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ENVIRONMENTAL ASPECTS OF CONTINUOUS BIODIESEL PRODUCTION AT PILOT LEVEL BY HETEROGENEOUSLY CATALYZED OIL METHANOLYSIS

Abstract: *The environmental aspects of a continuous biodiesel production pilot process based on the sunflower oil methanolysis employing the quicklime as a solid catalyst were analyzed. The environmental impacts of important factors present in all phases of the proposed pilot process were evaluated. The aim of the work was to indentify the potential harmful effects of the proposed process for biodiesel production on the environment and to propose the preventive and protective measures.*

Key words: biodiesel, ecological and environmental aspects, heterogeneous catalysis, methanolysis, safety.

INTRODUCTION

In past ten years demand for biofuels across the world has grown. Among the biofuels, the biodiesel draws attention to both researchers and producers because it represents an eco-friendly alternative to conventional diesel fuel due to its biodegradability, low toxicity and less harmful emmisions during the combustion (reduced particulate matter, carbon monoxide and dioxide, sulphur dioxide). By definition, it is a mixture of fatty acids and short chain alcohol that meets biodiesel quality standards.

Biodiesel has a significant position on the fuel market because of its properties that are very similiar to those of conventional diesel fuel. For instance, its application does not requare significant modification of existing diesel engine. In order to meet the world demands for biodiesel, the higher production capacities must be provided and therefore the interest in the installation of continuous production plants is growing. Generally, industrial production of biodiesel in the world at present is mostly based on the homogeneously catalyzed oil alcoholysis. Recently, much effort has been made aiming at developing the technologies based on the use of heterogeneous catalysts because of many advantages over homogeneous ones. By employing solid catalysts, the neutalization step of by-product (glycerol) is eliminated, so the process becomes cheaper and safer [1]. The best-known commercial process for biodiesel production based on the heterogeneously catalyzed vegetable oil methanolysis is the Esterfip process [2]. High quality biodiesel and glycerol are obtained in two successive fixed bed reactors at higher temperature and pressure. The mixed oxide of zinc and aluminium is used as a catalyst. This process is more environmentally friendly bacause the glycerol neutralization and methyl esters washing steps are not included. The envoronmental benefit of biodiesel is already well-known; however, the eco-environmental aspects of the overall biodiesel production are very important as well. Instalation of large capacity plant for biodiesel production not only increases the equipment size but also generates higher quantities of hazardous and flammable materials that need to be handled in a proper way to prevent potential pollution of the environment. Therefore, existing regulations for the eco-environmental impact assessment of industrial biodiesel production are very important. The regulations of the Republic of Serbia lay down the rules and the standards that any industrial production must meet. Environmental impact assessment considers the satus of the environment, the eco-environmental impact of implemented technology for biodiesel production, definition of measures for prevention and environmental monitoring program.

In this work the environmental aspects of a biodiesel pilot production process based on the vegetable oil methanolysis over quicklime as a cheap catalyst were analyzed. The aim of the work was to identify the potential eco-environmental impacts of the proposed biodiesel production process, to perform the hazard analysis of each production step and to define the protective measures in order to minimize, prevent or manage the potentional hazardous impacts according to the existing law regulations.

CONTINUOUS BIODIESEL PILOT PRODUCTION PROCESS BASED ON VEGETABLE OIL METHANOLYSIS OVER QUICKLIME

The sunflower oil methanolysis over quicklime bits in a flow packed-bed reactor [3] was assessed with respect to the environmental impact. The global scheme of the proposed process is presented in Figure 1.The pilot process includes the biodiesel production and purification. According to the proposed process, the sunflower oil methanolysis is performed at the atmospheric pressure and the reaction temperature of 60 °C, is kept constant by circulating hot water (at 60±2 °C) through the reactor jacket. Methanol is mixed with the vegetable oil prior to entering the reactor and then the mixture is heated up to 60 °C flowing through a heater by exchanging heat with hot water (60±2°C. Centrifugal pumps are used for transporting the reactants from the reservoirs to the reactor. Calcined quicklime bits (2-3.15 mm) are used as a catalyst in the form of packed bed. The catalyst bits are calcined in a furnace at 550 °C within 4 h.

