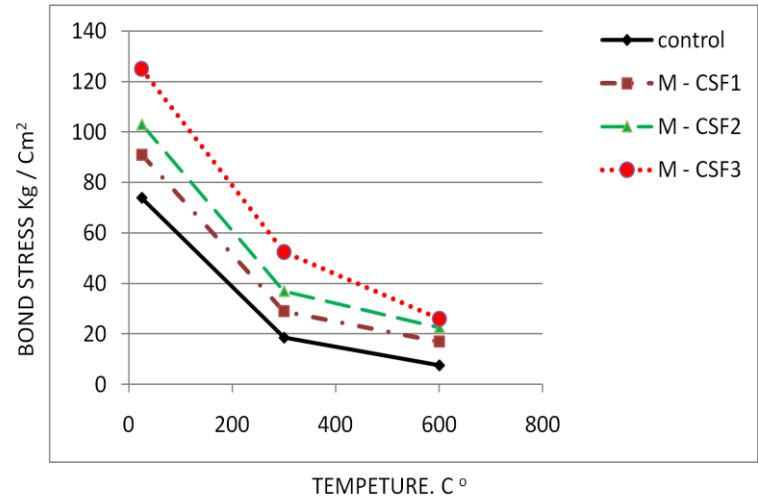


Figure (5a): Bond strengths of (CSF) fiber mixes at different temperatures

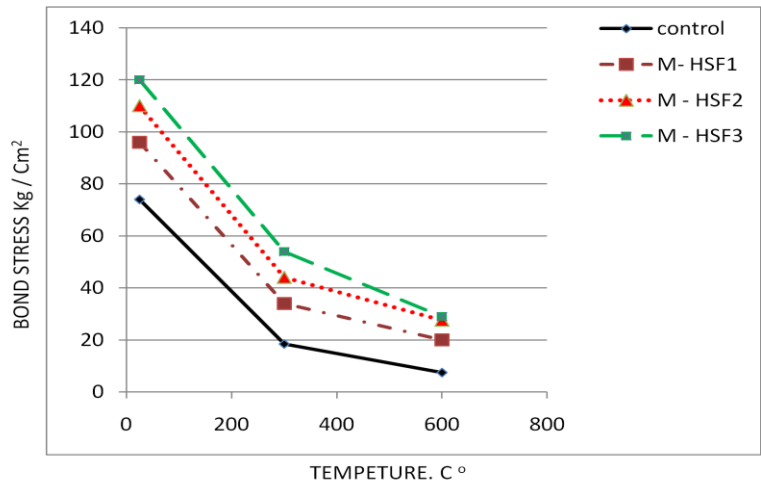


Figure (5b): Bond strengths of (HSF) fiber mixes at different temperatures

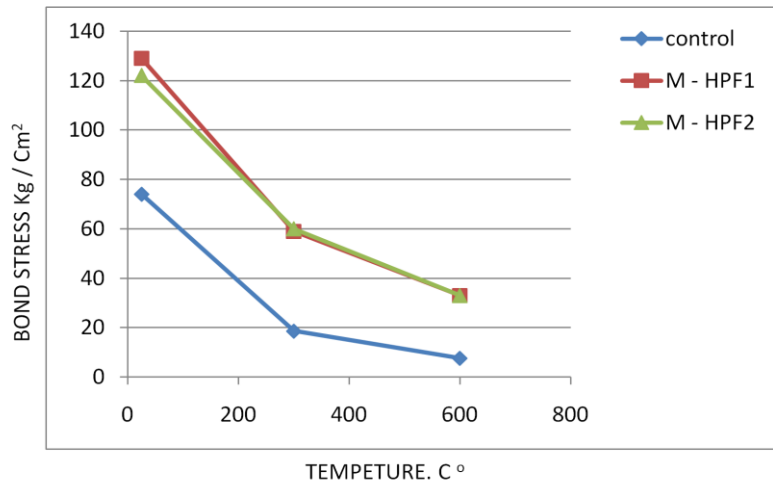


Figure (5c): Bond strengths of (HPF) fiber mixes (hybird mixes) at different temperatures

Conclusions

The following main conclusions can be drawn from the experimental results

experimental results:

1. Using corrugated steel fibers (CSF), hooked steel fibers (HSF) and hybrid fibers slightly improved the concrete compressive strength by 6.6, 5.2 and 8.50 %, respectively. While significantly enhance the bond strength between the reinforced bars and concrete by 43 , 47 and 68 %, respectively.
2. Exposure to 300 °C for 2 hrs. didn't affect the average of both compressive and bond strength significantly, as the average strength loss didn't exceed 16% of those in room temperature for all mixes,
3. Exposure to high temperature of 600 °C for 2 hrs. significantly reduces the compressive strength of no fiber concrete by 63% and that reduction improved by using corrugated steel fibers (CSF), hooked steel fibers (HSF) and hybrid fibers to reach 53 , 55 and 52.5 %, respectively of the original compressive strength for specimens in room temperature.
4. Exposure to high temperature of 600 °C for 2 hrs. significantly reduces the ultimate bond strength of no fiber concrete by 90% and that reduction enhanced by using corrugated steel fibers (CSF), hooked steel fibers (HSF) and hybrid fibers to become 79, 74 and 73%, respectively of the original bond strength for specimens in room temperature.
5. An improvement of bond strength compared to the control specimen after exposure to 600 °C up to 54 % occurred when using both types of (SF) fiber, while the greatest enhancement was recorded 62 % in hybrid mixes containing both (PP) and hooked steel fiber together .
6. Using of 1% steel fibers with 1% PP fibers recorded the highest bond strength among all tested specimens whether subjected to elevated temperature or not.
7. The control specimens failed in splitting (the cylinder splitted into two halves), but using the two types of steel fiber ,separately, obviously affected the mode of failure of tested specimens as the tested cylinders didn't split into two halves and cracks propagated up to failure. Those cracks became narrower when hybrid fibers were used.
8. For all fiber concrete mixes, bond failure partly occurs on the surface of the bar and partly in the concrete by peeling the cortical layer of the bar.

## REFERENCES

- [1.] Bond of Reinforcement in Concrete: State-of-Art Report, Bulletin 10. fib (CEB-FIP), 2000.
- [2.] L. Biolzi, S. Cattaneo, and G. Rosati, "Evaluating Residual Properties of Thermally Damaged Concrete," Cement & Concrete Composites, 30, 907– 916, 2008.
- [3.] C. Castillo, and A. J. Durrani, "Effect of Transient High Temperature on High Strength Concrete," ACI Materials Journal, 87(1), 47–53, 1990
- [4.] L. T. Phan, and N. J. Carino, "Review of Mechanical Properties of HSC at Elevated Temperature," Journal of Materials in Civil Engineering, 10(1), 58–64, 1998.
- [5.] F. M Özkal et al, "Mechanical Properties and Bond Strength Degradation of GFRP and Steel Rebars at Elevated Temperatures", Construction and Building Materials 184 45–57, 2018
- [6.] H.M. Selim et al, " Bond Characteristics of ASTM A1035 Steel Reinforcing Bars", ACI structural journal, July/ August, 530:539. 2009
- [7.] N. G.,et al, " Evaluation of Bond Strength Between Steel Rebars and Concrete for Heat Damaged and Repaired Beam-End Specimens", Cement and Concrete Research journal. 32 1009–1018. 2002,

- [8.] M. Haskett, D. J. Oehlers, and M. S. M. Ali, "Local and Global Bond Characteristics of Steel Reinforcing Bars," *Engineering Structures*, 30, 376–383, 2008.
- [9.] R. H. Haddad, R. J. Al-Saleh, and N.M. Al-Akhras, "Effect of Elevated Temperature on Bond Between Steel Reinforcement and Fiber Reinforced Concrete," *Fire Safety Journal*, 43 334–343, 2008.
- [10.] Annual Book of ASTM Standards, Concrete, American Society for Testing Materials, 1993.
- [11.] RILEM, Bond test for reinforcement steel: 2-pull-out test, Recommendation RC6, CEB News 73, 1983.
- [12.] V. Kodur, R. McGrath, Fire Endurance of High Strength Concrete Columns, *Fire Technol.* 89 (2003) 73–87.
- [13.] P. Morely, R. Royles, Response of The Bond in Reinforcing Concrete to High Temperatures, *Mag. Concr. Res.* 35 (123) 67–74. 1983
- [14.] J. P. C. Rodrigues, L. Laim, and A. M. Correia, "Behaviour of Fiber Reinforced Concrete Columns in Fire," *Composite Structures*, 92, 1263– 1268, 2010.
- [15.] A. Lau, and M. Anson, "Effect of High Temperatures on High Performance Steel Fiber Reinforced Concrete," *Cement and Concrete Research*, 36, 1698–1707, 2006.
- [16.] E.P.S de Linares, University of Jaén, Spain GEA 21, Spain, " The Fire Resistance of Concrete with Polypropylene Fibers", *MATEC Web of Conferences* 6, 02005 (2013).
- [17.] Éva Lublőy and Balázs L. György "Temperatures Effects on Bond Between Concrete and Reinforced Steel", *Journal of Faculty of Civil Engineering*, 26 (2014).
- [18.] ESS 262-2015, "reinforced steel in concrete" .
- [19.] ESS No. 1109- 2001," Aggregate for Concrete" .
- [20.] ESS No. 4756- 2013, "Cement, Part One; Composition, Specifications and Conformity Criteria for common Cements".
- [21.] ASTM C234-91a, Standard Test Method for Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel

# Analysis and Evaluation of the Theory Queue Management in a Company Instrument Branch Musical

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## ABSTRACT

The present work was carried out in October aiming to analyze the behavior of the queue in the productive system of a musical instrument company, established in the city of Leopoldina MG, associated with the tools of the Operational Research discipline. The research is based on Evaluating the FIFO queue management method and system deploying the SJF - NP in order to optimize the time in the production process. The results Showed que there was a better use of production with the SJF-NP system, with a decrease of 16.4 minutes, equivalent to 21.7% reduction in production team.

**Keywords**— FIFO Queue Management, SJF-NP

## I. INTRODUCTION

Today, with the increasingly competitive market, it is increasingly common for companies seeking for new tools and methodologies to achieve best practice in their production processes. Using these strategies the organization can raise their profits and minimize costs generated by time spent in the production process. These generated optimizations allow greater clarity and agility in the supply chain can thus find a balance with little investment.

Therefore, it uses operational research to diagnose operational problems, activities and routine operations and production. The finding of a problem leads to scientific method of construction was carried out with data collection and mathematical calculations in search of a better solution to the problem encountered [1].

Among the problems are commonly encountered is the formation of queues. According to Almeida et al (2016) these are routine in establishments where there is a large flow of people and operations [2]. For the authors waiting in lines is something uncomfortable to customers and can bring the same discontent. In operations research, in search of the solution to that problem applies to the Theory of Queues, which according to Romero et. al (2010), is understood as a science that is intended to address issues involving the waiting time for customers to services demanded [3].

In this context, the queuing theory is a methodology that applies the steps of positioning which offers several models processes for training queues, in order to minimize the waiting time and maximize production capacity.

The aim of this paper is to analyze the methodology applied in the order of lines of production of a branch company of musical instruments, and assess through comparative statements and the performance achieved.

## II. METHODOLOGY

This work was carried out a quantitative and descriptive research based on the case study method in a branch company of musical instruments, established in Leopoldina City, Zona da Mata Mineira.

According to Gil (2010) case study is a research methodology in the social sciences which is characterized by a detailed study of one or a few objects, allowing for ample notice and detailed study of the object in generating knowledge[4].

Data for this study were collected by on-site visits at the date of 11.11.2019, when the measurement of time spent for making order products given the company's service was held for further calculation and analysis of the management of existing queue .

Therefore, we used an actual service order designed the Art Finalist sector of the organization, which are finished products already reach semi finished the painting and production sectors. The measurement of time has been realized with the help of a timer, so that were timed continuously all stages of production, so as not to interfere with the routine production workers. idle times were not recorded as a pause for rest, bathroom, coffee and lunch.

After the survey data, with the aid of Excel software were carried out simulations of time of completion of that work order, in the FIFO and SJF-NP methods of managing queues, to perform comparison and analysis in order to determine the model to better attend the production sector.

## III. THEORETICAL

### A. Queuing Theory

In designing Krajewski (2012) the queue happens when you have an unstable disharmony between the degree of demand for a given system and the ability of this to meet this demand [5].

With early in the twentieth century, the theory of queues is set by applications in various areas, and known to be a mathematical study of lines or queues from arrival in the queue, past the waiting until serviced. For this fact, allows the calculation and the derivation of various performance measures, some of them: average waiting time in the queue or the system, estimate of expected or performance of the service and the likelihood waited for a while to be served [6].

According to Pereira et al. (2016) this theory works the system clogging problems where the main feature is the service request by "clients" [7]. To this end, the queuing theory depicts a procedure whereby its function, distribution of arrival and service, plus the number of servers, system capacity and the size of the population can be represented as follows for each model differing in some specific aspects [8].

Are elements of queues, according to Prado (2014):

"a) customer arrival process: one can quantify the arrival process saying that the average rate is a number of entities per unit of time. It is common to work with the average time between arrivals. Thus, the rate of arrival ( $\lambda$ ) of 20 customers per minute, representing the average interval between arrivals (CI) 3/2;

b) care process: it can also be quantified, as of the arrival process. Thus resulting in the pace of service ( $\mu$ ) and service time (TA);

c) Discipline of the Line: refers to the rule that the servers choose the next customer to be served. In practice it is adopted: First in First Out (first come first served - FIFO), Last In First Out (last come first served basis - LIFO) and Priority;

d) The ability of a system: Represents the maximum number of clients that support system and may be finite or infinite. [9]"

Andrade (2011), the study of queuing theory allows the organization to build information and a standby time of estimation of its customers in line, still determines the size of the queue and the best system to be used to minimize the waiting and enhance production or service [10]. According to Perera et al (2014, p 12) "management of queues and their variability deserve attention from service providers because of its influence with respect to the customer, they represent much of the satisfaction with the quality of service.[11]" For the authors, the management of queues is necessary in that it prevents customer dissatisfaction and brings agility and versatility in production. Define abbreviations and acronyms the first time they are used in the text, even after they have been defined in the abstract. Abbreviations such as IEEE, SI, MKS, CGS, sc, dc, and rms do not have to be defined. Do not use abbreviations in the title or heads unless they are unavoidable.

#### *B. Instrument*

Latin American percussion instrument, the cajón, which has its origin from African slaves passed through the improvement process in Peru for decades and was taken to Spain in the 70s to adapt to the needs and European musical costumes [12].

The instrument is played when the musician sits on it, where practicing strokes with hands hurt sounds, and when it hit the top of the front cover produces high tones and hitting the central lid produces the bass [13].

With friendly instrument, also known as "magic box" can produce the most diverse arrangements, from a simple melody to the accompaniment to a more elaborate drumming. The instrument, which received the Peruvian government the title of "Cultural Heritage of the Nation", has been used to monitor many more styles for acoustic performances [14].

### **IV. RESULTS AND DISCUSSION**

#### *A. Case Study*

The organization analyzed is a company located in the city of Leopoldina, the region of the forest zone of the state of Minas Gerais, whose main activity is the manufacture of drawer musical instrument.

The company is in business for five years and currently has 27 employees and has its highest sales volume by virtual means. In addition to the cajón the company manufactures accessories for the instrument and the instrument Drum Box Set, developed by hand, consisting of a battery derived from Cajon.

The option for the organization was motivated by the availability of the same to provide data / information and allow the observation and analysis of the production system and application of queuing theory in their activities.

#### *B. Application of the Theory of queues*

In order to analyze the operation of the queue management system in the production process of the organization under study, we used the Excel software to perform simulations, analysis and comparison across the table where they were related 05 corresponding products of a production order, as shown in Figure 1 below:



Figure 1: Production Order

**Ordem de Produção Nº 2019/00336**  
 Tipo de Produto: 04 - Produto Acabado

**Família**  
 <não informado>

**Quantidade a Produzir**  
 48,0000 UN

**Unidade**  
 Unidade (UN)

**Itens da Ordem de Produção**

Descrição do Item	Quantidade	Unidade	Tipo do Produto
WDPA-0013	6,0000	UN	04 - Produto Acabado
WDPA-0032	20,0000	UN	04 - Produto Acabado
WDPA-0018	7,0000	UN	04 - Produto Acabado
WDPA-0047	3,0000	UN	04 - Produto Acabado
WDPA-0008	12,0000	UN	04 - Produto Acabado

**Outras Informações**  
 Início da Produção: 11/11/2019  
 Conclusão da Produção: 11/11/2019

Source: Company analyzed

Figure 1 shows a real model of a usual production order within the company. As proposed by the study in this figure contains five (05) being manufactured products in industry Art finalist company indicating its description, quantity and type of product respectively.

For structuring the tables, the columns were found (Process) for the products to be produced (Home / Prod) for sequencing the queue to be produced (weight) for the quantities to be produced (Temp / MED) for time Average production for each product (Length) to the total time of production and (Priority) for products with greater relevance to be produced, as shown in figure 1.

Figure 2: Table basis for the simulations

							Desmembramento da Duração em minutos.													
Processos	Início/Prod.	Quantidade	Tempo Médio	Duração	Prioridades	Formula	1	2	3	4	5	6	7	8	9	10	11	12	13	14
P1 - CAB	0	6	00:05:00	00:30:00	5															
P2 - VAS	1	20	00:02:30	00:50:00	2															
P3 - CAR	2	7	00:07:00	00:49:00	4															
P4 - SHA	3	3	00:03:00	00:09:00	1															
P5 - CAN	4	12	00:06:20	01:16:00	3															

Source: The author

Figure 2 shows the table structure that has been used to obtain the simulation where the criterion for filling and reading thereof is 1 minute counts for each column of time intended for production of products.

