TECHNOLOGY FOR PURIFYING WASTE OILS WITH NANOSTRUCTURED MATERIALS FOR ENERGY PURPOSES

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The paper proposes the development of advanced, integrated technologies for materials with properties of adsorbents (e.g. bentonite), and the use of these material in processes of used oil recovery with remediation effect and energy recovery. The main objective is to replace liquid fuel, which is an expensive fuel, with revitalized oils for energy production. For these, laboratory experiments are required in two important directions. The first tests are related to the determination the optimum concentration of bentonite for removal of metals from used oil and determining the optimum mixing time. For this purpose, a mathematical model was developed. The revitalization of the used oil leads to the protection of the combustion plant, to the increase of its lifetime and reducing gaseous and solid pollutant emissions. The second set of experiments relates to the determination of energy characteristics of used oil and revitalized used oil for energy production. Elemental analysis, lower heating value, viscosity, density and ash analysis were determined. Experimental test of combustion process on a small scale boiler has been done and the results shows an improved combustion process of revitalized oil compares with used oil and the decrease of the pollutant emissions. Finally, the obtained results were transferred to a real combustion plant for the heating of an auto service.

Keywords: used oil, adsorbents, revitalized oil, energy characteristics, combustion, pollutant emissions.

1. Introduction

Environmental pollution is a topical issue and trying to obtain non-polluting fuels or to recover waste materials for energy purpose concerns many researchers today. It is necessary to solve the problem of waste oil from road vehicles according to DIRECTIVE 2008/98/EC on waste and the national legislation in force: Decision no 235 of 7 March 2007. The legislation provides all operations by which waste oils transferred from the holder to the economic operators who can collect, recover or eliminate those oils or use them as fuel with adequate heat recovery. The present research studies the problem of waste oils

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from the automotive industry and the possibility of their regeneration with energy recovery. Motor oils are used to lubricate the components of the engine assembly and the essential properties of it are: Greasing/lubrication to reduce friction between moving parts, cooling of the parts with which it comes into contact, especially the piston, cleaning of the parts on which residues of the combustion process are deposited (e.g. cylinders, segments, piston), protection against chemical corrosion of engine parts. The used motor oils are contaminated by contaminants and impurities resulted from undesirable oxidation processes: sediment, water, metallic particles and degraded additives [1]. There are several methods to remove these contaminants [2]: in **Mohawk** technology (vacuum distillation) the used oil is chemically pretreated to avoid precipitation of contaminants which can cause corrosion and fouling of the equipment. The distillate is hydropurified at high temperature and pressure in the presence of catalytic bed. The technology is applied in North Vancouver, California, Indonesia and Australia; in **Blowdec** technology (cracking/separation) the process is based on separation of hydrocarbons from the waste oil in a hot whirling bed created by solid particles (hot sand) in a reactor. Simultaneously with the separation, visbreaking process of hydrocarbons occurs. In Markuŝovce, Slovak Republic is applied; in **Dunwell WFE** technology (wipe film evaporation) the used oil from collectors is separated from water and solid particles by centrifuging, then heated to temperature around 150 °C and sent to a flash evaporator for removing water and light hydrocarbons. The technology is applied in Yuen Long Industrial Estate, Hong Kong; in **Prop** technology (chemical treatment/distillation/clay treatment/hydrofinishing) the used oil is treated by water solution of diammonium phosphate for separation of metals and ash-forming components. For this purpose, preheating mixture of used oil and treating agent is sent to contractor where water solution salt is dispersed into the oil. Metallic phosphates formed in the chemical reaction are removed by filtration. Some plants were built, but now they are not in operation because of financial difficulties. **The technology proposed** in the present research is aimed for the process of revitalization of residual oils from motor engines by contacting them semi-dynamic, with natural/modifies nanostructured compounds [3]. In this regard we choose bentonite as nanostructured material. Bentonite powder plays an important role in the regeneration and clarifying of wastes [3]. Research regarding the wastewater treatment using bentonite clay due to chemical composition and crystal morphology, which are indicative of effective physicochemical adsorption [4] shows that the structural configuration (octahedral and tetrahedral) is effective in sorption by intercalation of the turbid materials (heavy metals) onto the sheets and porous surface of the bentonite clay. Bentonite has also been reported to have a high adsorption capacity for heavy metals due to its high specific surface area, small particle size, high porosity and high cation exchange capacity [5] The adsorption technique has been found to be superior to the other techniques for removal of heavy metals, in terms of cost, flexibility, simplicity of design, ease of operation, insensitivity to toxic pollutants and better removal efficiency [6], [7], [8].