The biodiesel plant is consisted of two identical packed-bed reactors whereas one of them is used as a spare reactor. The laboratory research showed that after 30 h of running the flow through the catalyst bed was blocked due to the catalyst bits agglomeration [4]. Therefore, the process is running continuously in 24 h, when the feedstream is redirected to the spare reactor. While running the second reactor, the first one is prepared for the next use bydischarching the spent catalyst and filling with a fresh batch of the catalyst bits. The effluent (reaction mixture consisted mainly of methyl esters, glycerol and the excess of methanol) is pumped to a flesh evaporator in order to remove the excess of methanol which facilitates the methyl esters separation in a gravitational separator. After separating from glycerol (containing an amount of methanol), the methyl esters are purified by a two step process followed by filtration. The final separation of methanol and glycerol is done by vacuum evaporaton. Methanol from the two separation steps is reused with fresh methanol.

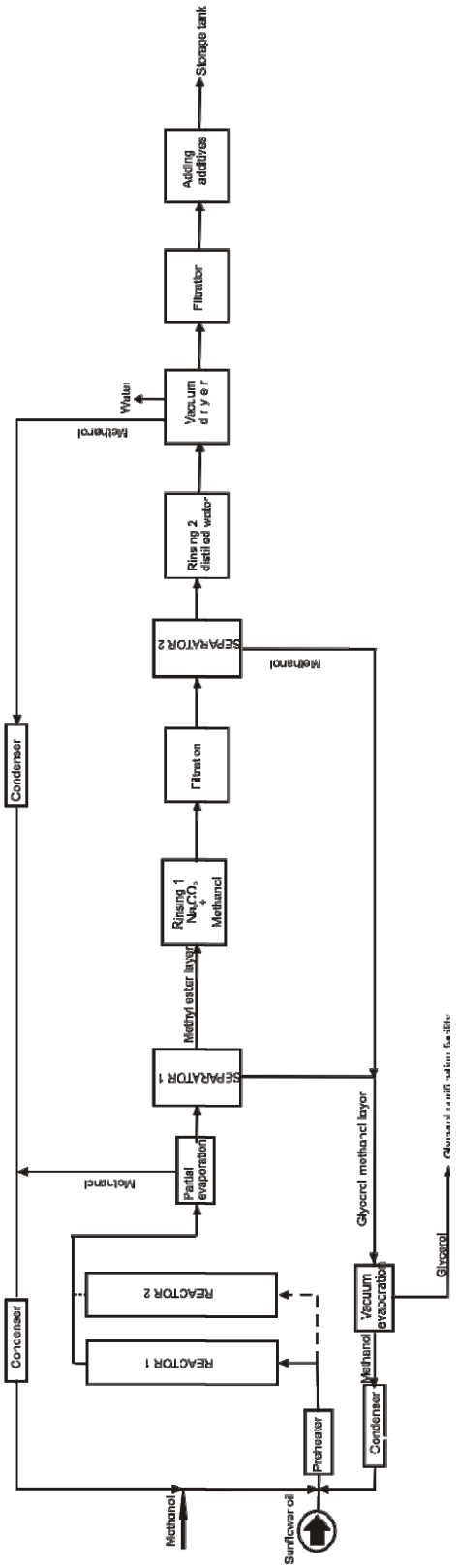

Figure 1 Global scheme for continuous sunflower oil methanolysis catalyzed by quicklime bits (dashed line - redirection of feedstream from reactor 1 to reactor 2)

ENVIRONMENTAL ASPECTS THE VEGETABLE OIL METHANOLYSIS OVER QUICKLIME

The environmental aspects of the biodiesel production pilot process were analyzed through following steps:

- Catalyst preparation,
- Feedstocks preparation and handling,
- Methanolysis of sunflower oil,
- Separation and purification of crude methyl esters,
- Addition of additives to methyl esters,
- Glycerol purification and
- Storage of the final products.