The first method was subjected to simulation FIFO, where the sequencing was performed and filling of the squares in the order of production according to the time taken to obtain each product. After all the fills were calculated duration in minutes (Dismemberment duration in Minutes), minus the total manufacturing time (duration) and early manufacturing (Home / Prod.). From the obtained results were

calculated from the average of manufacturing times and the final result in the average manufacturing minutes (quantity - Average).

Figure 3: Simulation method FIFO

FIFO							Desmembramento da Duração em minutos.											
Processos	Início/Prod.	Quantidade	Tempo Médio	Duração	Prioridades	Formula	1	30	32	81	83	131	133	141	143	218		
P1 - CAB	0	6	00:05:00	00:30:00	5	0												
P2 - VAS	1	20	00:02:30	00:50:00	2	30												
P3 - CAR	2	7	00:07:00	00:49:00	4	80												
P4 - SHA	3	3	00:03:00	00:09:00	1	129												
P5 - CAN	4	12	00:06:20	01:16:00	3	138												
QUANT - Média							75,4											

Source: The author.

Figure 3 shows as a result the average amount of 75.4 minutes to manufacture products of certain production order. For best performance in the table analysis, we packed up the squares of the minutes at longer intervals of time (only informing start and end time).

Then we carried out a simulation using the queue management system SJF-NP, started from the sequencing and filling the square, initially following the listed production order. Then filler was used as criterion, the most relevant products to be produced until the end of the manufacturing process. After all fills, as in the previous methodology, we calculated the duration in minutes (Dismemberment duration in Minutes), minus the total manufacturing time (duration) and early manufacturing (Home / Prod.). With the results were calculated the average of the manufacturing times and with the end result in the average minutes of manufacture (Quant - Average).

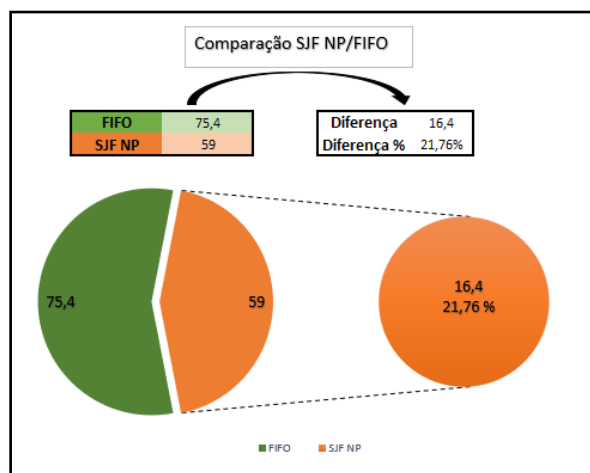
Figure 4: Simulation Methodology NP SJF

SJF NP							Desmembramento da Duração em minutos.											
Processos	Início/Prod.	Quantidade	Tempo Médio	Duração	Prioridades	Formula	1	30	32	40	42	90	92	141	142	218		
P1 - CAB	0	6	00:05:00	00:30:00	5	0												
P2 - VAS	1	20	00:02:30	00:50:00	2	90												
P3 - CAR	2	7	00:07:00	00:49:00	4	39												
P4 - SHA	3	3	00:03:00	00:09:00	1	28												
P5 - CAN	4	12	00:06:20	01:16:00	3	138												
QUANT - Média							59											

Source: Author

Figure 4 shows the average amount as a result of 59 minutes to manufacture products of certain production order. For best performance in the analysis table, we used the same compression method as described in figure 2.

Figure 5: Comparison between the methods.



Source: Author

The data presented in Fig 5 are related to the results comparing the two methods used. In comparison it was observed that there was a decrease of 16.4 minutes in the production process, equivalent to 21.7% reduction in production time.

Thus, after analyzing for queue management, the tested methods, remains proven that the company and production in question the most effective method is the SJF NP due to optimization of time in the production process in its application.

## V. FINAL CONSIDERATIOS

The Operational Research aims to use one of its queue management theory of tools to optimize the management of time in production. The queue management allows the reduction of the waiting time in the queue, by optimizing the production process, thereby promoting certain agility in the production chain and avoiding the discomfort and dissatisfaction of customers waiting for products.

In the case study, analysis of the results of the application of simulations of the queue management methods, suggests the application of SJF-NP model, which according to the mathematical calculations promoted the optimization of the production system, which has succeeded in reducing 21.7 % in the average manufacturing time. Thus, it is evident that the objective proposed by the said work was achieved with the proposal of an ideal model for the management of queues in the company.

## REFERENCES

- [1] HILLIER, Frederick. Stanford; Lieberman, Gerald J. Introduction to operational research. 9. ed. Porto Alegre: AMGH, 2013.
- [2] ALMEIDA, Amanda Claunino; PINA, Gabriel Silva; LIMA, Lucas Erick Pereira; MONTEIRO, Robert Romano; SANTOS, Yvelyne Bianca lunes. Application of the Theory of Queues in a bakery in the city of Belém / PA. Proceedings of the XXIII Production Engineering Symposium. Bauru - SP, 2016. Available at: <[http://www.simpep.feb.unesp.br/abrir\\_arquivo\\_pdf.php?tipo=artigo&evento= 11 & art = 114 & cad = 22579 & option = com\\_id](http://www.simpep.feb.unesp.br/abrir_arquivo_pdf.php?tipo=artigo&evento= 11 & art = 114 & cad = 22579 & option = com_id)> Accessed on: 22.11.2019.
- [3] ROMERO, Camila Mendonça; SALES, Diego da Silva; VILAÇA, Luisa Lemos; CHAVEZ, Jose Ramon Arica; CORTES, Jacqueline Magalhães. Application of queuing theory on maximizing the flow of pallets in a chemical industry. Operations Research for Development, v.2, no.3, 2010, p.226-231.
- [4] GIL, Antonio Carlos. How to design research projects. 5th ed, São Paulo: Atlas, 2010K.
- [5] Ritzman, Larry. P.; Krajewski, Lee. J. Administration of production and operations, 8th. ed. São Paulo: Pearson Prentice Hall, 2012.
- [6] SANTOS, Paulo Henrique; SILVA, Solange's; Bucchianeri, Juliano Resende; MATA, Nilson Oak; MORAIS, Thiago Moura. Analysis of the truck loading of a distribution center in Annapolis / GO: A study on modeling and simulation of M queuing systems / M / 1, comparing the analytical model to the simulated model. Proceedings of XLIX Symposium of Operational Research Blumenau-SC 27 to August 30, 2017.. J. Magn. Japan, vol. 2, pp. 740-741, August 1987 [Digests 9th Annual Conf. Magnetism Japan, p. 301, 1982].
- [7] Pereira, Lucio Adan et al. Analysis of Congestion Problems of a Steel Plant Canteen. Brazilian Journal of Production Engineering-BJPE, v. 2, no. 1, p. 53-71, 2016.
- [8] Merelles, Leonardo RO; Dantas, Maria JP; Menezes, Jose E .; Dias, S. Vivianequality control of the application to determine the heating period in simulation queues. Proceedings of XLIX Brazilian Symposium on Operations Research, Blumenau-SC 27 to August 30, 2017.
- [9] PRADO, Darci. Queues theory and simulation. 5th ed. Falconi, 2014
- [10] ANDRADE, Eduardo Leopoldina. Introduction to Operations Research: Methods and models for decision analysis. 4. ed. Rio de Janeiro: LTC 2011.
- [11] PEREIRA, Ricardo Fazonato; SOARES Lorena Paula; GONÇALVES, Rafaela. Queue management: a case study of a company providing outsourced service in the digital printing industry. XXXIV NATIONAL PRODUCTION ENGINEERING MEETING, Curitiba, 2014. Available at: [http://www.abepro.org.br/biblioteca/enegep2014\\_TN\\_STP\\_195\\_101\\_24487.pdf](http://www.abepro.org.br/biblioteca/enegep2014_TN_STP_195_101_24487.pdf). Accessed on 20/11/2019
- [12] EL CAJON Peruvian. Available in <http://www.musicaperuana.com/cajon/> access 11.08.2019
- [13] FAT CONGAS: Available at <[http://www.fatcongas.com/Product\\_Review.html](http://www.fatcongas.com/Product_Review.html)>. Accessed on 11/08/2019.
- [14] WITLER DRUMS. Available in: [https://www.cajonwitlerdrums.com.br/pagina.php?info\\_id=11](https://www.cajonwitlerdrums.com.br/pagina.php?info_id=11). Accessed: 10/11/2019

# Environmental Protection within a Gas Production Section

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## ABSTRACT

*The motivation of choosing the research of the theme is the immeasurable importance that the protection of the environment has in carrying out any activity, and in meeting the objectives.*

*The study focuses on the protection of the environment, which represents: the set of measures, actions and regulations aimed at maintaining, protecting and improving natural environmental conditions, reducing and eliminating, where possible, environmental pollution and pollution sources, for the purpose fulfillment of the activities and objectives described within the Gas Production Section . Only an extremely rigorous and constant approach at all levels of the objectives of the Section can lead to an efficient control over the activities and to the reduction of risk factors.*

**Keywords**—environment, environmental protection, waste, wells, waste management, Gas Production Section

## I. INTRODUCTION

The natural gas industry can affect physical environmental factors through the following large groups of successive activities: prospecting, drilling and drilling (onshore and offshore); transport of natural gas and derived products; conditioning of natural gas; storage, distribution and use of natural gas. In order to protect the environment, we identify and analyze the possible sources of pollution, and in direct agreement with environmental policies we manage the waste produced as a result of the activities within a Gas Production Section.

## II. THE MAIN POSSIBLE SOURCES OF POLLUTION

At the drilling wells: drilling fluids, materials used for landscaping, debris resulting from drilling, settling of land and roads, following the completion of the works to restore the affected land according to the zonal landscape and play in the agricultural circuit;

In the wells in production: leaks in the environment of the fluids from the wells spreading on the ground of substances and materials with which operations of stimulation, injection, sand consolidation, hydraulic cracking and acidification that are performed;

At wells in capital repairs: circulation and killing fluids, as well as other fluids from the well;

For pipes: phenomena of wear, corrosion, cracking, breakage, mechanical deformation;

At separator and reservoir parks: gas emissions into the atmosphere, accidental liquid discharges, sludge from cleaning tanks, separators, decanters, stored sludge;

At waste and / or industrial water injection stations: liquid discharges outside the permissible limits;

In industrial areas and utilities: noise pollution, vibration;

Concerning the access roads: removing from circulation some areas of agricultural land, forest affected by the respective objective, in operation;

### **III. PRESENTATION OF A GAS PRODUCTION SECTION**

The Gas Production belongs to the Production Branch, being directly subordinated to the Production Manager of the Branch and has in its composition: 168 employees {Extraction operators, mechanical locksmiths, intervention team, training heads, Tesa personnel (senior civil servant, economist, engineers, deputy section chief and section head)}; 13 gas fields; 39 working points; 333 natural gas wells in production; 9 Gas drying stations (6 stations with triethylene glycol and 3 stations with delusional salts); 3 field compressors; 4 wastewater injection stations; 6 wastewater injection wells; 1 Waste deposit specific to the gas extraction activity.

I mention some of the main activities carried out: Natural gas extraction; conditioning of natural gas; compression of natural gas; optimization of natural gas production; safe operation of production capacities; carrying out maintenance and annual revisions to all the production infrastructure (Break heads, adduction pipes, collecting pipes, field compressors, gas drying stations, measuring and control devices); supply, management, and distribution of materials and spare parts; elaboration and analysis of the working procedures in force at the Company level; coordination and supervision of the entire staff of the Section; waste management resulting from the production activity.

The Gas Production section has the following objectives: Carrying out the annual Production Plan; incorporation in the annual technological consumption; carrying out the works of interventions and re-equipping wells; conducting annual technical revisions to well groups and technological installations, collecting pipes, wastewater injection stations, TEG gas / salt drying stations; compliance with environmental requirements; failure to record any work-related injury or occupational disease; continuous assurance of the quality of the natural gas delivered; increased production capacity; increasing the portfolio of gas resources and reserves, by discovering new resources and by developing and improving the recovery of resources already discovered; identifying new opportunities for growth and diversification; increasing the performance of the company, and of the staff employed.

### **IV. STATEMENTS OF ENERGY POLICY AND OF THE INTEGRATED MANAGEMENT SYSTEM**

At the company level there are published:

Energy Policy Statement, which has the following objectives: Communication throughout the organization of the importance of energy management for continuous improvement of energy performance, detailed analysis of energy flows and consumption on each process in order to determine the opportunities for continuous improvement of energy performance, compliance with applicable legal requirements and with other requirements to which the organization has subscribed regarding the use of energy, energy consumption and energy efficiency, awareness and education of employees in spiritual energy saving and use of clean energy (solar panels), reducing the impact on the environment and the costs of energy use, promote the purchase of energy efficient products and services

Policy statement in the field of integrated management, mentioning some of the objectives: Prevent pollution and reduce the unwanted effects of our operations against the environment, protection of the occupational health and safety of the employer, through adequate control of the working environment, continuous monitoring and systematic analysis of the processes carried out in order to ensure their efficiency and effectiveness, responsibility of each employee regarding the personal contribution to the performances of the integrated management system of quality, environment, health and occupational safety, development of production capacities with higher yields, in order to use resources efficiently and in order to reduce the quantities of waste generated in their operation, reducing air pollution by reducing the amount of pollutant emissions (CO<sub>2</sub>, NO<sub>x</sub>), by using new, high-performance technologies that contribute to increasing energy performance, maintaining within the permissible limits of the quality of the water discharged into the natural stream, concomitant with the significant reduction of the volumes of cooling water discharged.

## V. THE ENVIRONMENTAL AUTHORIZATION

The environmental authorization for the Gas Production Section, natural gas extraction through wells located in the 13 gas structures, and which contains:

a) The authorized activity: Equipments: installations, equipment, means of transport used in the activity (337 wells in production, 6 wells for injection of water from the field, 9 gas drying stations 3 field compressors, one headquarters); Raw materials, auxiliaries, fuels, packaging used - way of packaging, storage, quantities (fuels, methane gas, lubricants, triethylene glycol, foam, methanol, alkyd paints, primer, diluents, lithium chloride, calcium chloride; Utilities: industrial water, drinking water, electricity, natural gas; Description of the main phases of the technological process or other activities; natural gas production, interventions at wells, special operations, cracks, acidification, maintenance of access roads; The obtained products and by-products; Data regarding the thermal power plant - equipment, used fuels;; Other specific data; The operating program

b) Environmental protection installations, measures and conditions; Stations and installations for the retention, evacuation, and dispersion of pollutants in the endowed environment: reservoirs water reservoir, sewage treatment plant, decanter, (Periodically water withdrawals are made and bulletins for analyzing liquid samples for wastewater are discharged from the station purification-evacuation in the emissary); baskets for the evacuation of flue gases in the atmosphere, the construction of injection wells, which does not allow the migration of water in groundwater; Other special arrangements, equipments and measures for environmental protection: transport of reservoir water, free flow of wells, ensuring soil protection; Concentrations and mass flows of pollutants, noise levels, radiation, admitted to evacuation in the environment, permissible exceedances and in what situations,

c) Monitoring of the environment: Physico-chemical, bacteriological, and biological indicators emitted, pollutant emissions, frequency, mode of exploiting the results: determination of the concentration in the pollutants of the flue gases, measurement of the noise level; The data to be reported to the territorial authorities for environmental protection and periodicity;

d) The way of managing the waste and packaging: Waste products: types, composition, quantities; Waste collected: types, composition, quantities, frequency; Waste temporarily stored: types, composition, quantities, storage mode; Valor Waste used: types, composition, quantities, destination; Waste transportation mode; Removal mode; Monitoring of waste management; Used packaging and results; The management of the packaging

e) The mode of management of dangerous substances and preparations: Hazardous substances and preparations produced, or used; UI Management mode; The mode of management of packaging used or resulting from hazardous substances; Installations, equipment arrangements, and protective measures; Monitoring the management of dangerous substances and preparations

f) The compliance program - measures to reduce the present and future effects of the activities: Domain: soil and groundwater protection: this is not the case; Source of financing: not the case



Authorization of water management regarding: "Probes for injection of water from the field belonging to the Gas Production Section"

We also hold Notification of the functioning of the objective of the Gas Production Section, comprising the general data of the section, water supply, waste water discharge, quality indicators and obligations, and Environmental authorization regarding the Waste Deposit Specific to the gas extraction activity

We also have contracts for sale, purchase or service, for: Waste oils derived from the activity carried out; Recyclable industrial waste: iron, paper, plastic; Sludge removal and transport services; Municipal waste collection / transport services; Controlled collection, transport and disposal of triethylene glycol waste and emulsion waste; Controlled transport and disposal services for hazardous waste generated on company sites; Use / exploitation of groundwater resources

## **VI. PREVENTIVE MEASURES REGARDING THE ENVIRONMENTAL PROTECTION**

Preventive measures regarding the Environmental Protection for a good organization regarding the management of hazardous and non-hazardous materials, of waste, and to avoid accidents and environmental incidents at the Gas Production Section, the following documents are taken and the following documents are drawn up: Plan for preventing and combating accidental pollution at potentially polluting water uses, where the mode of action is passed, the persons and tasks drawn to each person involved; List of addresses of interest of the regulatory and control authorities and of other institutions with which our company headquarters collaborates, in the field of environmental protection; Environmental communication diagram; List of significant environmental issues; Program of actions for achieving the environmental objectives; List of critical points generating potential ecological accidents; List of dangerous chemical substances and preparations; The record of the packages delivered; Secure register for loading and unloading registration of hazardous waste; List of environmental normative acts; List of waste generated from the activities of the section; Waste records; The situation of the injected reservoir water; The situation of the injection wells; Situation regarding industrial water: injected water, technological water consumption, industrial water from wells, water delivered to consumers; List of observed environmental events: runoff water at wastewater installations; The storage of dangerous substances (HCl, methanol, diethylene glycol, triethylene glycol, gasoline, diesel, paints, diluents) is done in separate containers or buildings;

Example of the actuation procedure in the event of an imminent threat or the occurrence of a reservoir water leak:

Head of formation:

- a) Take immediate measures to prevent and limit the effects of leaks, evacuating the reservoir water from the damaged bay;
- b) Immediately notify the head of the section about the imminence of the production, or about the production of spills of chemical substances or preparations

Head of Section:

- a) Immediately notify the production manager and the head of the environmental protection service in the branch, about the ecological accident or potential accident.
- b) Summons the intervention team.
- c) It provides the materials, equipment, equipment and human resources, necessary for the actions of limitation and / or prevention on the environment, of the product / potential accident.
- d) Initiates the report of the observed / potential non-compliance, and records the non-compliance in the non-compliance register online.
- e) Upon completion of the corrective / preventive actions, close the non-compliance in the online register.