2. Materials and methods

Samples analyzed are two types of used motor oils: one comes from an spark-ignition engine from a Renault vehicle brand, Symbol model, 1589 cm3 cylinder capacity, manufactured in 2008, and the other from a compression-ignition engine from a Volkswagen vehicle brand, Passat model, 1989 cm3 cylinder capacity, production year 2008.

As material for regenerating the used oil, activated bentonite has been used. Activated bentonite is obtained by processing the crude bentonite $Al_2O_3 \cdot 4SiO_2 \cdot 4H_2O$ exploited from the calcium bentonite quarry from Orasu-Nou, activated by a specific process. Due to the many physical and chemical properties such as high water absorption capacity, high ion exchange capacity, high intercrystalline swelling in aqueous medium and high plasticity, bentonite can be appropriate for contaminants removal. The purpose of using bentonite in this experiment is related to its absorption capacity of the contaminants and regeneration of the waste motor oil technical characteristics of bentonite are: maximum granulation: rest on sieve Ø 0,063 mm - max.25% bulk density: 800-850 kg/m^3, humidity: max. 10% pH: min.8, bentonite number: min. 0.85.

The physico-chemical characteristics of motor oils change as a result of their exploitation [4]. The operating conditions lead to pollutants accumulation in the oil that changes the quality of engine oil. Analyzes on density and viscosity of the different kind of motor oils (raw and used) were performed. Three samples have been considered: raw motor oil, waste oil from spark-ignition engine and waste oil from a compression-ignition engine.

The carbon (C), nitrogen (N), hydrogen (H) and sulphur (S) contents of the waste motor oil samples were determined by a CHNS elemental analyzer EA 1110 (**Fig. 1**). Determinations were performed at ICEMENERG. The Lower Heating Power was determined according to ASTM D240-17;

Fig. 1 Elemental analyzer EA 1110

The experimental researches were conducted in the Combustion laboratory of the Department of Thermodynamics, Engines, Thermal and Refrigeration Equipment, Faculty of Mechanical Engineering and Mechatronics of Politehnica University of Bucharest. For combustion tests a pilot boiler of 55 kW has been used. The boiler is equipped with a special burner for liquid which ensures the preheating of the fuel up to 90 °C, temperature imposed by the high viscosity of the analyzed fuel [9].

For the tests, used diesel oil and regenerated used diesel oil after treatment with bentonite with 30 g/liter (optimal concentration is between 20 and 40 g bentonite/liter, see Chapter 3.2) has been used. Obviously, several tests were performed to determine the optimal concentration range. During the experiments the pollutant emissions from the flue gases has been analyzed using a Maxilyzer NG gas analyzer.

Fig. 2. Experimental pilot boiler of 55 kW

Fig. 3. Burner Anyo-12

Fig. 4. Flue gas analyzer MAXILYZER NG

3. Experimental determinations

3.1 Determination of energy characteristics

High viscosity of used motor oil determined during the experiments (see Table 1) imposes the necessity of used oil preheating for the combustion process. The results of the elemental analysis are presented in the Table 2.