Catalyst preparation

Quicklime (basically, calcium oxide) is a low cost material and it will be employed as a heterogeneous catalyst in the process of oil methanolysis. It is purchased from a quicklime plant in the form of bits (1-5 mm), which are sieved to separate the bits of the specified size. Since dust can be generated during the sieving, the working room must properly be ventilated. It is recommended to collect dust settled down by means of wet clothes, which are then water washed in a bucket. Quicklime belongs to the group of non flammable solid materials (melting point 2572 °C and boiling point 2850 °C) that vigorously react with water realasing a large amount of heat. Since this can cause the ignition of combustible materials with a harmful impact on the environment, quicklime should be stored properly. Qucklime must not be stored near hydrogen halides, halogen compounds, acids, light metals and water. It should be stored in well closed containers made of proper materials in a cool and dry place. Quicklime is classified as Fx IV J by the Regulation on technical and other requirements for materials and goods according to their behavior in a fire [5]. Also, since quicklime contact with eyes and skin can cause irritation or burning, the employees that work with it should wear safety goggles, gloves and clothing that fully cover arms and legs. Quicklime does not show any bioaccumulation effect and can be used for soil stabilization and remediation. If it is transported, handled and stored properly, it does not have any hazardious effect on the environment.

Feedstocks preparation and handling

The main feedstocks for biodiesel production are sunflower oil and methanol. They do not require any special pretreatment and will be used in the form as they are purchased. Methanol is mixed with sunflower oil in the required molar ratio (6:1); the mixture is heated to 60 °C flowing through the preheater and then introduced into the reactor at its bottom. The centrifugal pumps are used for the sunflower oil and methanol transportantion from the external overground reservoirs to the reactor. Sunflower oil from a tanker truck is discharged by means of a pipeline (or hose) into the external storage tank. It is very important for sunflower oil to be stored in a dry atmosphere. It belongs to non-hazardous materials as its auto-ignition temperature is over 300 °C; therefore, it does not have any negative effect on the environment. Methanol is colorless, clear, toxic, volatile, highly flammable liquid with dinstinctive odor and soluble in water. It boils at 64,5 °C and solidifies at -98 °C. Its ignition and auto-ignition temperature are 11°C i 455 °C, respectively. It can combust in the presence of oxygen and carbon dioxide with a pale blue flame that changes colour with time. Methanol vapours form an exsplosive mixture with air in the concentration of 6.7–36 % [6] that is flammable in the presence of static electricity. Also, methanol reacts with oxidizing materials. Considering the high toxicity of methanol and its vapours as well as its solubility in water, any accident with metanol causes the pollution of the water, soil and air and is harmful for public health. If methanol is handled and stored properly according to the standards and regulations, then the accidents and pollution of environment will be prevented.

Generally, the liquid feedstocks must be handled following the standards and regulations in order to avoid incidents and accidents during transportation, operation and storage [1, 7]. For the purpose of their storage, the external vertical tanks equipped with all required technical devices for the parameters (temperature, pressure and quantity) control and monitoring will be used. A particular requirement for the methanol storage tanks is to maintain the overpressure at a constant level in the inert atmosphere in order to minimize the risk of fire and methanol emission in the air. The storage tanks must be constructed according to the corresponding safety standards and regulations and must be placed in protective, waterproof bund. In this way, the pollution of soil and underground water will be prevented.

During certain production steps, mostly related to the manipulation and maintaince such as handling of feedstocks and final products or leakage from pipelines, the wastes can be generated. This type of wastes could be also harmful to the environment because of its toxicity, explosivity and flammability. Also, the certain amount of waste can be generated during the lab testing of feedstocks and products quality. In the case of spilled liquid wastes, an adequate absorbent should be used, which should, after the use, be disposed properly in a PE HD bag in the waste disposal storage. It is also necessary to provide the spill boxes for collecting spills at the connection points of transport hoses. The collected waste should immediately be disposed in the container specified for the corresponding waste located in the waste disposal storage.