Head of environmental protection service:

- a) Informs about the ecological accident produced or potential, with the agreement of the branch manager, the county agency for environmental protection and the county commissioner of the National Environmental Guard, respecting the legal requirements of the O.U. 68/2007 regarding environmental responsibility.
- b) Informs the county agency for environmental protection and the county commissioner of the National Environmental Guard, about the measures taken, respecting the legal requirements of the O.U. 68/2007 regarding environmental responsibility.
- c) Communicates the ecological accident product / potential to the service of environmental protection / headquarters.
- d) Check the removal of the non-conformity, which caused the accident.

## VII. WASTE MANAGEMENT

Waste management is held separately for each category, and collection, storage, storage and recovery / surrender take place as follows:

a) Example: Paper, plastic, metal, hazardous packaging (paint containers, diluent containers, used TEG) are selectively withheld at the probe groups and at the Drying Stations, then collected at the Section Headquarters, stored separately, and then delivered to the supplier of services.

Example of generation, collection, storage and recovery of paper and cardboard waste, code 20 01 01:

Current Number	Month	Category of waste:			
		Generate	From which:		
			Harnessed	Finally disposed of	Remaining in stock
1.	January	0.5	0	0	16.5
2.	February	23.3	39.8	0	0
3.	March	0.5	0	0	0.5
4.	April	0.5	0	0	1
5.	May	0.5	0	0	1.5
6.	June	0.5	0	0	2
7.	July	0.5	0	0	2.5
8.	August	0.5	0	0	3
9.	September	0.5	0	0	3.5
10	October	0.5	0	0	4
11	November	1	0	0	5
12	December	1	6	0	0
	The total	45.8	45.8	0	0

Table 1: generation, collection, storage and recovery of paper and cardboard waste

The economic agents with which our company has contracts carry out the operation of collecting, transporting and processing waste.

b) The reservoir water is measured by the gas extraction operator at each discharge with the help of the calibration beam and noted on a standardized form after which it is directed to the Reservoir Group and then transported by self-survey to the 4 Storage Basins Injection Station wastewater, after which it is injected with the help of injection pumps in the injection wells belonging to the section, keeping also the water management transported from groups and injected on standardized forms.

c) At the Waste Deposit Specific to the extraction activity there are:

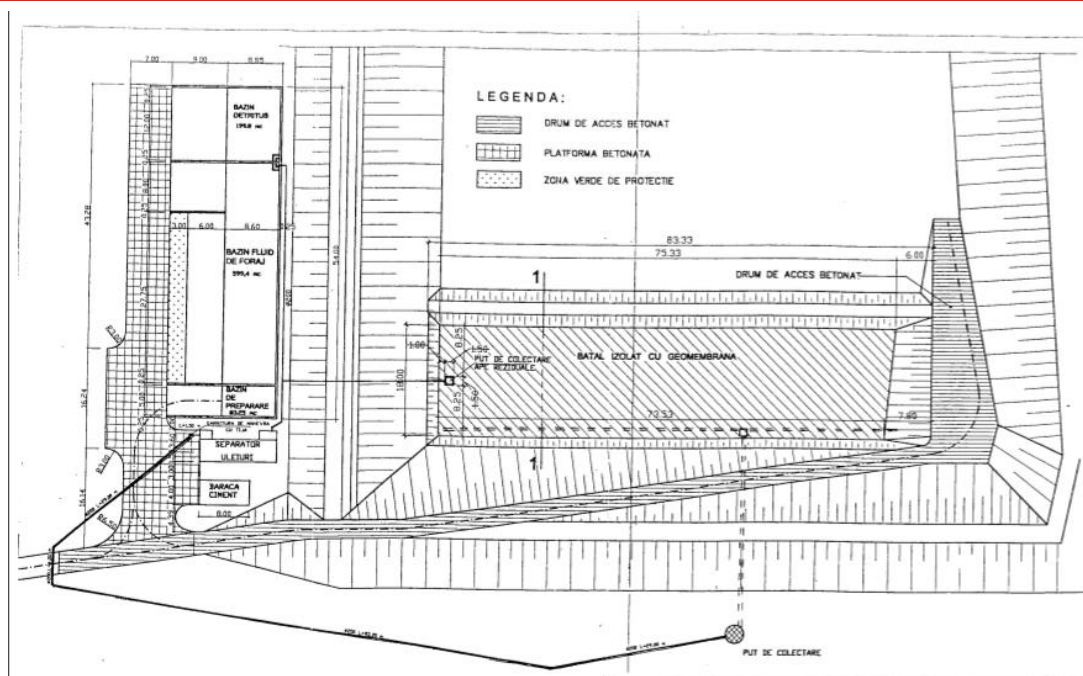


Figure 2: Specific waste deposit

A reservoir for drilling fluids depreciated from wells in drilling, intervention, RK, special fluids, and muddy water; A debris storage tank; An isolated basin with an area of about 1 ha for storage of petroleum products; A separator basin with liquid impurities with pumps, where the water resulting from the separation of the first three basins is pumped, the water that is transported to the storage basin injection station, which is then injected into the wells; A sludge storage tank, debris, sediment, processed using cement.

### CONCLUSIONS

Keeping under control of equipment, materials, waste is continuously monitored, and training on environmental protection is performed monthly.

Also, periodic internal and external inspections are carried out where all the environmental aspects are verified, the gas production process, the obligatory documents to be present at the Headquarters of the Section, authorizations, contracts, Safety Data Sheets, reports, waste management situations, records, collection, storing, and handing over or using them, environmental monitoring, the plan of interventions in case of accidental pollution, checking the on-line training for environmental protection. Having the right recipe, and the constant attention, can lead us to success quickly, helping both the staff and the company to have the results they want in a quick time, being friendly with the environment!

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### REFERENCES

- [1] AVRAM LAZĂR, ELEMENTS OF FORAGE MANAGEMENT, Petroleum-Gas University Publishing House in Ploiești, Ploiești, Romania, 2011;
- [2] C. Ionescu, Gh. Dumitrașcu, D. Ciuparu Pollution and Environmental Protection in Oil and Petrochemistry, Brilliant Publishing House, Buc., 1999;

- [3] Onuțu, I., Stănică – Ezeanu D., Environmental protection, Editure UPG 2003;
- [4] Onuțu, I, Jugănar, T., Pollutants in oil and petrochemicals, Editure UPG 2010.
- [5] Graziosi Dean, Millionaire Success Habits, Growth Publishing, Phoenix, Arizona USA, 2017;
- [6] [www.romgaz.ro](http://www.romgaz.ro);
- [7] Data and analysis applied within a Gas Production Section.

# Comparative Study on the Introduction of Solid Foam Substances in Gas Wells to Remove Water from the Bottom of the Well

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## ABSTRACT

Currently, at more than 90% of the gas wells in Romania, the accumulation of liquids in the depth area is recorded, the average flow per well is about  $5000 \text{ Nm}^3 / \text{day}$ , the working pressures at the wells are between 2.5 - 10 bar, at 70% daily wells are below  $3000 \text{ Nm}^3$ . At these flows and pressures there is accumulation of liquids in the deep area, resulting in the permanent tendency to stop production, and one of the methods by which we manage to train the water from the surface of the well to the surface is the use of solid foam substances.

**Keywords**—solid foam substances, wells, production, comparison, tests, parameters, gas production fields

## I. INTRODUCTION

Considering that the gas fields are in an advanced state of depletion, the working pressures are relatively small, between 2.5-10 bar, most of the wells produce flow rates between 1-5 thousand  $\text{Nm}^3 / \text{day}$ , and the fact that these wells produce accumulations of liquid at the bottom of the well, it is necessary to bring water to the surface to extend the life of the well and thus avoid its self-flooding. One of the methods used is the introduction of foam substances into the gas wells, namely solid subunit or superunit sticks, depending on their efficiency on each individual probe, or the introduction of liquid foam, with the help of lubricants, or newer. automatic devices for inserting foam substances into the well.

For the wells for which the use of superunit solid foam substances is suitable, we present in the following a comparative study on the behavior of the probes when stimulated with two types of superunit solid foam substances, having different compositions.

## II. COMPOSITION AND TECHNICAL CHARACTERISTICS OF ANALYZED SPUMOGENIC SUBSTANCES

The two types of solid superunitary foam substances, which we will refer to below, type A sticks and type B sticks, have the following compositions:

Sticks type A: The composition is a mixture of nonionic and anionic surfactants, namely sulphated and ethylated fatty alcohols in different percentages.

Curr. number	Physico-chemical characteristics	Conditions of admissibility
1	Appearance at 20 ° C	Waxy yellow-white stick
2	Solubility in water	Soluble
3	Active substance content%	97 %
4	Density, g / cm <sup>3</sup>	>1
5	Ph of the solution	6-9.5
6	Diameter, cm	3.2-3.5
7	Length, cm	35
8	Foaming power in water with a content of 200 g / l NaCl	200 cm <sup>3</sup> at 5 minutes

Table 1 Technical characteristics of type A sticks

Type B sticks: The composition is a mixture of nonionic surfactants with varying degrees of ethoxylation.

Curr. number	Physico-chemical characteristics	Conditions of admissibility
1	Appearance at 20 ° C	Solid white
2	Solubility in water	Soluble
3	Active substance content%	97 %
4	Density, g / cm <sup>3</sup>	>1
5	Ph of the solution	6-7
6	Diameter, cm	3.5
7	Length, cm	35
8	Foaming power in water with a content of 200 g / l NaCl	220 cm <sup>3</sup> at 5 minutes

Table 2 Technical characteristics of type B sticks

Since the recipes are kept under control by the manufacturers, the chemical composition of the products is not fully known.

### III. COMPARATIVE ANALYSIS ON THE INTRODUCTION OF THE TWO TYPES OF STICKS

In the following we will present the comparative analysis regarding the use of the two types of sticks on a number of six probes:

Day	Date	Nozzle (Ømm)	Pt (barr)	Pc (barr)	P <sub>1</sub> (bar r)	P <sub>2</sub> (barr)	Flow (m <sup>3</sup> /day)	Impurities (Liters)	Sticks introductions (Pieces)
Well parameters for stimulation with type A sticks									
1	20.01.2020	5.5	4	5.7	4	2.8	1.9	180	1
2	21.01.2020	5.5	4.8	6.2	4.8	3	2.4	100	1
3	22.01.2020	5.5	3.8	7.2	3.8	3	1.6	220	1
4	23.01.2020	5.5	4.1	5.8	4.1	3.2	1.7	160	1
5	24.01.2020	5.5	3.8	5.8	3.8	2.7	1.8	180	1
6	25.01.2020	5.5	4	5.2	4	2.7	2	155	1
7	26.01.2020	5.5	3.9	6.3	3.9	2.8	1.8	150	1
8	27.01.2020	5.5	3.9	5.2	3.9	2.8	1.8	180	1
Total							15	1325	8
Well parameters for stimulation with type B sticks									
1	28.01.2020	5.5	3.9	5	3.9	2.8	1.8	180	1
2	29.01.2020	5.5	4.2	5.3	4.2	2.8	2.1	110	
3	30.01.2020	5.5	3.6	7	3.6	2.7	1.6	220	1
4	31.01.2020	5.5	4.3	5	4.3	2.8	2.1	150	
5	01.02.2020	5.5	3.3	7.6	3.3	2.7	1.3	210	1
6	02.02.2020	5.5	4.2	5.3	4.2	2.8	2.1	110	

7	03.02.2020	5.5	3.1	7.5	3.1	2.8	0.9	185	1
8	04.02.2020	5.5	4.2	5.6	4.2	2.7	2.1	150	
Total							14	1315	4

Table3: Well 1 tracking sheet

From the analysis of the aforementioned parameters, there is a significant equal value of the water discharged from the top of the well, the frequency of introduction of the type B sticks is 1 piece every two days, compared to 1 daily piece type A sticks, but the flow of the well is not constant.

Day	Date	Nozzle (Ømm)	Pt (barr)	Pc (barr)	P <sub>1</sub> (barr)	P <sub>2</sub> (barr)	Flow (m <sup>3</sup> /day)	Impurities (Liters)	Sticks introductions (Pieces)
Well parameters for stimulation with type A sticks									
1	19.01.2020	8	4.1	7.1	3.1	2.4	3.1	280S	2
2	20.01.2020	8	4.4	7.5	3.5	2.4	3.7	130S	2
3	21.01.2020	8	5.4	7.5	4.4	3.7	3.5	270S	2
4	22.01.2020	8	5.6	7.8	4.5	3.5	4.1	310S	2
5	23.01.2020	8	5.4	7.3	4.4	3.4	4	200S	2
6	24.01.2020	8	4.5	7.5	3.5	2.6	3.5	222S	2
7	25.01.2020	8	4.4	7.3	3.5	2.4	3.7	500S	2
8	26.01.2020	8	4.4	7.2	3.4	2.4	3.5	280S	2
Total							29.1	2192	16
Well parameters for stimulation with type B sticks									
1	27.01.2020	8	4.3	7	3.4	2.5	3.4	220S	1
2	28.01.2020	8	4.3	7	3.5	2.4	3.7	270S	1
3	29.01.2020	8	4.4	6.9	3.4	2.3	3.6	290S	
4	30.01.2020	8	2.8	9.2	2.4	2.3	1.1	445S	1
5	31.01.2020	8	4	7.2	3.5	2.3	3.8	150S	1
6	01.02.2020	8	5	7.4	3.7	2.4	4	220S	1
7	02.02.2020	8	4.4	7.3	3.5	2.5	3.6	240S	1
8	03.02.2020	8	4.4	7.1	3.5	2.4	3.7	200S	1
Total							26.9	2035	7

Table4: Well 2 tracking sheet

From the analysis of the aforementioned parameters, there is a very equal value of the water discharged from the bottom of the well, the frequency of insertion of the sticks type B is 7 pieces in 8 days, compared to 2 daily pieces of sticks type A.

Day	Date	Nozzle (Ømm)	Pt (barr)	Pc (barr)	P <sub>1</sub> (barr)	P <sub>2</sub> (barr)	Flow (m <sup>3</sup> /day)	Impurities (Liters)	Sticks introductions (Pieces)
Well parameters for stimulation with type A sticks									
1	19.01.2020	7	3.8	26.7	3.7	2.4	3.1	650S	2
2	20.01.2020	7	3.3	26.9	3.3	2.9	1.8	600S	2
3	21.01.2020	7	3.3	26.6	3.3	2.7	2.2	600S	2
4	22.01.2020	7	3.4	26.7	3.4	2.8	2.2	520S	2
5	23.01.2020	7	3.4	27	3	2.9	0.9	620S	2
6	24.01.2020	7	3.4	26.8	3.2	2.4	2.4	700S	2



7	25.01.2020	7	3.1	26.5	3.1	2.5	2.1	730S	2
8	26.01.2020	7	2.5	20	2.5	2.4	0.8	515S	2
Total							15.50	4935	16
Well parameters for stimulation with type B sticks									
1	27.01.2020	7	3.5	26.7	3.4	2.2	2.8	500S	2
2	28.01.2020	7	3	26.8	3	2.7	1.5	600S	2
3	29.01.2020	7	2.7	24.9	2.7	2.4	1.5	800S	2
4	30.01.2020	7	3.2	26.5	3.2	2.8	1.8	740S	2
5	31.01.2020	7	3.1	26.5	3.1	2.7	1.8	930S	2
6	01.02.2020	7	3	26.8	3	2.2	2.3	900S	2
7	02.02.2020	7	3.6	26.6	3.6	2.8	2.6	940S	2
8	03.02.2020	7	2.7	26.9	2.7	2.4	1.5	730S	2
Total							15.80	6140	16

Table5: Well 3 tracking sheet

From the analysis of the aforementioned parameters, it appears that probe 3 involved more water when stimulated with type B sticks, the frequency of introduction being the same of two pieces of sticks per day, and the flow values being substantially equal.