Table 1

Viscosity and density of motor oil

	Type of sample /property	**Temperature, t**	**Viscosity**	**Density, ρ**
		°C	E^0	*kg/m³*
1.	Raw motor oil	75-80	2.4	840-850
2.	Used oil from spark-ignition engine	75-80	2.6	870-880
3.	Used oil from compression-ignition engine	75-80	2.7	840-850
4	Regenerated oil with 30 g/liter	70-75	2.5	830-850

Table 2

Elemental analysis of the used oil

Type of sample	***C***	***H***	***N***	***S***	***LHV***
	%	%	%	%	MJ/kg
Used oil from spark-ignition engine	83.56	13.77	0.41	0	41.96
Used oil from compression-ignition engine	84.12	14.29	0.33	0	42.33
Regenerated oil with 30 g/liter	84.52	14.36	0.31	0	42.51

The energy characteristics are generally quite close between the various types of oil. The composition of the used motor oil, regenerated (revitalized) oil and the value of LHV are comparable with liquid fossil fuel as diesel fuel and this recommends it for energy recovery [10]. In further the experiments will be performed according to the logic diagram in Fig. 5.

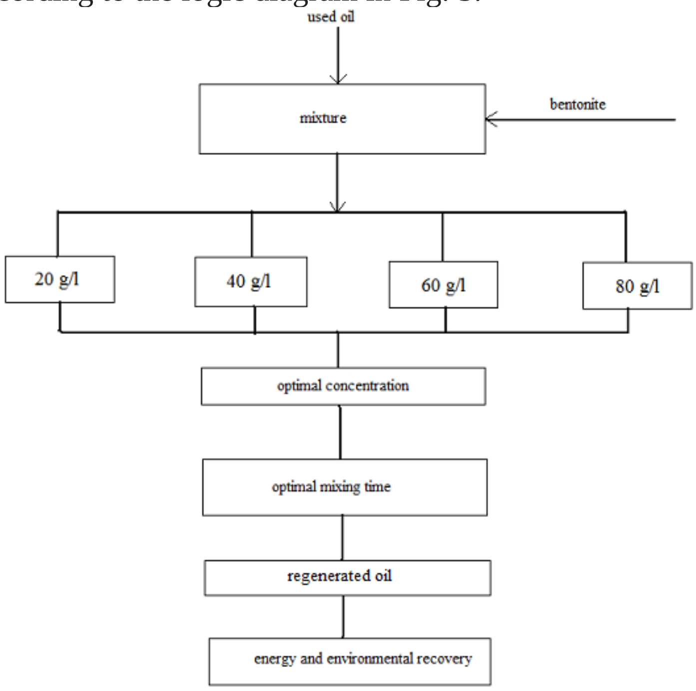


Fig. 5. Logical scheme for performing experiments

3.2 Mathematical model concerning to the adsorption capacity and mixture time

Starting from the Kul and Koyuncu equation [11]:

$$Q_t = \frac{C_0 - C_t}{m} \cdot V \quad (1)$$

where: Q_t is the amount of metal ions adsorbed, mg / g; C_0 - initial concentration of pollutant solution, mg / liter; C_t - solution concentration at time t, mg / liter; V - volume of solution in tank, liters and m - amount of adsorbent, g. The adsorption is done from the initial concentration, to the equilibrium concentration. The adsorption capacity at equilibrium is given by Freundlich's isotherm:

$$Q_e = K_F \cdot C_0^{1/n} \quad (2)$$

where, in addition: Q_e it is the adsorption capacity at equilibrium, mg / g; K_F - Freundlich's isothermal constant; n is Freundlich's constant. The constant 1 / n has values between 0 and 1. Both constants depend on the adsorbent material (in this case, bentonite). First, the adsorption capacity at equilibrium is determined and then the contact time from the initial concentration to the equilibrium is determined (V is required, different m , K_F = (0.1 - 0.2) and 1 / n = 1). From the analysis of waste oils (both from M.A.S. and from M.A.C.), the initial concentration of metal ions is between 50 and 200 mg / liter. For classes are required, namely: 50, 100, 150 and 200 mg / liter. The adsorption capacity at equilibrium is calculated for these 4 classes. The values are shown in the Table 3.