Day	Date	Nozzle (Ømm)	Pt (barr)	Pc (barr)	P <sub>1</sub> (barr)	P <sub>2</sub> (barr)	Flow (m <sup>3</sup> /day)	Impurities (Liters)	Sticks introductions (Pieces)
Well parameters for stimulation with type A sticks									
1	19.01.2020	8	6.3	8.5	6.3	2.6	6.8	300	1
2	20.01.2020	8	6	8.1	6	2.6	6.6	310	1
3	21.01.2020	8	6	8	6	2.5	6.6	350	1
4	22.01.2020	8	5.8	8.4	5.8	3	6.3	320	1
5	23.01.2020	8	6.1	8.1	6.1	3.1	6.6	300	1
6	24.01.2020	8	6	6.3	5.7	2.9	6.2	320	1
7	25.01.2020	8	6.2	8.5	6.2	3.1	6.7	320	1
8	26.01.2020	8	6.2	8.3	6.2	2.5	6.7	300	1
Total							52.50	2520	8
Well parameters for stimulation with type B sticks									
1	27.01.2020	8	5.7	8.3	5.7	2.5	6.3	320	1
2	28.01.2020	8	5.7	8.3	5.7	2.5	6.3	400	1
3	29.01.2020	8	5.9	8.1	5.9	2.5	6.5	250	
4	30.01.2020	8	6	8.1	6	2.5	6.6	300	1
5	31.01.2020	8	5.8	8.1	5.8	2.5	6.4	250	
6	01.02.2020	8	5.8	8	5.8	2.5	6.4	150	1
7	02.02.2020	8	5.8	8.1	5.8	2.5	6.4	340	
8	03.02.2020	8	5.4	8.5	5.4	2.5	6	300	1
Total							50.90	2310	5

Table6: Well 4 tracking sheet

From the analysis of the aforementioned parameters, results are obtained of equal values of the water discharged from the bottom of the well, the frequency of insertion of the sticks type B is 1 piece every two days, compared to 1 daily piece of sticks type A, the flow values being substantially equal.

Day	Date	Nozzle (Ømm)	Pt (barr)	Pc (barr)	P <sub>1</sub> (barr)	P <sub>2</sub> (barr)	Flow (m <sup>3</sup> /day)	Impurities (Liters)	Sticks introductions (Pieces)
Well parameters for stimulation with type A sticks									
1	17.01.2020	5	5.8	6	5.7	5.4	1.06	5	2
2	18.01.2020	5	6	6.2	5.9	5.4	1.36	350	
3	20.01.2020	5	5.7	6	5.7	5.3	1.21	5	2
4	21.01.2020	5	6.1	6.4	6	5.5	1.37	300	
5	23.01.2020	5	5.8	6	5.8	5.5	1.07	5	2
6	24.01.2020	5	6.2	6.4	6.1	5.6	1.38	300	
7	26.01.2020	5	5.8	6	5.7	5.4	1.06	5	2
8	27.01.2020	5	6.1	6.3	6	5.6	1.24	400	
9	29.01.2020	5	5.9	6.1	5.9	5.6	1.07	0	2
10	30.01.2020	5	6.3	6.6	6.1	5.6	1.38	350	
11	01.02.2020	5	6	6.2	5.9	5.7	0.89	5	2
12	02.02.2020	5	6.1	6.4	5.9	5.5	1.23	300	
13	04.02.2020	5	6	6.1	5.9	5.7	0.89	10	2
14	05.02.2020	5	6.2	6.4	6.1	5.7	1.25	350	
15	10.02.2020	5	6.1	6.3	6.1	5.8	1.09	5	2
16	11.02.2020	5	6.4	6.8	6.2	5.8	1.26	250	
17	13.02.2020	5	5.7	6	5.7	5.4	1.06	10	2
18	14.02.2020	5	6.3	6.7	6.1	5.7	1.25	400	
19	16.02.2020	5	5.6	5.7	5.6	5.5	0.62	0	2
20	17.02.2020	5	6.2	6.6	6	5.5	1.37	400	
21	19.02.2020	5	5.7	5.9	5.7	5.4	1.06	10	2
22	20.02.2020	5	6.2	6.6	6.1	5.7	1.25	300	
23	22.02.2020	5	6.2	6.3	6.1	5.8	1.09	5	2
24	23.02.2020	5	6.4	6.8	6.2	5.8	1.26	350	
<b>Total</b>							<b>27.77</b>	<b>4115</b>	<b>24</b>
Well parameters for stimulation with type B sticks									
1	25.02.2020	5	5.3	5.5	5.2	4.8	1.16	5	2
2	26.02.2020	5	5.9	6.8	5.8	5.3	1.35	500	
3	27.02.2020	5	6	6.2	5.8	5.3	1.35	20	2
4	28.02.2020	5	5.7	6	5.6	5.2	1.2	100	
5	29.02.2020	5	5.9	6	5.9	5.3	1.48	50	
6	01.03.2020	5	5.8	6	5.7	5.3	1.21	100	
7	02.03.2020	5	5.8	6	5.6	5.3	1.05	30	2
8	03.03.2020	5	5.8	6.4	5.8	5.2	1.47	450	
9	04.03.2020	5	6	6.2	5.8	5.3	1.35	150	
10	05.03.2020	5	5.7	6	5.6	5.2	1.2	10	2
11	06.03.2020	5	6	6.7	5.8	5.3	1.35	500	
12	07.03.2020	5	5.8	6	5.8	5.3	1.35	50	

13	08.03.2020	5	5.7	6.2	5.5	5.2	1.04	50	2
14	09.03.2020	5	6.2	6.5	5.6	5.2	1.2	450	
15	10.03.2020	5	5.8	6	5.7	5.3	1.21	150	
16	11.03.2020	5	5.8	6	5.7	5.2	1.34	0	
17	12.03.2020	5	6	6.2	5.7	5.5	0.87	10	2
18	13.03.2020	5	6.4	7.1	5.9	5.5	1.23	400	
19	14.03.2020	5	6.1	6.3	5.9	5.4	1.36	200	
20	15.03.2020	5	6.2	6.4	5.9	5.5	1.23	100	2
21	16.03.2020	5	6.2	6.7	6	5.4	1.49	450	
22	17.03.2020	5	6	6.2	5.9	5.4	1.36	100	
23	18.03.2020	5	6	6.2	5.9	5.4	1.36	20	2
24	19.03.2020	5	5.8	6	5.7	5	1.56	100	
<b>Total</b>							<b>30.77</b>	<b>3995</b>	<b>16</b>

Table7: Well 5 tracking sheet

From the analysis of the aforementioned parameters, there is a very equal value of the water discharged from the bottom of the well, the frequency of insertion of the sticks type B is 2 pieces every three / four days, compared to 2 pieces every two days of type A sticks, and the volume of extracted gas is higher due to the use of type B sticks, with about 3 thousand Nm<sup>3</sup> / 24-day period, the flow can be influenced also by the fact that the suction pressure is lower.

Day	Date	Nozzle (Ømm)	Pt (barr)	Pc (barr)	P <sub>1</sub> (barr)	P <sub>2</sub> (barr)	Flow (m <sup>3</sup> /day)	Impurities (Liters)	Sticks introduction s (Pieces)
Well parameters for stimulation with type A sticks									
1	22.02.2020	12	6.1	15.4	5.7	5.4	6.24	5	2
2	23.01.2020	12	7.4	9.1	6.3	5.5	10.22	500	
3	24.01.2020	12	6.6	10.8	6.1	5.6	8.17	10	2
4	25.01.2020	12	7.1	8.8	6.4	5.6	10.3	600	
5	26.01.2020	12	5.9	16	5.6	5.4	5.1	10	2
6	27.01.2020	12	7.2	9.4	6.3	5.6	9.64	600	
7	28.01.2020	12	6.3	17	6	5.6	7.31	10	2
8	29.02.2020	12	7.7	9.5	6.4	5.6	10.3	500	
9	30.01.2020	12	5.8	18.2	5.6	5.3	6.19	5	2
10	31.01.2020	12	8	10.3	6.3	5.5	10.22	600	
11	01.02.2020	12	6.7	10.5	6.2	5.7	8.23	20	2
12	02.02.2020	12	7.3	9	6.2	5.5	9.57	700	
13	03.02.2020	12	6.8	11.5	6.3	5.7	9.01	10	2
14	04.03.2020	12	6.8	11.5	6.3	5.7	9.01	500	
15	05.02.2020	12	6.6	11.4	6.1	5.7	7.37	20	2
16	06.02.2020	12	8.1	10.1	6.4	5.7	9.72	600	
17	07.02.2020	12	6.6	12	6.1	5.7	7.37	10	2
18	08.02.2020	12	8.8	10.4	6.4	5.7	9.72	500	
19	09.02.2020	12	5.9	17.3	5.8	5.6	5.18	15	2
20	10.02.2020	12	8.6	10.2	6.6	5.8	10.46	700	
21	11.02.2020	12	7.6	9.8	6.4	5.8	9.08	20	2

22	12.02.2020	12	8.6	10.4	6.5	5.8	9.79	500	
23	15.02.2020	12	7	11	6.5	5.7	10.38	10	2
24	16.02.2020	12	6.5	8.2	6.2	5.5	9.57	850	
Total							208.15	7295	26
Well parameters for stimulation with type B sticks									
1	25.02.2020	12	5.4	17	5.2	4.8	6.84	10	2
2	26.02.2020	12	8.6	11.3	6	5.3	9.42	800	
3	27.02.2020	12	6.5	8.4	6.1	5.3	10.06	30	2
4	28.02.2020	12	9	11.3	5.9	5.2	9.34	800	
5	29.02.2020	12	7	8	6.2	5.3	10.65	100	2
6	01.03.2020	12	8.7	10.8	6	5.3	9.42	800	
7	02.03.2020	12	6.8	9.5	5.9	5.3	8.73	10	2
8	03.03.2020	12	9	12	5.9	5.2	9.34	700	
9	04.03.2020	12	6.7	7.9	6.1	5.3	10.06	150	
10	05.03.2020	12	6.7	12.5	5.7	5.2	7.91	20	2
11	06.03.2020	12	8.8	11	5.9	5.3	8.73	1000	
12	07.03.2020	12	6.5	8.5	6.4	5.4	11.31	150	
13	08.02.2020	12	6.2	23.1	5.7	5.2	7.91	100	2
14	09.03.2020	12	8.8	11.5	5.8	5.2	8.65	1000	
15	10.03.2020	12	6.5	8.2	6.1	5.3	10.06	150	
16	11.03.2020	12	6.2	12.4	5.7	5.2	7.91	20	2
17	12.03.2020	12	9	11.7	6	5.5	8.1	800	
18	13.03.2020	12	6.7	8.3	6.3	5.5	10.22	200	
19	14.03.2020	12	6.3	13.3	5.8	5.4	7.2	10	2
20	15.03.2020	12	9	10.3	6.1	5.5	8.87	950	
21	16.03.2020	12	6.8	8.1	6.2	5.4	10.14	100	
22	17.03.2020	12	6.4	13.5	5.9	5.4	8.04	100	2
23	18.03.2020	12	9.1	12.6	6.2	5.5	9.57	750	
24	19.03.2020	12	6.4	8.3	5.8	5	9.81	100	
Total							218.29	8850	18

Table8: Well 6 tracking sheet

From the analysis of the aforementioned parameters, higher values of 8850 liters of water are generated (following the stimulation with B-type sticks compared to 7295 following the stimulation with A-type sticks, the frequency of insertion of the B type sticks is stabilized at 2 pieces at 3 days, and the frequency of introducing A type sticks is 2 pieces at two days, and the volume of extracted gas is higher after using B type sticks, with about 10 thousandNm<sup>3</sup> / 24 days period .

### CONCLUSIONS

The above study was conducted on a number of six wells, of which four wells are freshwater and two wells are saltwater. The frequency of insertion of the superunitary sticks type B is lower than the frequency of insertion of the superunitary sticks type A, in all the wells, the quantities of water drawn are relatively similar to an increase of the water trained on the wells 3 and 6 after the use. B-type superunit sticks, also the quantities of gas produced are relatively similar to a small plus on probe 6 after using B-type superunit sticks. We make the specification that the values of the flow rates of the probes may vary depending on the collection pressure, which differs from one day to another.

Considering the technical aspects we consider the possibility of using the type B superunitarian sticks optimally, of course the economic aspect must be taken into account when making a decision.

#### **Acknowledgment**

I wish to bring my thanks to my teachers from the University Of Lucian Blaga Din Sibiu, the university professor dr. ing. Avram Lazar. We work from the University Of Petrol Gas From Ploiesti, and from the company ROMGAZ S.A.

#### **REFERENCES**

- [1] Data and analysis applied within a Gas Production Section.

# Assessment of Extracts from dry Fruits Blackberry (*Vaccinium Myrtillus* L.) by Regression Analysis for of Amount Anthocyanins

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## ABSTRACT

The aim of the study is to develop a technology for obtaining extracts of dried fruits of blackblueberries. The basic extraction parameters have been established. The influence of the technological parameters of the extraction process on the content of the amount anthocyanins in the extracts is analyzed. Mathematical data processing was performed by one-dimensional regression analysis.

Estimates were made on the degree of influence of the factors as well as on their level of significance. Fischer's criterion is assessed, as well as its probability. Residue assessment and analysis was performed by normal probability plot of residues, the scatter plot of the predicted residual values and the residual histogram. The resulting extracts determine the amount of anthocyanins for the purpose of enriching fruit juices with biologically active substances.

The effect of the extractant type, the duration and temperature of the extraction and the hydromodule on the color parameters were investigated.

The results of the planned experiment are statistically processed with the Statistica program. Residue assessment and analysis was performed by normal probability plot of residues, the scatter plot of the predicted residual values and the residual histogram.

The results obtained suggest that 70% ethyl alcohol, temperature 65-80°C, duration 3-4 h and 1:30 hydromodule are technologically reasonable choices for obtaining extracts with a maximum content of amount of anthocyanins. Adequate mathematical models were described describing the dependencies of the individual parameters in the extraction of the common phenols.

Technology for obtaining extracts with maximum content of amount of anthocyanins has been developed.

**Keywords:** *extracts, dry fruits black blueberry, amount of anthocyanins, regression analysis.*

## I. Introduction

Anthocyanin-rich plants are blueberries, blueberries, blackberries, black currants, chokeberry, hibiscus, etc. [1].

Anthocyanins (polyphenolic compounds) biologically active substances contained in blueberries have P-vitamin activity and increase the strength of the vascular wall and capillaries, preventing their



fragility and permeability. Anthocyanins are powerful antioxidants - they bind free oxygen radicals and prevent damage to cell membranes. They have a positive effect on the health of the visual organ [2].

A number of studies have proven the indisputable benefits of consuming such plant foods, especially in reducing the risk of cancer. A separate study of anthocyanins in laboratory conditions showed their undeniable positive effect on the human body, its strengthening and restoration. [3]

Blueberries extract in numerous studies has shown a positive effect on microcirculation. Its ability to reduce vascular permeability, improve the tone of the vascular wall, stimulate capillary blood flow, eliminate microstasis of the venous canal - widely used in the complex therapy of venous and lymphatic insufficiency and other vascular disorders. It normalizes metabolic processes, antioxidant, angioprotector. Blueberries are mainly an effective tool in complex therapy and for the prevention of diseases of the visual organs. [4]

## II. Material and Methods

**Material.** The object of the study is dried fruits of blackberries. In wild plants, there are a number of biologically active substances that can affect the life processes of the human body.

Forest fruits are rich in phenolic compounds and have great antioxidant activity. This makes them a potential raw material for producing extracts that can be used to develop functional beverages.

Various variants of water and ethanol extracts from dried berries and black currant have been developed. The aqueous and ethanol extracts of the fruits are respectively 1:10, 1:20 and 1:30 fruit / extractanthydromodul; at an extraction temperature of 35-80°C and 1, 2, 3 and 4 hours.

The physicochemical analyzes were conducted using standardized methods approved by good manufacturing practice.

Anthocyanins - spectrophotometric such as cyanidine-3,5-diglucoside, by pharmacopoeial method [Pharmacopoeia Russia]. General method of analysis [5].

For each of the test quantities the mean values of three independent experiments are presented.

**Mathematical Methods.** Mathematical data processing was performed by one-dimensional regression analysis. By which were studied and evaluated the possible functional dependencies between two random variables. The main questions are whether there is a functional dependence between two dependent random variables and if so - to find a function that describes it sufficiently accurately. Various models have been studied, with the best-described dependencies being selected. Estimates were made on the degree of influence of the factors as well as on their level of significance. Fischer's criterion is assessed, as well as its probability. Residue assessment and analysis was performed by normal probability plot of residues, the scatter plot of the predicted residual values and the residual histogram. All results are presented analytically and graphically.

The processing was done through the statistical program STATISTICA (StatSoft, Inc.).

All data are processed at level of significance  $\alpha=0,05$ .

## III. Results and discussion

The experimental results obtained were used to obtain a regression model of the effect of the concentration of ethyl alcohol on the anthocyanin content in mg%, and to examine its suitability. After studying polynomials of the first and second degree, the model of the type is the best:

$$y = b_0 + b_1x + b_2x^3 \quad (1)$$

where  $x$  is the concentration of ethyl alcohol in percent and  $y$  is the concentration of Sum of anthocyanins, mg%.

After the statistical processing of the data, it is evident that the coefficient of determination

$R^2 = 0,999$ , which means that 99% of the change in parameter  $Y$  is due to the control factor  $x$  and is described with the model used. Of all the models studied, the coefficient of certainty is the

highest. All the coefficients of the model are statistically significant, since they  $p\text{-level} \ll 0,05$  are as follows:

$$b_0 = 28,66667, b_1 = 1,13786, b_2 = -0,00012$$

Fisher's criterion,  $F(3,8) = 3056,4$   $p < 0,00000$ , as well as its corresponding probability indicate that the model describes a significant part of the  $Y$ . change. The model does better than the so-called naive forecasts average values.

The regression equation is:

$$y = 28,66667 + 1,13786x - 0,00012x^3 \quad (2)$$

The resulting regression model describes the rights  $y = f(x)$ , that we can depict in  $R^2$ .

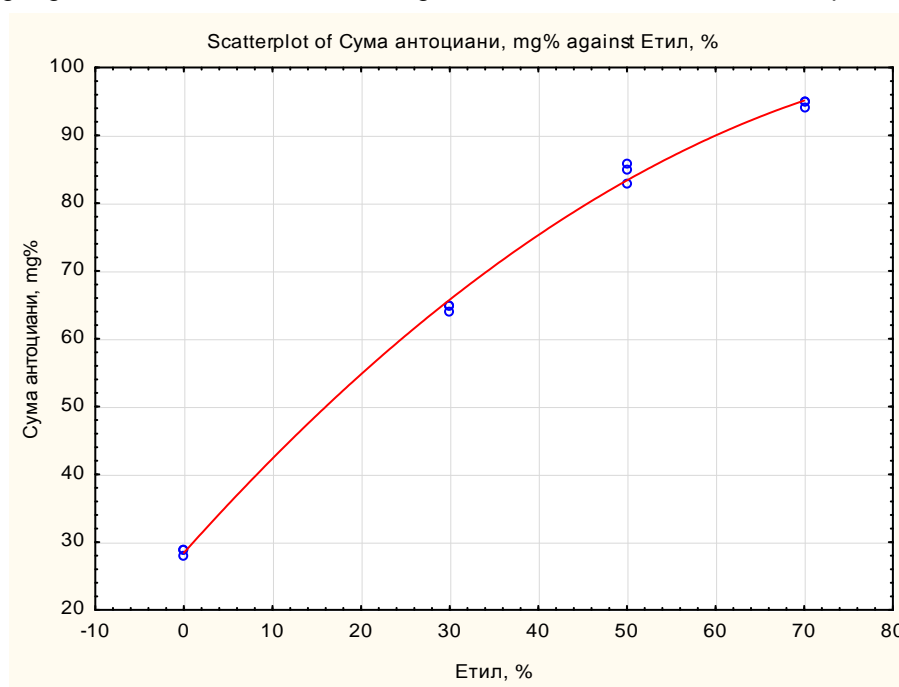


Figure 1. Model response line.

The analysis of residues and their graphical representations are shown in Figure 2 in the so-called normal probability graph.

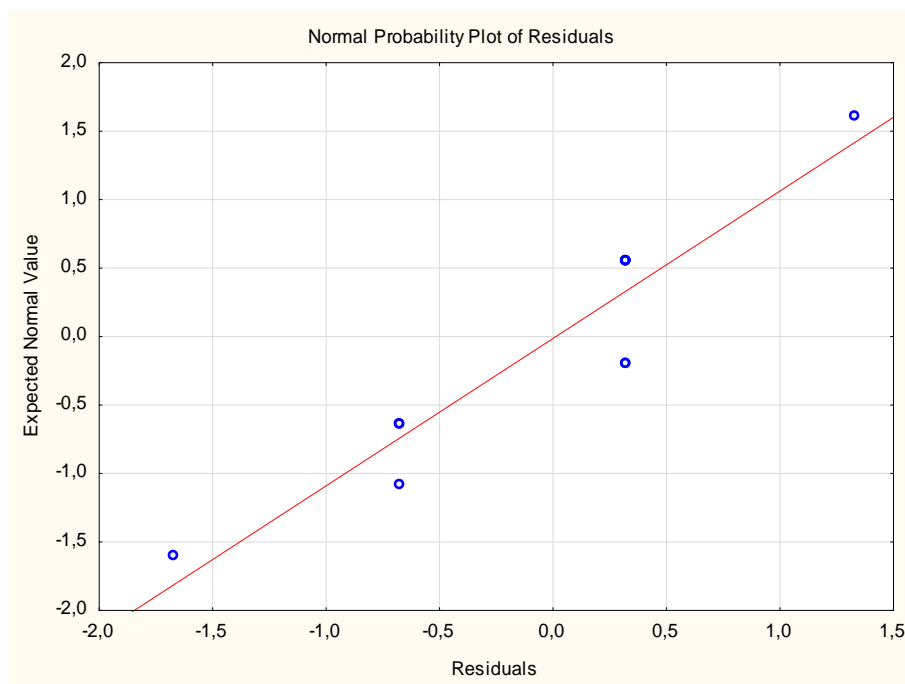


Figure 2. Normal probability plot of residuals.

The analysis shows a lack of systematic deviation of the actual data from the theoretical curve, which indicates a normal distribution of residuals.

We will check for residual dependence on predicted values from the model. For this purpose, we will analyze the scatterplot of the residuals from the predicted values - Figure 3.

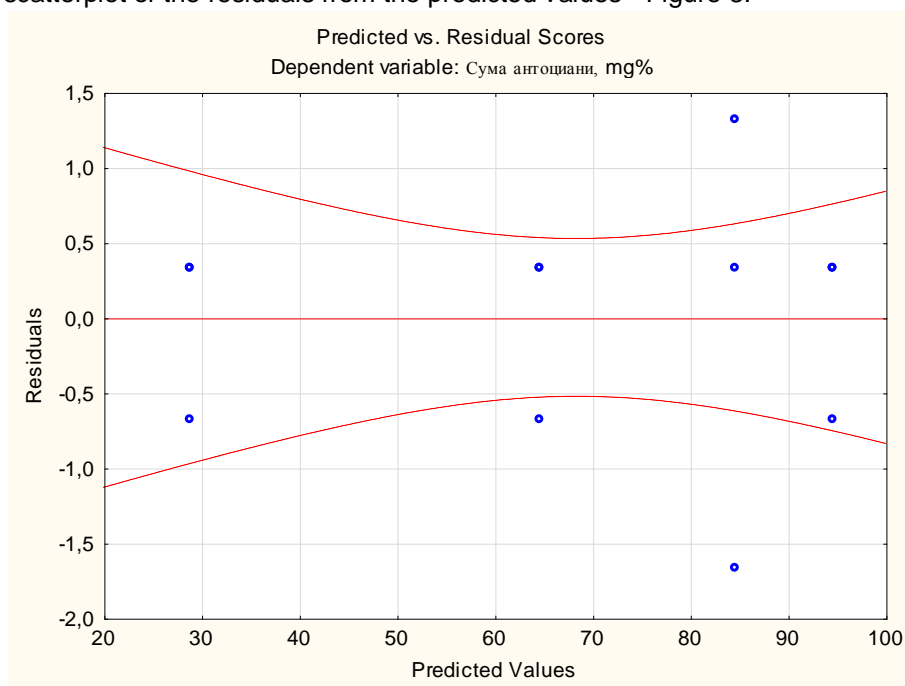


Figure 3. Scatterplot of residual values from predicted values.

The obtained graph shows that the systematic residuals are lacking and are sufficiently chaotic. We can conclude that the residuals do not depend on the predicted values.

**Conclusions.** From the obtained results we can draw the following conclusions:

1. From the analysis of residuals we can conclude that the obtained model is adequate.
2. The resulting model is cubic and describes with great precision the experimental data obtained.

The experimental results obtained were used to obtain a regression model for the effect of the extraction temperature on the anthocyanin content in mg% and to study its suitability. After studying the polynomials of the first and second degrees, a model of the following appears to be the best:

$$y = b_0 + b_1x \quad (3)$$

where  $x$  is the temperature in degrees Celsius and  $y$  is the concentration of Sum of atocyanates, mg%.

After statistical processing of the data, it is evident that the coefficient of determination  $R^2 = 0,93$ , which means that 93% of the change in parameter  $Y$  is due to the control factor  $x$  and is described with the model used. Of all the models studied, the coefficient of certainty is the highest. All the coefficients of the model are statistically significant, since they  $p\text{-level} < 0,05$  are as follows:

$$b_0 = 54,17778, \quad b_1 = 0,39111$$

Fisher's criterion,  $F(1,10) = 121$   $p < 0,000$ , as well as its corresponding probability, show that the model describes a significant part of the change in  $Y$ .

The regression equation is:

$$y = 54,17778 + 0,39111x \quad (4)$$

The resulting regression model describes the rights  $y = f(x)$ , that we can depict in  $R^2$ .

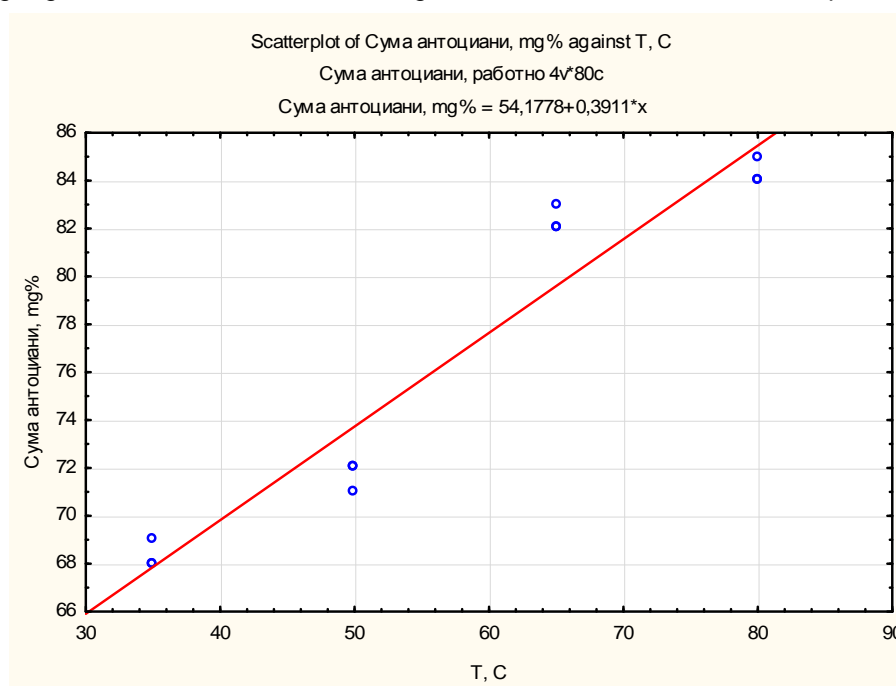


Figure 4. Model response line.

The analysis of residues and their graphical representations are shown in Figure 5 in the so-called normal probability graph.

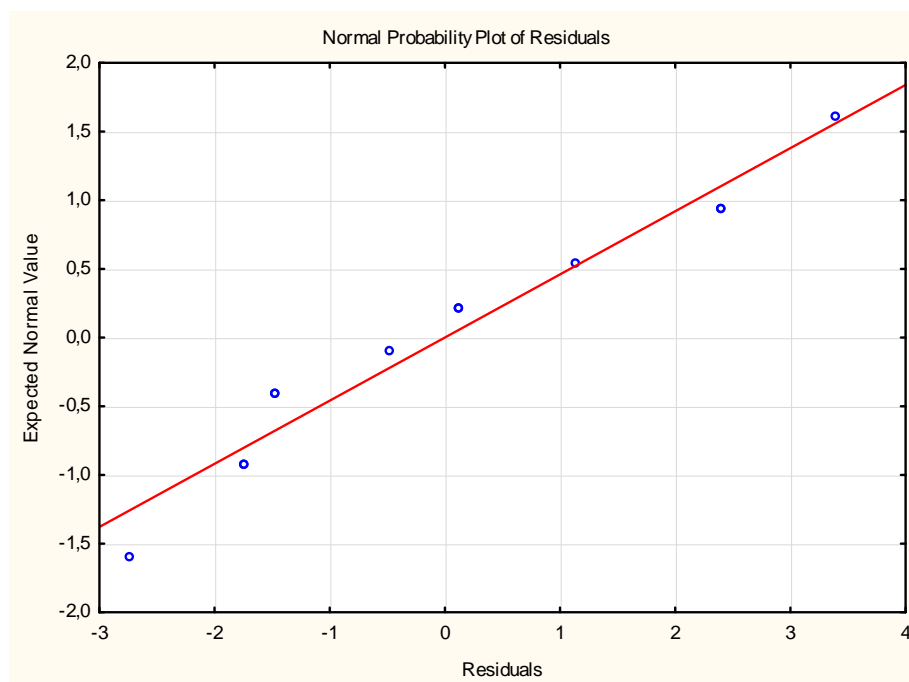


Figure 5. Normal probability plot of residuals.

The analysis shows a lack of systematic deviation of the actual data from the theoretical curve, which indicates a normal distribution of residuals.

We will check for residual dependence on predicted values from the model. For this purpose, we will analyze the scatterplot of the residuals from the predicted values - Figure 6.

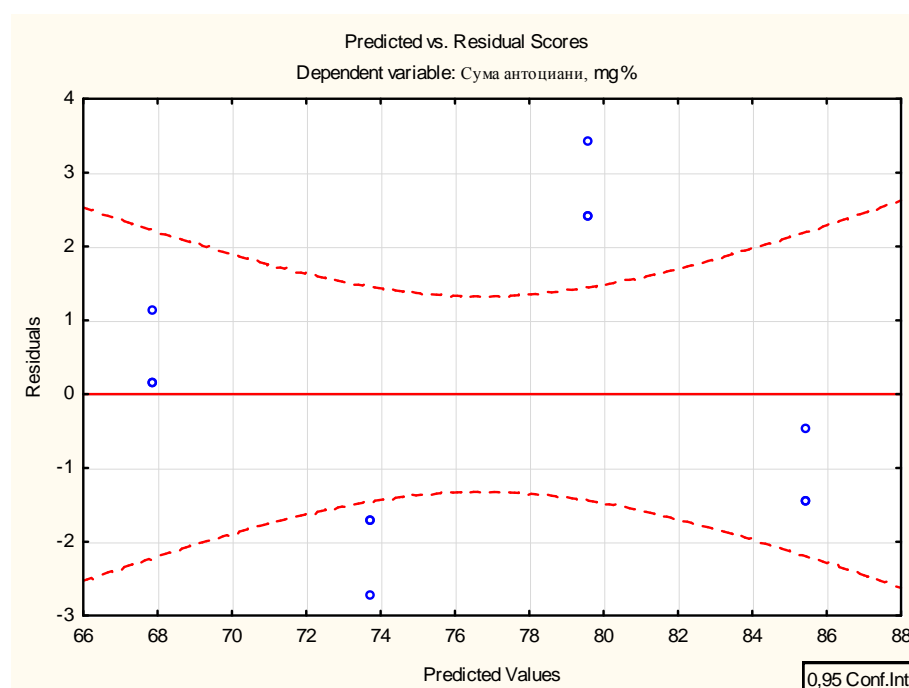


Figure 6. Scatterplot of residual values from predicted values

The obtained graph shows that the systematic residuals are lacking and are sufficiently chaotic. We can conclude that the residuals do not depend on the predicted values.

**Conclusions.** From the obtained results we can draw the following conclusions:

3. From the residue analysis, we can conclude that the model obtained is adequate.

4. The resulting model is linear and describes with great precision the experimental data obtained.

The experimental results obtained were used to obtain a regression model for the effect of extraction duration on the anthocyanin content in mg% as well as to study its suitability. After studying the polynomials of the first and second degrees, a model of the following appears to be the best:

$$y = b_0 + b_1 x^2 \quad (5)$$

where  $x$  is the time in minutes and  $y$  is the concentration of Sum of anthocyanins, mg%.

After the statistical processing of the data, it is evident that the coefficient of determination

$R^2 = 0,99$ , which means that 99% of the change in parameter  $Y$  is due to the control factor  $x$  and is described with the model used. Of all the models studied, the coefficient of certainty is the highest. The coefficient before  $x$  of the model is statistically insignificant.

$$b_0 = 81,58333, \quad b_1 = 0,00035$$

Fisher's criterion,  $F(2,9) = 404,55$   $p < 0,00000$ , and its corresponding probability indicate that the model describes a significant part of the change in  $Y$ . The model performs better than the so-called naive forecasts of average values.

The regression equation is:

$$y = 81,58333 + 0,00035 x^2 \quad (6)$$

The resulting regression model describes the rights  $y = f(x)$ , that we can depict in  $R^2$ .

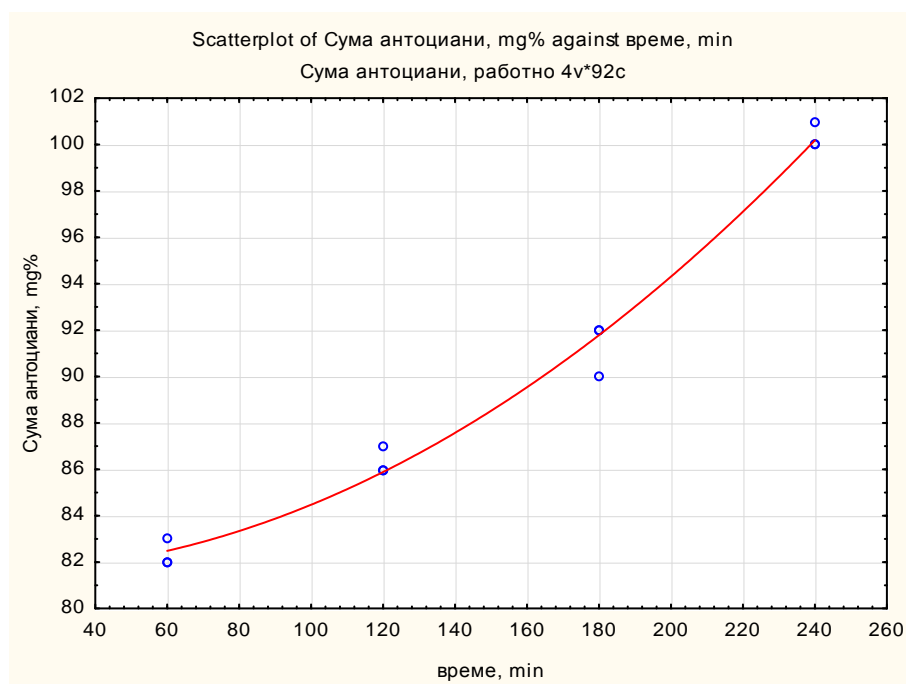


Figure 7. Model response line.

The analysis of the residuals and their graphical representations are shown in Figure 8 in the so-called normal probability graph.