Table 3

Initial amount of metal ions and the adsorption capacity

The initial amount of metal ions, mg / liter	50	100	150	200
The adsorption capacity, mg / liter	5	10	15	20

After mixing, we removed the mixture between adsorbed metal ions and bentonite. The mixing time was not monitored in these experiments. The ash sample was burned by burning the mixture for 2 hours at 815 degrees Celsius. We then performed the elemental ash analysis using an Agilent 8800 Inductive Coupled Plasma Mass Spectrometry (ICP-MS) equipment (Agilent Technologies, Japan). Prior to the measurements at the ICP-MS, the samples were individually weighed and placed in special tubes of TFM. After the addition of 2 ml of HNO3, the samples were digested in a microwave system (Milestone Ethos, FKV, Bergamo, Italy), at 200 ° C for 35 minutes, maximum power of 1800 W. After cooling, the digestion liquids were diluted with ultrapure water to 25 ml [12], [13]. The measurements were made for the most abundant isotopes of each element. The results are presented in Table 4 and in Fig. 6 and Fig. 7.

Table 4

The results from the analysis of the ash of bentonite mixed with the used oil

Sample	Quantity [g]	56 Fe [mg/ g]	63 Cu [mg/ g]	66 Zn [mg/ g]	111 Cd [mg/ g]	208 Pb [mg/ g]
B 20 g	0,1009	63,24	0,077	9,8	0,0012	0,122
B 40 g	0,1005	75,24	0,058	10,5	0,00078	0,178
B 60 g	0,0999	71,16	0,043	9,4	0,00091	0,101
B 80 g	0,1002	71,93	0,046	9,8	0,0011	0,482

From these analyzes, two graphs were drawn to choose the best sample for the experiment.

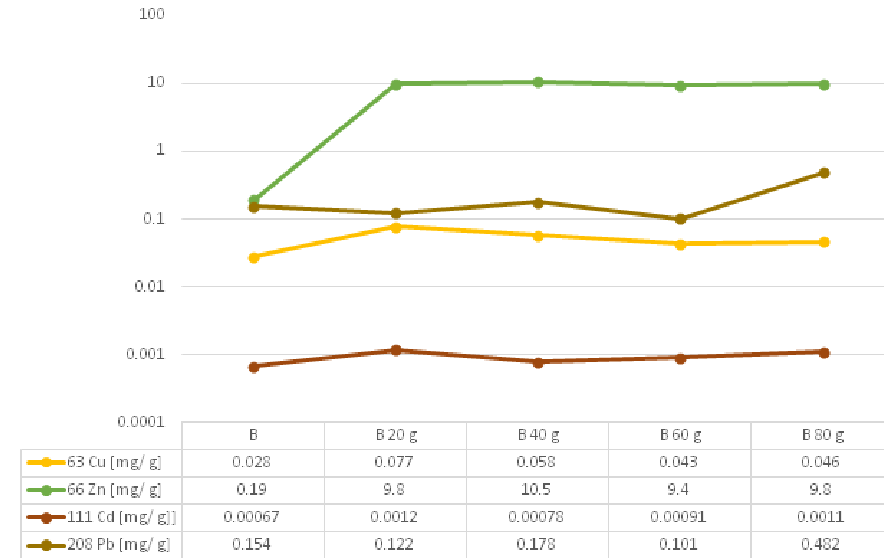


Fig. 6. Graphical determination of the impurities Cu, Zn, Cd, Pb, adsorbed by bentonite