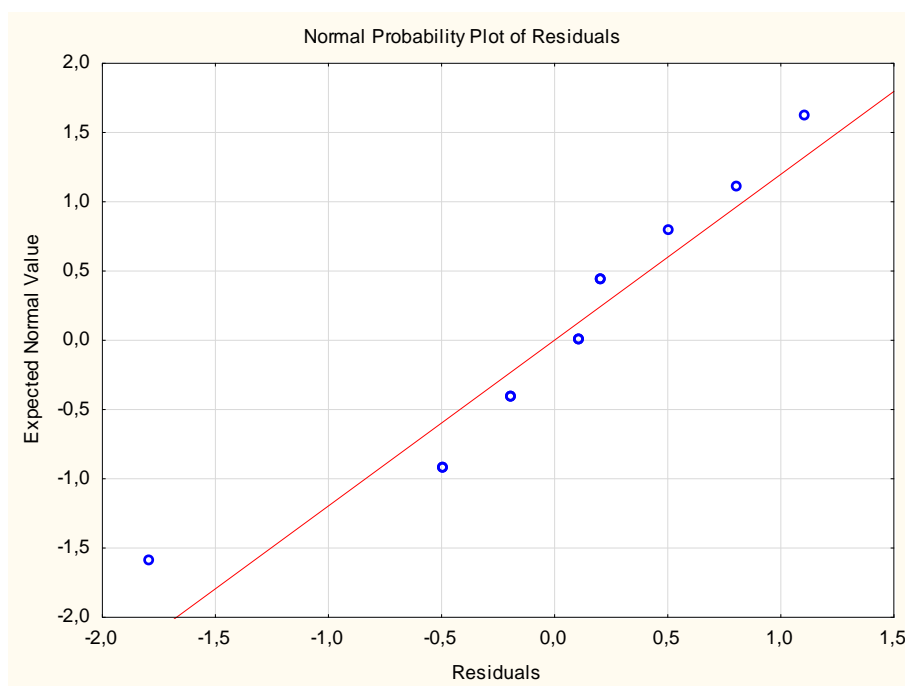


Figure 8. Normal probability plot of residuals.

The analysis shows a lack of systematic deviation of the actual data from the theoretical curve, which indicates a normal distribution of residues.

We will check for residual dependence on predicted values from the model. For this purpose, we will analyze the scatterplot of the residuals from the predicted values - Figure 9.

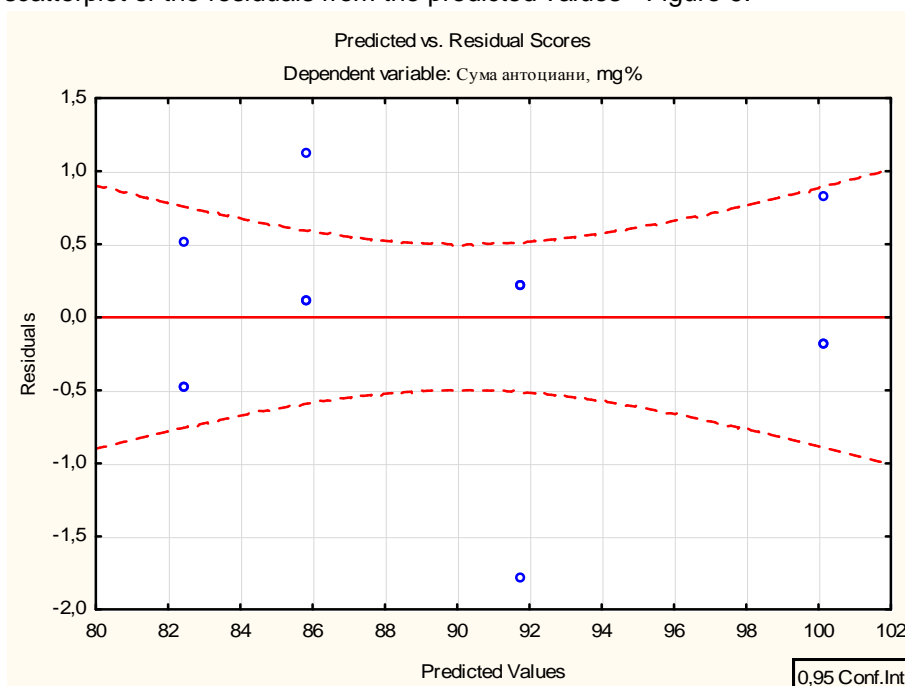


Figure 9. Scatterplot of residual values from predicted values.

The obtained graph shows that the systematic residuals are lacking and are sufficiently chaotic. We can conclude that the residuals do not depend on the predicted values.

**Conclusions.** From the obtained results we can draw the following conclusions:

5. From the analysis of residuals we can conclude that the model obtained is adequate.

6. The resulting model is quadratic and describes with great precision the experimental data obtained. The experimental results obtained were used to obtain a regression model for the effect of the hydromodule on the anthocyanin content in mg% and to study its suitability. After studying the polynomials of the first and second degrees, a model of the following appears to be the best:

$$y = b_0 + b_1x + b_2x^2 \quad (7)$$

where  $x$  is the hydromodule and  $y$  is the concentration of the sum of atocyanates, mg%.

After the statistical processing of the data, it is evident that the coefficient of determination  $R^2 = 0,999$ , which means that 99% of the change in parameter  $Y$  is due to the control factor  $x$  and is described with the model used. Of all the models studied, the coefficient of certainty is the highest. All the coefficients of the model are statistically significant, since they  $p\text{-level} \ll 0,05$  are as follows:

$$b_0 = 5,33333, b_1 = 10,81667, b_2 = -0,23833$$

Fisher's criterion,  $F(2,6) = 3186.3$   $p < 0,0000$ , and its corresponding probability indicate that the model describes a significant part of the change in  $Y$ . The model performs better than the so-called naive forecasts average values.

The regression equation is:

$$y = 5,33333 + 10,81667x - 0,23833x^2 \quad (8)$$

The resulting regression model describes the rights  $y = f(x)$  that we can depict in  $R^2$ .

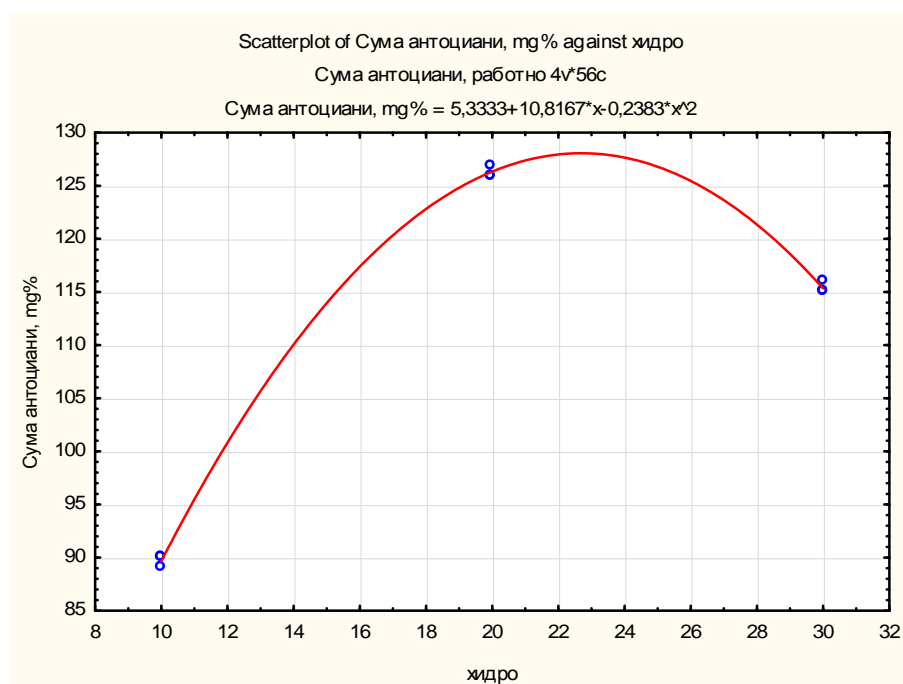


Figure 10. Model response line.

The analysis of residuals and their graphical representations are shown in Figure 11 in the so-called normal probability graph.

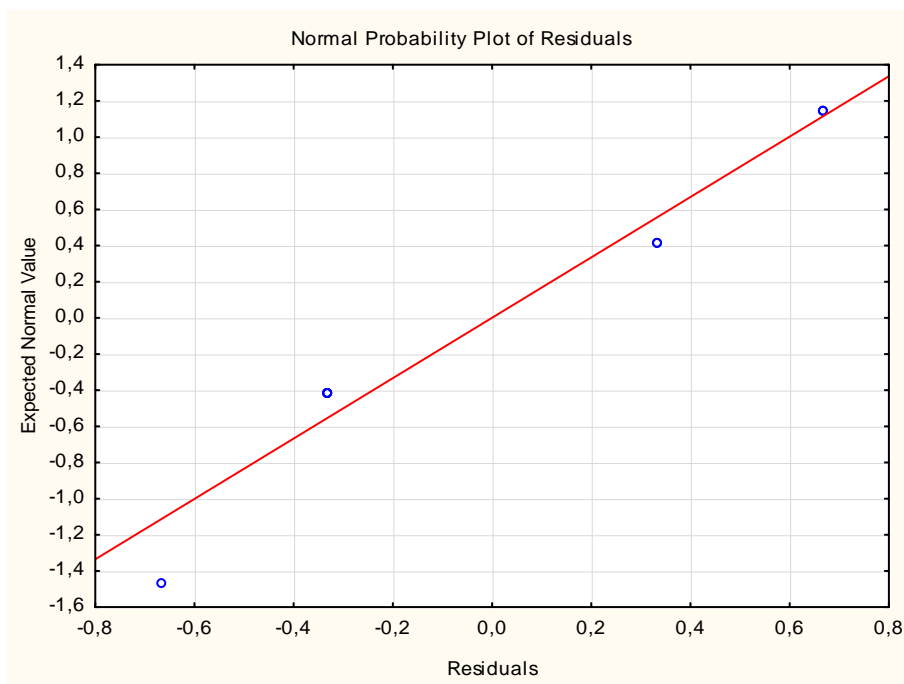


Figure 11. Normal probability plot of residuals.

The analysis shows a lack of systematic deviation of the actual data from the theoretical curve, which indicates a normal distribution of residuals.

We will check for residual dependence on predicted values from the model. For this purpose, we will analyze the scatterplot of the residuals from the predicted values - Figure 12.

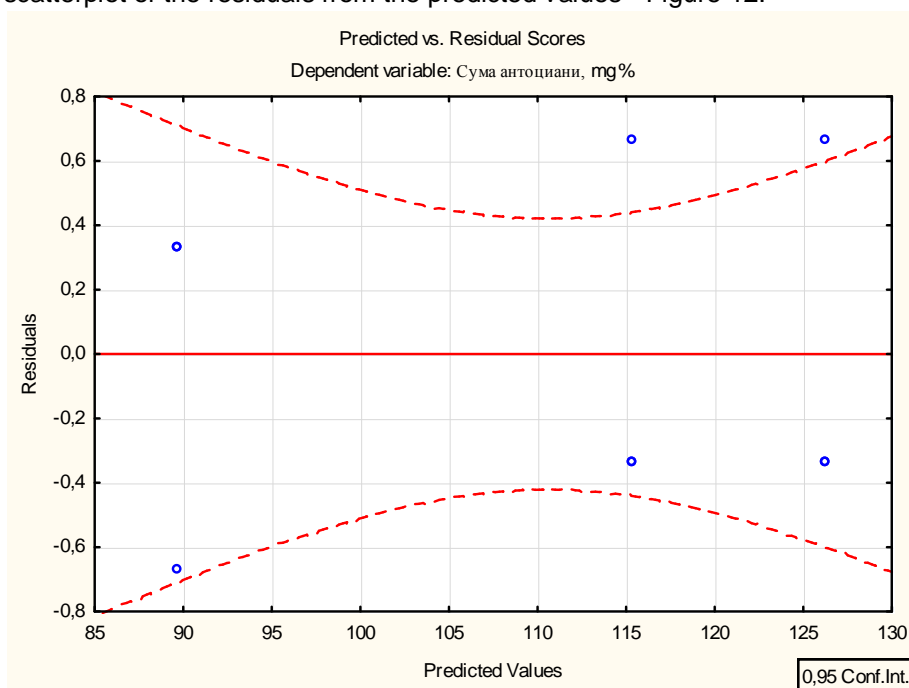


Figure 12. Scatterplot of residual values from predicted values.

The obtained graph shows that the systematic residuals are lacking and are sufficiently chaotic. We can conclude that the residuals do not depend on the predicted values.

### Conclusions

From the obtained results we can draw the following conclusions:

7. From the analysis of residuals we can conclude that the model obtained is adequate.
8. The resulting model is quadratic and describes with great precision the experimental data obtained.

#### References

- [1.] Tanchev, S.S. Anthocyanins in fruits and vegetables / S.S. Tanchev. - Moscow: Food Industry, 1980. - 304 p.
- [2.] A.M. Makarevich, A.G. Shutova, EV Spiridovic et al. / Functions and properties of anthocyanins from plant material / Proceedings of BSU: scientific journal, 2009. - P. 237-245.
- [3.] Artamonov VI Recreational plant physiology / V.I. Artamonov. - Moscow: Agropromizdat, 1991.-231s.
- [4.] V.I. Deineka [et al.] Study of blueberry anthocyanins in fruits and preparations based on it by HPLC // Journal of the Factory Laboratory. Diagnosis of materials. " - 2006. - P. 16-20.
- [5.] StatePharmacopoeia of Russia. General methods of analysis - II ed., Medicine. 1987

# Investigation of the Effect of Extraction Parameters on the Content of the General Phenolic Compounds and the Antiradical Activity of Extracts from dry Fruits Black Blueberry /*Vaccinium Myrtillus L.*/And Black Currant /*Ribes Nigrum L.*/

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## ABSTRACT

The purpose of the study is to develop a technology for the preparation extracts of dried fruits currant and black blueberry . Basic retrieval parameters are established of maximum total phenolic compounds. The influence of the technological parameters of the extraction process on the antiradical activity of the extracts is analyzed.

The results obtained suggest that 70% ethyl alcohol as an extractant, a temperature of 65° C, a duration of 3-4 hours and a 1:30 hydromodule are technologically sound choices for obtaining extracts with maximum total phenolic compounds and maximum antiradical activity.

**Keywords:** *extracts, параметри на екстракция, dry fruits blackcurrant, dry fruits black bluiberrries, total phenolic compounds, antiradical activity.*

## I. INTRODUCTION

Increasing the content of free radicals in the cells creates conditions for the so- oxidative stress in which free radicals oxidize vessel walls, protein molecules, DNA and lipids. These radicals actively interact with membranes of lipids containing unsaturated bonds and alter the properties of cell membranes [1].

In recent years, berries have received much attention because of their health benefits, including antimutagenesis and anticancer activity for the prevention of various cancers and age-related diseases [2].

Fruits of */vacciniummyrtillus L./* and */ ribesnigrum L./* contain powerful antioxidants and a proper balance of bioactive compounds. They are considered to be a good source of phenolic compounds, especially flavonoids and phenolic acids, which mostly contribute to their high antioxidant activity [3].

Beverages are an optimal form of food that can be used to enrich the nutritional portion of irreplaceable nutrients and biologically active substances that have a beneficial effect on metabolism and immune resistance of the body [4].

In order to increase the nutritional value and antioxidant properties of juice-containing beverages, extracts of wild-growing raw materials having a prophylactic and functional effect can be introduced into the production technologies.

The aim of the study is to develop a technology for obtaining extracts of dried fruits of black blueberries and blackcurrants. The basic extraction parameters have been established. The extracts obtained were analyzed for total phenolic compounds content and antiradical activity.

## II. Material and Methods

**A. Material.** The object of the study is the fruits of *Vaccinium myrtillus* L. and *Ribes nigrum*. In wild plants, there are a number of BAV that can affect the life processes of the human body.

Forest fruits are rich in phenolic compounds and have great antioxidant activity. This makes them a potential raw material for producing extracts that can be used to develop functional beverages.

Various variants of water and ethanol extracts of dried fruits currant and black blueberry have been developed for total phenolic compounds and their antiradical activity determined.

The aqueous and ethanol extracts of the fruits are respectively with the hydromodule 1:10, 1:20 and 1:30 fruit / extractant; obtained at an extraction temperature of 35 ° -80 ° C and an extraction time of 1, 2, 3 and 4 hours.

### B. Methods:

- Determination of antiradical activity by DPPH radical - 2,2-diphenyl-1-picrylhydrazyl.
- General Phenol Compounds- spectrophotometric method with Folin-Denis reagent, % as gallic acid [5].

## III. Results and discussion

The effect of phenolic compounds on the antiradical activity of the extracts obtained was investigated. It has been found that with increasing the content of total phenolic compounds, antiradical activity also increases.

The concentration of total phenolic compounds (Fig. 1) is higher when extracted with ethyl alcohol compared to extraction with water. It increases with increasing concentration of ethyl alcohol in the extracts, reaching its maximum in the extract with 70% ethyl alcohol.

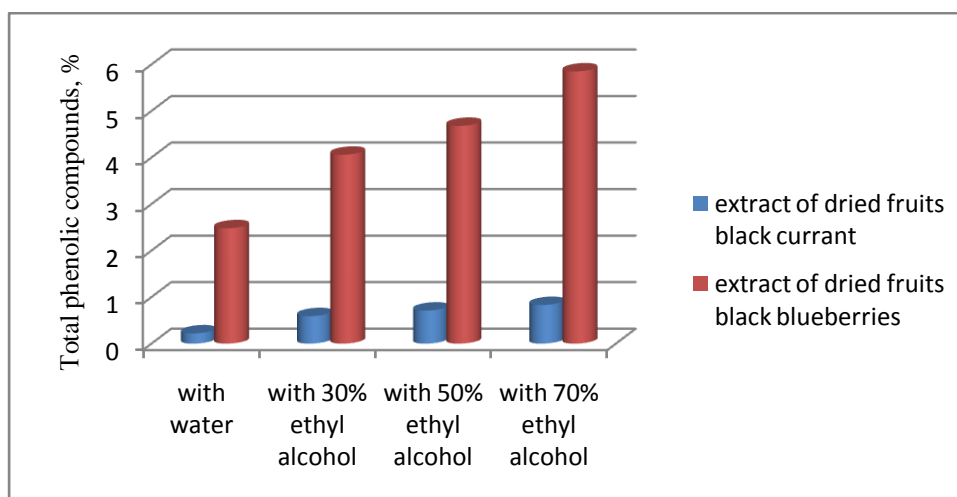


Fig.1. Effect of the extractant concentration on the the total phenolic compounds content in extracts of dried fruits black blueberry and black currant



When extracting of dried fruits blackcurrant with an increase in the concentration of ethyl alcohol from 30 to 50%, the amount of total phenolic compounds increased by 20%, and on extraction with 70% ethyl alcohol the total phenolic compounds increased by 41% - Fig. 1.

When extracting dried fruits blackblueberry with increasing ethyl alcohol concentration from 30 to 50%, the amount of total phenolic compounds increased by 15%, and at extraction with 70% ethyl alcohol the total phenolic compounds increased by 44% - Fig. 1.

The concentration of phenol carboxylic acids upon extraction dried fruits blackblueberry with 30%, 50% and 70% ethanol is higher than the aqueous extracts and is respectively 2.2; 2.3 and 2.4 times higher. For flavonoid phenolic compounds the increase in concentration was 1.8; 2.1 and 2.2 times (fig. 2, 3).

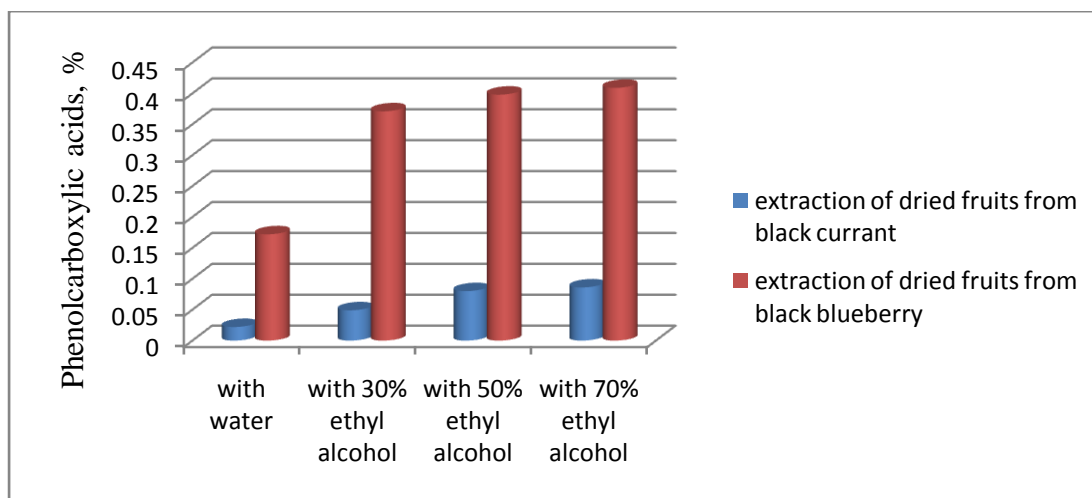


Fig.2. Effect of extractant concentration on phenolcarboxylic acids content in extracts of dried fruits black blueberry and black currant

Concentration of the phenolic acids by extraction dried fruits black currant with 30%, 50% and 70% ethanol is higher, respectively, of 2.2; 3.6 and 3.9 times compared to their concentration in the aqueous extracts. For flavonoid phenolic compounds the increase in the concentration is 2.1; 3.3 and 3.5 times higher (fig. 2, 3).

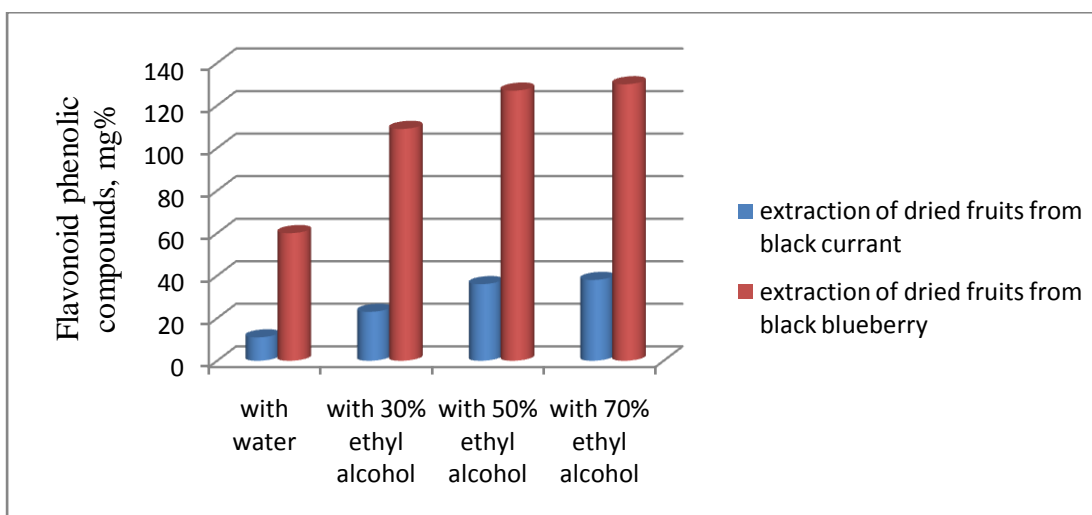


Fig.3. Effect of extractant concentration on flavonoid phenolic compounds content in extracts of dried fruits black blueberry and black currant

The antiradical activity of the extracts against the DPPH radical (Fig. 4) corroborates the literature data for the high anti-radical activity of blackcurrant and black blueberry due to the high content of phenolic compounds. The ethanol extract shows the highest activity against the DPPH radical.

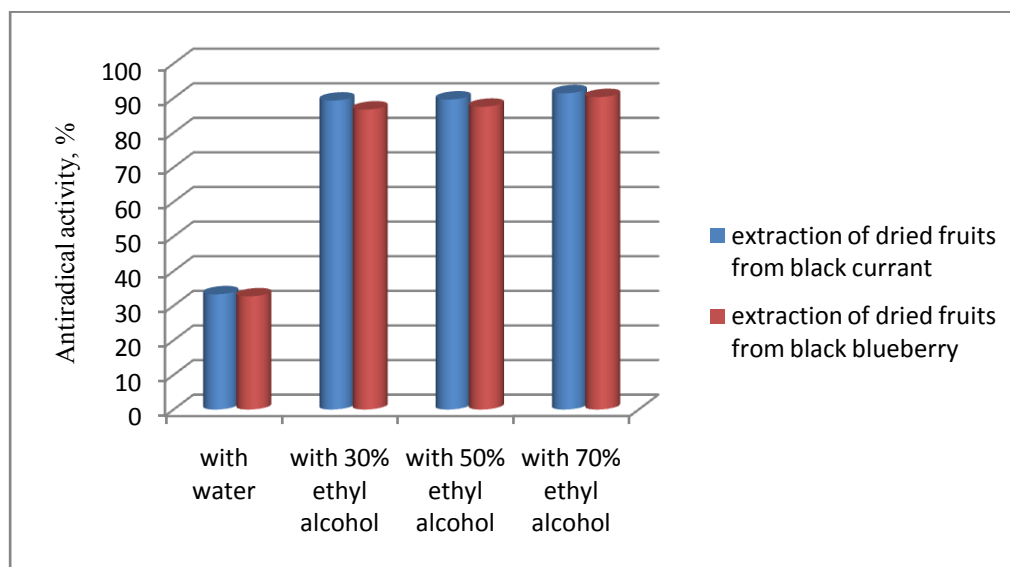


Fig. 2. General antioxidant activity by DPPH method for the effect of type and concentration of the extractant on antiradical activity

Not only phenolic substances and flavonoids but also vitamins C, A and E have antioxidant activity, but when heated, these antioxidants are partially destroyed and the antioxidant activity inherent in the phenolic complex remains.

The blackcurrant and black blueberry may also contain components other than the phenolic compounds, which also exhibit antiradical activity.

The highest DPPH inactivation value was reported in the extract obtained with 70% ethyl alcohol and the lowest in the extract with water - Fig. 4. After storage, the antiradical activity decreased between 54 and 72%, with the highest reduction observed in the extracts obtained with 20 and 90% ethanol. The smallest reduction was observed in the extracts with 70% ethanol.

Figure 3 shows the content of total phenolic compounds of the extracts obtained at a temperature of 35-80°C with 70% ethyl alcohol.

The content of total phenolic compounds in the extracts varies from 3.87% at 50 °C to 4.78% at 65°C for black blueberries and from 0.40% at 50 °C to 0.71% at 65 °C for the currant. The technologically justified extraction temperature is 65°C - Fig. 5.

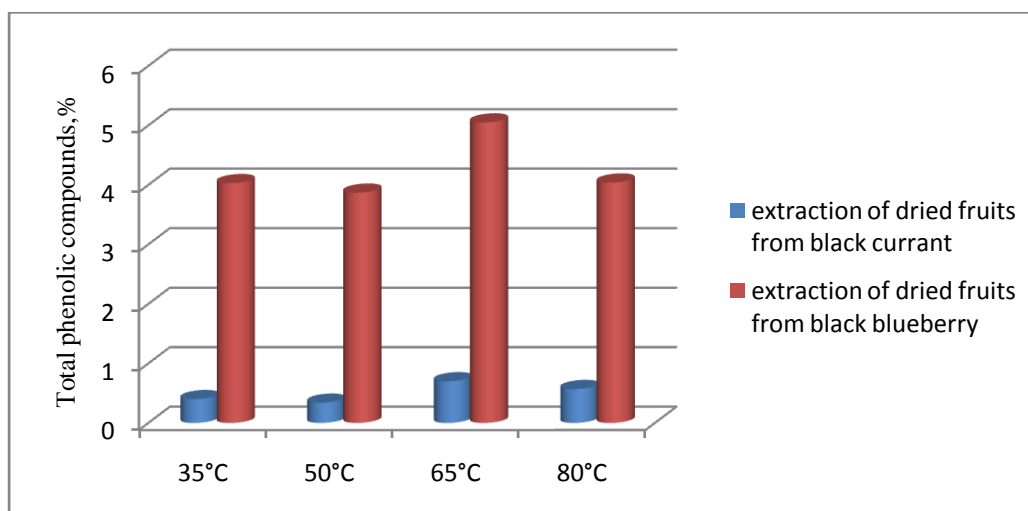


Fig. 5. Influence of the extraction temperature on the total phenolic compounds content in extracts of dried fruits black blueberry and black currant

The trend in the content of phenol carboxylic acids is similar to that of the flavonoid phenolic compounds in extracts of dried fruits black blueberry and black currant. Favorable extraction temperature is 65°- 80°C (fig. 6 and fig. 7).

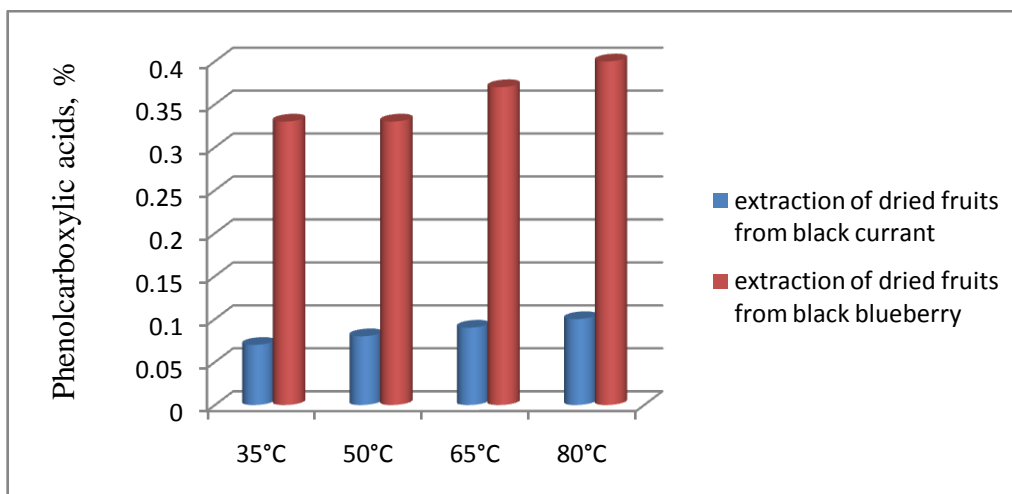


Fig. 6. Influence of the extraction temperature on phenolcarboxylic acids content in extracts of dried fruits blackblueberryand black currant

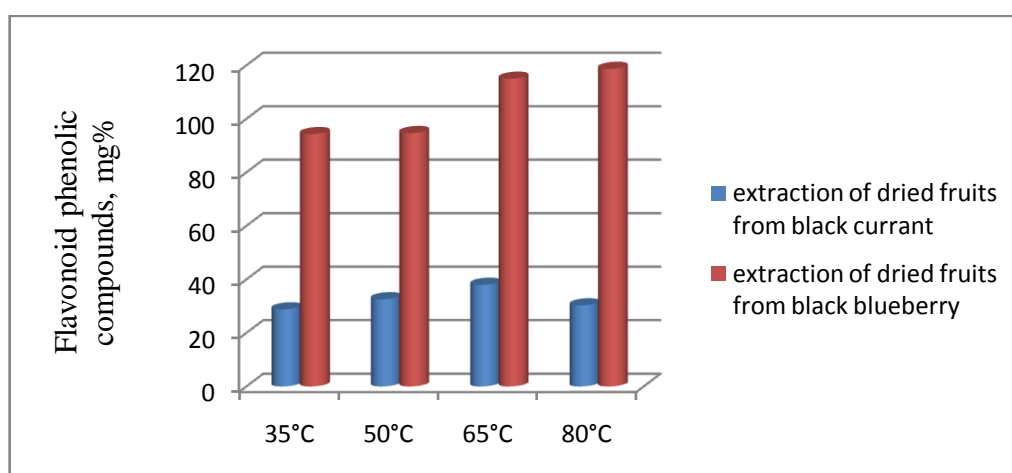


Fig. 7. Influence of the extraction temperature on flavonoid phenolic compounds content in extracts of dried fruits black blueberry and black currant

Figure 8 shows the indicators for the antiradical activity of the extracts obtained at a temperature of 35-80°C with 70% ethyl alcohol.

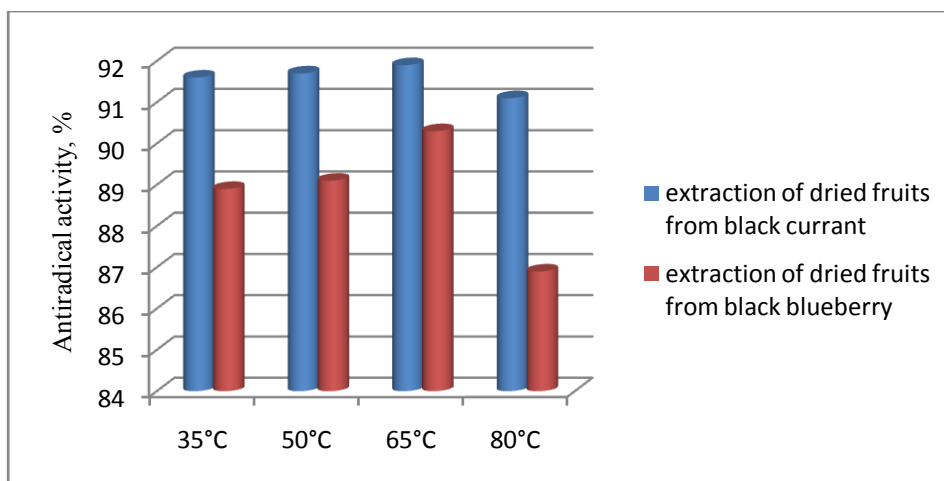


Fig. 8. Total antioxidant activity by DPPH method for the effect of extraction temperature on antiradical activity

Based on the results obtained for the effect of extraction temperature, the following conclusion can be drawn:

- The DPPH free radical capture ability is 65°C.

Figure 8 shows the results for the total phenolic compounds of the extracts obtained at a temperature of 65 ° C with 70% ethyl alcohol at different duration of the extraction process.

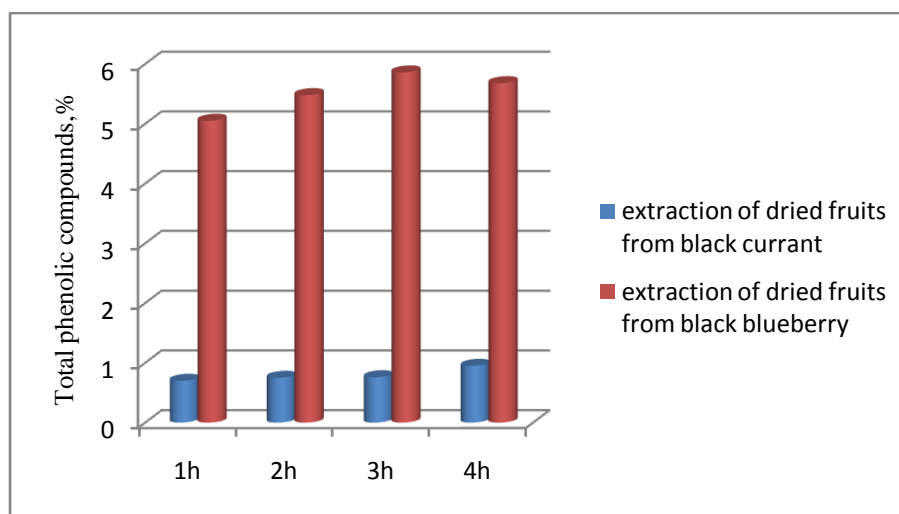


Fig. 9. Influence of the duration of extraction on the content of the total phenolic compounds in extracts of dried fruits black blueberry and black currant

The concentration of total phenolic compounds in black blueberry extracts varies with different time-temperature extraction regimes- fig. 9. With an extraction duration of 1 hour to 3 hours, it increases from 5.22% to 5.86% by 12.3%. By increasing the extraction time to 4 hours, the content of total phenolic compounds decreases by 3.1%

This dependence is different when extracting from the dried fruits of the black currant. The concentration increased steadily from 0.68% for 1 hour to 0.95% for 4 hours by 39.7% - Fig. 9.