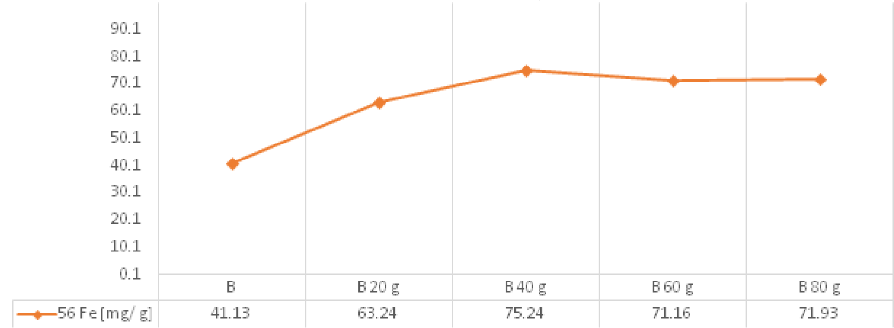


Fig. 7. Graphical determination of Fe impurities, adsorbed by bentonite

The results obtained lead to the following conclusion: for the elements Cu, Zn, Cd and Pb, the best adsorption efficiency is at a mixture of 20 g of bentonite per liter of waste oil, and for Fe the optimal concentration is 40 grams. The optimal mixing time is in the order of minutes (result from the mathematical model, solved only for the optimal concentrations of 20 and 40 grams of bentonite). It was mentioned that the mixing was done manually, with a mixer with battery. Obviously, at the installation to be implemented, the mixture from the waste oil tank will be coordinated by an automatically operated electrical device.

In order to calculate the minimum mixing time between waste oil and bentonite, in order to reach the equilibrium adsorption capacity with minimum electricity consumption, we derived, in relation to time, the equation:

$$\frac{dQ_t}{d\tau} = \frac{V}{m}\left(-\frac{dC_t}{d\tau}\right)\Big|_{Q_t}^{Q_e} \tag{3}$$

The adsorption is done from the initial concentration, to the equilibrium concentration. The adsorption capacity at equilibrium is given by Freundlich's isotherm, relation 2. The equation that describes the variation of concentration in time is:

$$\frac{dC_t}{d\tau} = ak_L(C_e - C_t) \tag{4}$$

where, in addition: ak_L is the volumetric mass transfer coefficient (does not vary in time). This constant was determined by tests and validated by experiments; $d\tau$ – time period.

$$\frac{dC_t}{(C_e - C_t)} = ak_L \cdot d\tau \Rightarrow -\ln|C_e - C_t| = ak_L \cdot d\tau + ct. \tag{5}$$

At $\tau = 0$, $ct. = -\ln|C_e - C_t|$ and $C_t = C_0$. Finally:

$$(C_e - C_t) = (C_e - C_0) \cdot e^{-ak_L \cdot \tau} \Rightarrow C_t = C_e + (C_0 - C_e) \cdot e^{-ak_L \cdot \tau} \tag{6}$$

The relationship that establishes the equilibrium equation is:

$$(C_0 - C_e) = \frac{m \cdot K_F \cdot C_0^{\frac{1}{n}}}{V} \tag{7}$$

The mathematical model was solved by means of a program adapted in Excel using the equations presented above. The results are presented in the Table 5.

Table 5

Optimal mixing time to adsorption

The initial amount of metal ions, mg / liter	50	100	150	200
The adsorption capacity, mg / liter	5	10	15	20
Mixing time, at 20 g of bentonite, sec.	2261	2335	2409	2483
Mixing time, at 40 g of bentonite, sec.	1269	1354	1456	1590

From Table 5 were represented graphically 2 representative variants, Fig. 8 and Fig. 9.

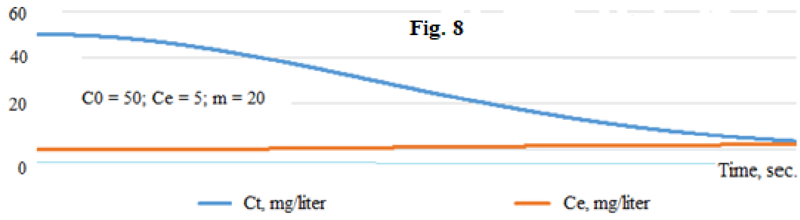


Fig. 8

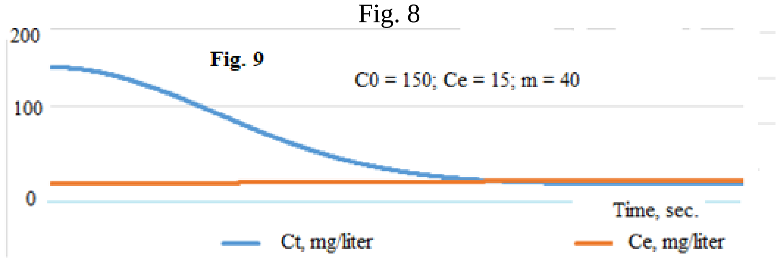


Fig. 9.

3.3 Tests for combustion processes

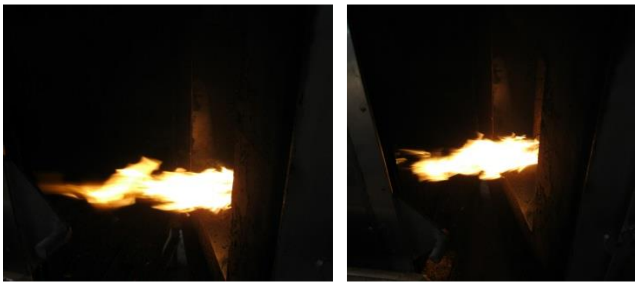
The experimental researches were conducted in the Combustion laboratory of the Department of Thermodynamics, Engines, Thermal and Refrigeration Equipment, Faculty of Mechanical Engineering and Mechatronics of Politehnica University of Bucharest. The combustion process of the samples was analyzed in terms of pollutants emissions and efficiency of the process and the results are presented in Table 6. The aspect of flame in the burning process is presented in Fig. 10 and Fig. 11.

Table 6

Result of the combustion experimental tests

No.	**O_2 [%]**	**CO [ppm]**	**CO_2 [%]**	**NO_x [ppm]**	**SO_2 [ppm]**	**t_g [∘ C]**	λ
Used oil							
1	17.3	1628	2.7	11	0	238	5.68
2	17.3	1722	2.7	14	0	238	5.68
3	17.2	1813	2.8	15	0	242	5.53
Revitalized oil (30 g/liter)							
1	16.9	1172	3.0	32	0	280	5.12
2	16.9	1293	3.0	31	0	278	5.12
3	17.4	1277	2.6	24	0	263	5.83

The concentration of CO in flue gas is high because we used a burner (design for CLU combustion) coupled with a natural circulation boiler, strongly cooled and the burner's thermal power is lower than the furnace thermal power. It is mentioned that CO emitted in the flame does not burn below the temperature of 750 C, the temperature reached only in the final part of the furnace. Considering all this, it is recommended a boiler with chamotte insulation, uncooled which due to high combustion temperature will significantly reduce the final CO emissions. Analyzing the results of the combustion process, it note that the used oil regenerated with bentonite has better performance compared to raw used oil with a decrease of CO emissions of about 25%.



a b

Fig. 10. Aspect of flame in the burning process a. raw used oil; b. regenerated used oil

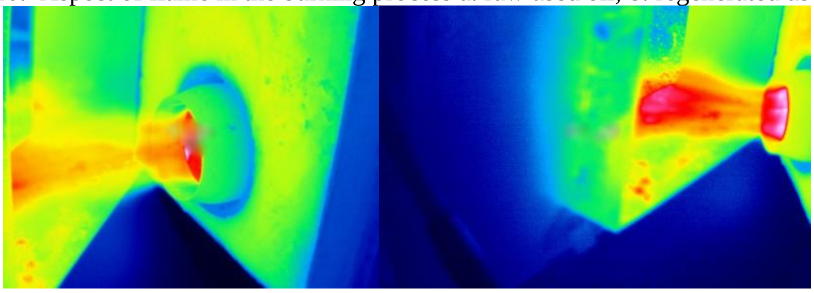


Fig. 11. The appearance of the flame with the help of the thermal imaging camera
a. raw used oil; b. regenerated used oil

Due to the low volatile matter content of the used oil motor the flame presents pulses and a longer length than the reference flame (diesel oil). It is noted that the regenerated oil has attenuated pulses comparing with raw used oil and the ignitions closer to the spray nozzle. The use of bentonite as a material to regenerate used engine oils proves to be an effective solution in lowering the metal content and improving the combustion process. The reducing of metals concentration in used oil influence directly the burner lifetime by protecting the spray nozzle. Improvement of the combustion process was achieved by higher flame stability and lower CO emissions. It expects that by better adaptation of the burner-furnace assembly to increase the efficiency of the combustion process. The results can be extrapolated to other types of used oils such as oils from the energy industry or waste oils from the food [14].