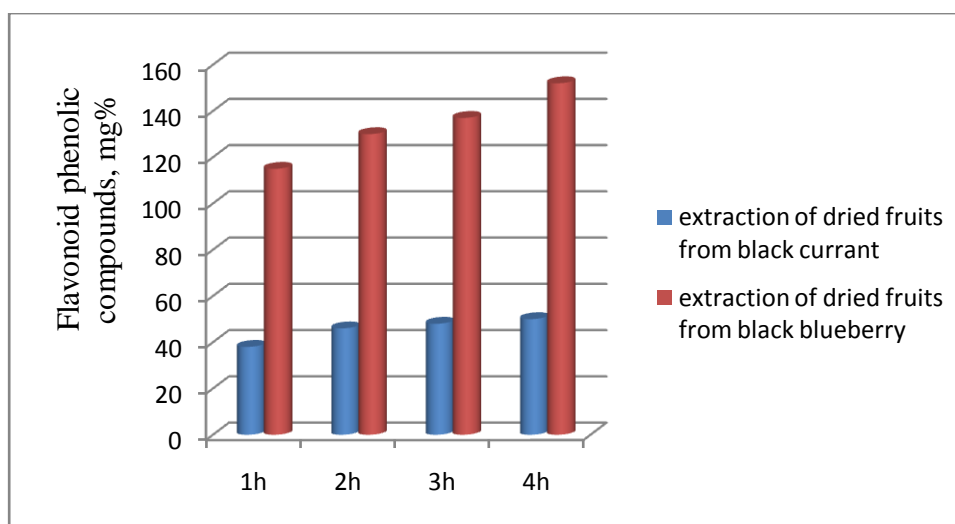


Fig. 10. Influence of the duration of extraction on the content of flavonoid phenolic compounds in extracts of dried fruits black blueberry and black currant

A similar tendency is observed in the flavonoid phenolic compounds. Their concentration ranges from 115 mg% to 152 mg% in black blueberry extracts. The increase is in the order of 32.2%. The content of the flavonoid phenolic compounds increased by 31.6% with an extraction time of 1 to 4 hours (fig.10).

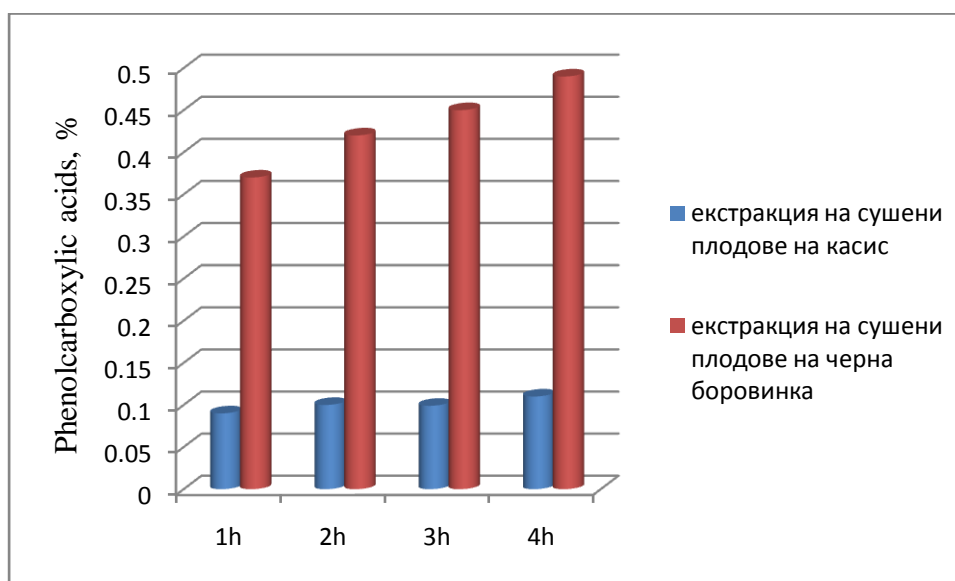


Fig. 11. Influence of duration of extraction on the phenolcarboxylic acids content in extracts of dried fruits black blueberry and black currant

The content of phenolcarboxylic acids extracted from the dried fruits of the black blueberry varies from 0.37% with an extraction duration of 1 hour to 0.49% with an extraction lasting 4 hours. The increase is 32.4%. The amount of phenolcarboxylic acids extracted from the dried fruit currant by extraction of different duration does not lead to a significant difference (fig. 11).

Figure 12 shows the results for the antiradical activity of the extracts obtained at a temperature of 65°C with 70% ethyl alcohol at different duration of the extraction process.

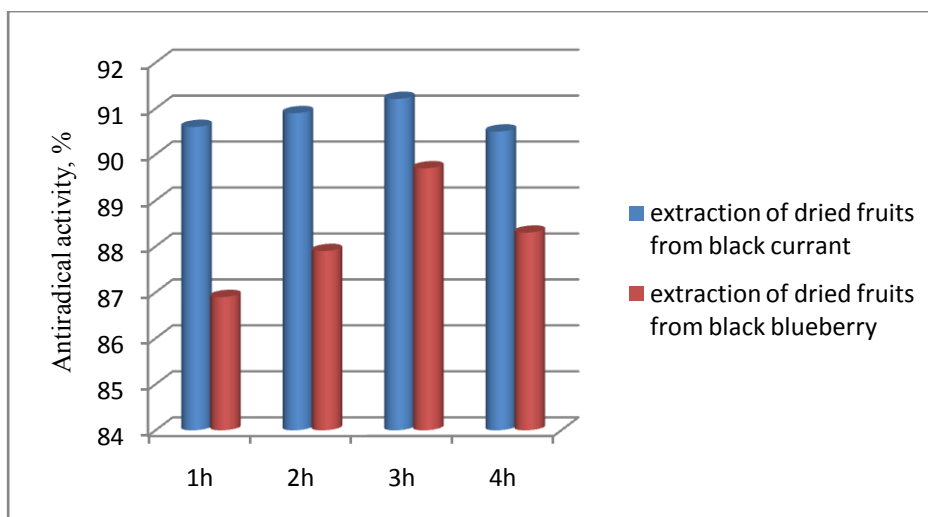


Fig. 12. Total antioxidant activity by DPPH method for the effect of extraction duration on antiradical activity

The content of total phenolic compounds in the extracts varied for different hydromodules from 5.86% for hydromodule 1:10 to 6.27% for hydromodule 1:20 and 7.34% for hydromodule 1:30 for black blueberry and 0.76, respectively. % for hydromodule 1:10 to 1.02% for hydromodule 1:30 for black currant. The technologically justified extraction hydromodule is 1:30 - Fig.13.

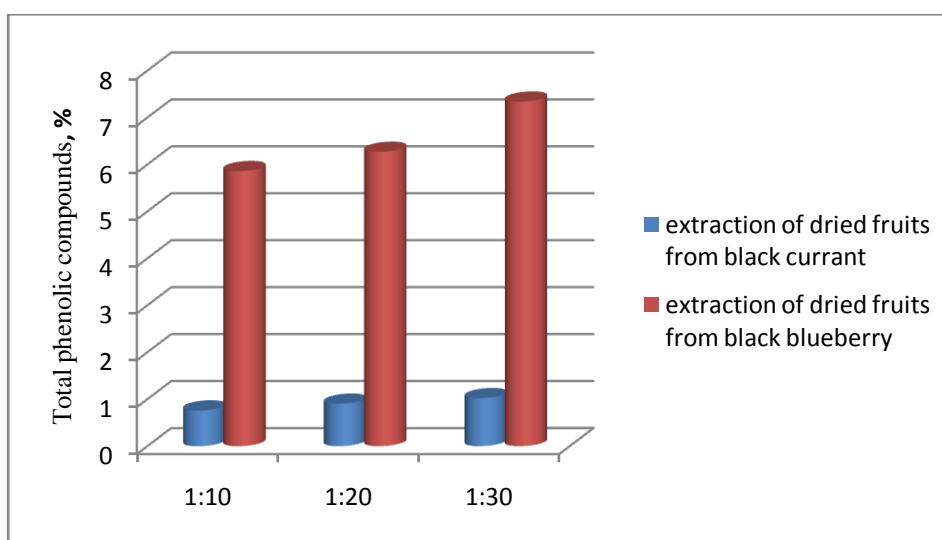


Fig. 13. Influence of the extraction hydromodule on the content of the total phenolic compounds in extracts of dried fruits black blueberry and black currant

The content of the flavonoid phenolic compounds in extracts of dried fruits black currant increased by 31.6% with an extraction time of 1 to 4 hours (fig.14).

A similar tendency is observed in the flavonoid phenolic compounds in extracts of dried fruits black blueberry. Their concentrations ranged from 137 mg%, 176 mg% to 194 mg%, respectively, for hydromodule 1:10, 1:20 to 1:30. The increase is in the order of 28.5% and 44.8% relative to the hydro modul 1:10 (fig. 14).

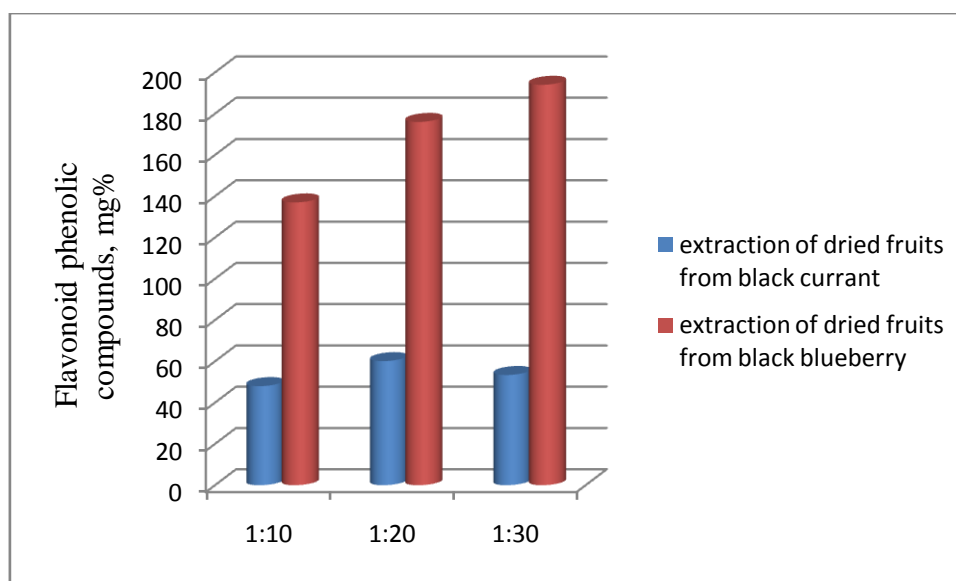


Fig. 14. Influence of the duration of extraction on the content of flavonoid phenolic compounds in extracts of dried fruits black blueberry and black currant

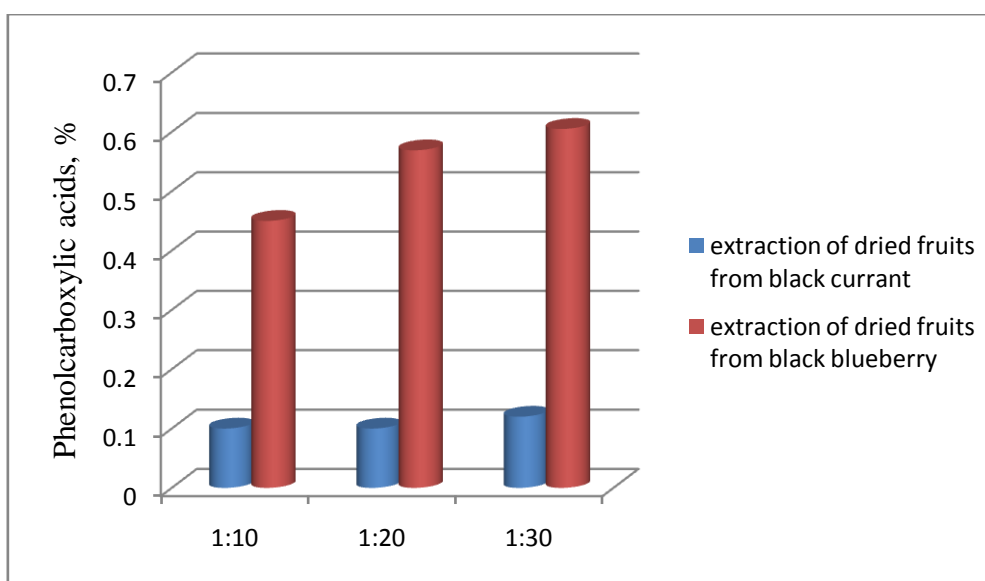


Fig. 15. Influence of duration of extraction on the phenolcarboxylic acids content in extracts of dried fruits black blueberry and black currant

The phenolic carboxylic acids content extracted from the dried black blueberry fruits ranged from 0.45% with hydromodule 1:10 to 0.61% with hydromodule 1:30. The increase is by 35.5%. The amount of phenolcarboxylic acids extracted from the dried fruit currant by extraction of different duration does not lead to a significant difference (fig.15).

In Fig. 16 shows the variation of the antiradical activity of black blueberry and black currant extracts depending on the hydromodule.



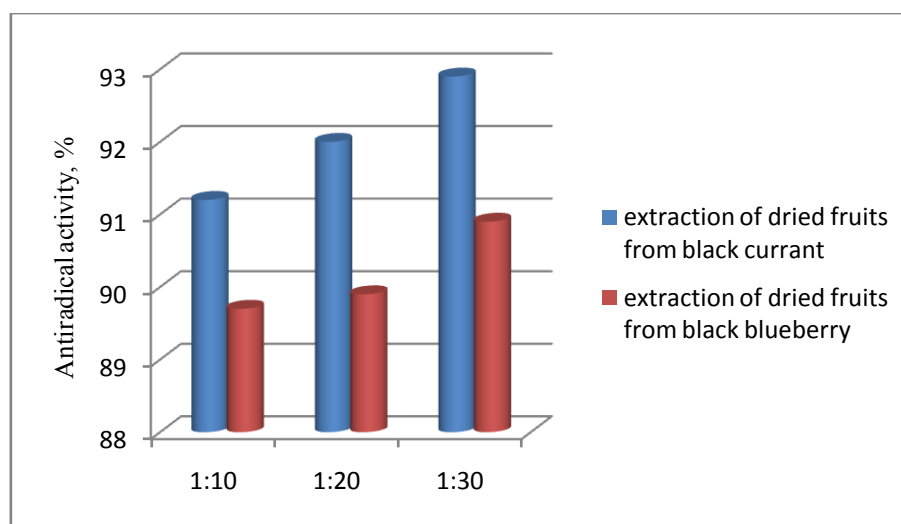


Fig. 7. Total antioxidant activity by DPPH method for the effect of hydromodule extraction on antiradical activity

The results obtained testify to this that by indicator restorative power the optimal hydromodule for carrying out the extraction is 1:30. According to the indicator, the extract exhibits the highest antiradical indicators for the given hydromodule.

Based on the results obtained for the influence of the extraction duration on the phenolic complex content of the dried fruits black currant and black blueberry extracts, the following conclusion can be drawn:

- The ability to capture DPPH free radicals is maximal at a technological extraction time of 3 hours.

### Conclusions

1. Technology for obtaining extracts with maximum content of common phenols has been developed.
2. The results obtained suggest that 70% ethyl alcohol, temperature 65 ° C, duration 3-4h and 1:30 hydromodule are technologically reasonable choices for obtaining extracts with a maximum content of common phenolic compounds.
3. It has been experimentally shown that by increasing the content of total phenolic compounds, the antiradical activity of the extracts increases.
4. The highest is the antiradical activity of the extracts with 70% ethyl alcohol, 65-80 ° C, extraction duration 3-4 hours and hydromodule 1:30.

### References

- [1] Yashin, Y. Natural Antioxidants. Content in Food Products and Impact, Health and Aging, ed. TransLat, 2009, p.212. (in Russian)
- [2] Vesna, T.; Jasna, Č.B.; Lars, G.; Sonja, D.; Gordana, Č. Superoxide anion radical scavenging activity of bilberry (*Vaccinium myrtillus* L.). *J. Berry Res.* 2010, 1, 13–23.
- [3] Stefanova R., A.Georgieva, K.Krastev „RESEARCH ON THE PHYSICO-CHEMICAL COMPOSITION OF BLACK BLUEBERRY AND BLACK CURRANT USED AS THE SOURCES OF BIOLOGICALLY ACTIVE SUBSTANCES IN MAKING FUNCTIONAL BEVERAGES“, International Conference on Technics, Technologies and Education, October 16-18/ 2019, Yambol, Bulgaria
- [4] Akulinina, V. Enriched drinks - a new niche in the juice market. *Tara and opakovka*, 2007, vol. 4, p.16,. (in Russian)
- [5] State Pharmacopoeia of Russia. General methods of analysis - II ed., Medicine. 1987