4. Industrial combustion test

In this chapter, first of all, the diameter of the spray nozzle was determined. This nozzle will equip the mechanical injector of the combustion plant. The boiler is imposed by the calculations of the heat required of the building in Chapter 4.2

4.1 Calculation of the maximum drop diameter

The efficiency of the combustion plant depends on the judicious choice of the constructive and functional parameters of the burners used [15]. The characteristics of the spray jet in the mechanical burners with whirling chamber (with return) are:

- Fuel flow (capacity of burner) and its adjustment range;
- The jet expansion angle;
- Density of distribution of liquid fuel;
- Spray fineness (average diameter and droplet distribution);
- Maximum diameter of the fuel drop.

These characteristics must be achieved for the well-defined physical properties of the revitalized oil used: (viscosity, surface tension and density) and at a preheating temperature required (75-85ºC) for an appropriate fluidity (2-3ºE). To obtain a good fineness of the drops formed when using the revitalized oil (characterized by the kinematic viscosity ν = 12-19 cSt, the surface tension σ = 2,4-2,6 N / m and the density ρ = 830-870 kg/m^3), between the supply pressure and the outlet diameter, the following correspondence is recommended: $p_{al} \leq 15$ bar, flow rates G ≤ 1500 kg / h, diameter of the whirling chamber (can be chosen 6 ... 7 mm) and the convergent angle of the nozzle output γ = 90...120º.

An important characteristic of the mechanical centrifugal burner, which largely determines its applicability, is the size distribution of the droplets formed. The simplified method for estimating the fineness of the spray by the maximum drop diameter is described in the paper. Experimental studies have shown that the maximum diameter of the droplets in the sprayed liquid is proportional to the radical of the injector scale and inversely proportional to the square root of the total pressure drop in the injector, according to the relationship below:

$$d_{\max} = C \cdot \sqrt{M / \Delta p} \quad (8)$$

where: d_{max} is the maximum droplet size, in μm; M-scale of the injector, in mm^2, Δp-pressure drop through the injector (generally equal to the supply overpressure), in bar; C is a proportionality coefficient, which depends mainly on the physical properties of the spray liquid and the processing quality of the injector component parts. For injectors with higher processing quality C = 1500 and for those with average quality C = 2000. As a first step, calculate the scale of the injector with the relation:

$$M = \frac{G}{0{,}77 \cdot \sqrt{\Delta p \cdot \delta}} \quad (9)$$

For small and medium thermal powers ($P_t \leq 200$ kW) results a burner capacity of approximately 25 kg / h, at a lower calorific power of the oil of 37,000 kJ / kg. In these conditions the injector scale M = 0.288 mm^2 (for a Δp = 15 bar). Finally, the maximum drop diameter is calculated:

$$d_{max} = 1500 \cdot \sqrt{0.288/15} = 207\ \mu m$$

It can be concluded that a diameter of the 0.4--0.5 mm spray nozzle is optimal for the mechanical centrifugal burner which will equip the combustion plant on which the revitalized oil combustion experiments were carried out.

4.2 Validation of experimental results

To demonstrate the viability of laboratory experiments, waste oil regenerated technology was implemented on a low-power industrial combustion plant. The purpose was to heat in the cold period (November - March) a building consisting of 2 connected halls (**Fig. 12**). The first building is intended for the periodic technical inspection of vehicles and the second is intended for a mechanical workshop (car service, which obviously includes the change of engine oil). The constructive dimensions of the halls are: first hall (length 17.9 m, width 6.9 m height 4.7 m); second hall (length 14.3 m, width 6.9 m height 4.7 m). The thickness of the walls is 0.20 m. The other characteristics of the halls are: the concrete floor, the brick walls, the sheet and the glass wool roof, the PVC joinery and the metal resistance structure. Both halls are equipped with garage doors.

The thermal requirement was calculated for an outdoor temperature of -10 degrees Celsius and for an indoor temperature of +20 ºC for the first hall and +18 ºC for the second. The result was a thermal demand of 80 kW (43 for the first hall and 37 for the second). Then, the efficiency of the combustion plant and the consumption of fuel (revitalized oil) were calculated. Two combustion plants of the HITON HP145 (**Fig. 13**) hot air generator type of 42 kW each were chosen. The oil is sprayed through a mechanical return injector with a spray nozzle diameter of 0.5 mm. In 2019, January 14, the flue gas analysis was performed (Maxilyzer NG analyzer) for the combustion plant that operated with waste oil and in 2020, February 10, on the same installation, but supplied with revitalized oil. The results are shown in the **Table 7**. The regenerated of the waste oil (at a concentration of 30 grams of bentonite per liter of waste oil) was done in an external tank, after which the own tank of the combustion plant was supplied.

Table 7

Result of the industrial combustion tests

No.	**O_2 [%]**	**CO [ppm]**	**CO_2 [%]**	**NO_x [ppm]**	**SO_2 [ppm]**	**t_g [◦ C]**	λ
Used oil							
1	12.8	169	6.1	33	0	179	2.56
2	12.9	170	6.1	34	0	178	2.56
3	12.7	168	6.1	33	0	180	2.56
Revitalized oil							
1	14.1	91	5.4	51	0	200	3.04
2	13.7	94	5.2	52	0	196	2.88
3	13.9	76	5.3	51	0	198	2.96
4	13.6	92	5.0	53	0	197	2.84

Fig. 12. The building with 2 halls

Fig. 13. The Hiton combustion plant

The experiments will continue, by modernizing the used oil revitalization plant, according to Fig. 14. This will increase the degree of automation, the quality of the used oil-bentonite mixture and the purity of the revitalized oil.

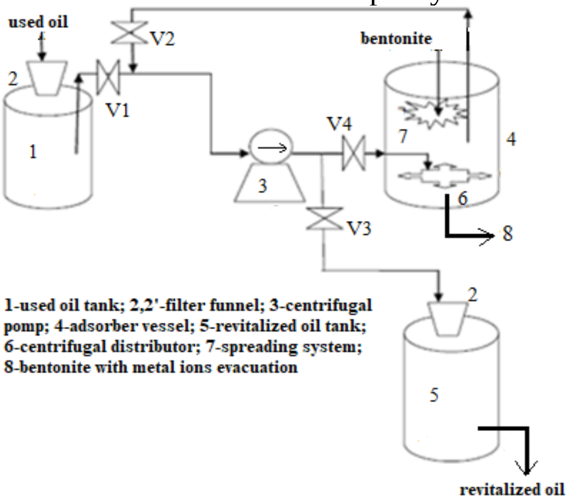


Fig. 14. Used oil revitalization technology scheme. Phase I, V1 and V4 open, V2 and V3 closed; phase II, V1 and V4 closed, V2 and V3 open.

5. General conclusions

- Waste oil purification technology using nanostructured materials is a new technology. It is simple, efficient, inexpensive and non-polluting;
- By revitalizing the oils, their combustion process improves, the combustion temperature increases, the concentration of carbon monoxides decreases at the laboratory installation approximately 25% and at the industrial one by 40-50%;
- The revitalization method led to a 90% reduction in the emission of solid particles (in the form of metal ions);
- By increasing the temperature, the concentration of nitrogen oxides also increases. However, this falls within the legal limit concentration;

- By retaining metallic impurities, the service life of the spray nozzle increases. The initial spray angle of the injector is also maintained, ensuring optimum combustion;
- Increasing the degree of automation by performing the new installation will improve the efficiency of the method presented in the article;
- The objectives assumed at the beginning of the experiments were achieved. Moreover, by continuing research, they can be surpassed and exported to other types of waste oils.

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