Methanolysis of sunflower oil

The production of biodiesel by methanolysis of sunflower oil over quicklime is conducted continuously, as a closed process with full automatization of the reactor system. As the spillage of raw materials, intermediate or final products into waterway or soil that can endanger the working and

living environment, it is essential that the process takes place in a closed production hall. The facility should be made in the form of an impermeable bund connected by a pipeline with the lowest reservoir for incidental spills that prevents any further spillage.

The sunflower oil methanolysis will be performed in the flow packed-bed reactor running 24 h at atmospheric pressure. Since the catalyst bed is blocked after 24 h operation, the flow of the reactants mixture is redirected to the second reactor. During the in the operation of the second reactor, the first reactor is emptied and then filled with the fresh catalyst bits. The spent catalyst can cause an environmental pollution problem if disposed improperly as a waste. In the case of spillage of the spent catalyst, it is necessary to collect it in the proper container which is the same as that used for quicklime dust. Since the spent catalyst contains Ca diglyceroxide on the catalyst surface [8], it is necessary to conduct a further investigation in order to define the appropriate procedure of its treatment.

In order to achieve a high oil conversion degree, the methyl ester synthesis should be realized with the methanol excess. After completing the reaction, the unreacted methanol will stay in the final reaction mixture that complicated the separation of the two layers that is methyl ester and glycerol, due to the solubility of glycerol in methanol [9]. Therefore, in order to achieve a faster separation in the gravitational separator, it is necessary to perform flash evaporation of methanol. A certain amount of methanol retaining in the glycerol layer can be removed by distillation. The methanol vapors are condensed in a condenser using cooling water from the cooling unit, and then the liquid methanol goes back to the process through the circular flow.

All tanks are equipped with independent sensors for the control of maximum liquid level that will regulated the operation of transport pumps. Also, the parts of the production facility where methanol vapors may occur must be equipped with sound and light signal devices, which will register its presence at the concentration above the maximum allowed concentration. For this reason, the production facility must be equipped with a proper ventilation system with the independent power generator.

The potential ecological problems could occur during the methanolysis process if the anticipated fluid pressures are exceeded during the transportation using pumps. For the protection, the entire floor in the facility must be built in the form of "impermeable bund" for accidental spills. During pumping the raw materials and the final products from tanker trucks to the reservoirs or vice versa, flexible fittings and special tubes resistant to the transported fluids should be used. Since being insensitive to biodiesel, teflon, viton, nylon, aluminum, stainless steel or fiberglass can be used as materilas for all equipments in the contact with biodiesel [10].

Also, it may happen that a part of the fluid drains from the pipeline when cleaned with compressed air. Therefore, the containers for collection of accidentaly generated wastes should be placed at appropriate places. These wastes will then be disposed (without delay) in a collective container, marked for the corresponding waste material and located in the storage of hazardous waste. In the case of the accidental spilliage of large quantities of hazardous wastes are spilled, it is necessary to use an appropriate absorbent for collecting the waste, which will then be disposed in the storage of hazardous wastes. The quality of water discharged into the sewerage during the process must be in accordance with the Law on Waters [12], the Regulation on hazardous substances in water [13] and the Regulation on the methods and the minimum number of wastewater quality testing [14]. Generally, the pollution of water and the environment during the biodiesel production cannot be expected if the prescribed protective measures are applied.

Product separation and purification

In order to separate the crude methyl ester product from the glycerol-methanol layer, the final reaction mixture is led directly to the gravitational separator (Fig. 1). The crude methyl esters should further be purified in order to fulfill the prescribed standard specification. The main problem is caused by the leaching of calcium from the catalyst bits into the reaction mixture. The use of the method developed by Alba Rubio et al. [15] is recommended, which includes the methyl ester washing by the suspension of Na_2CO_3 in methanol (5% of the ester mass) under reflux, vacuum drying and polishing.

The methyl esters product contains volatile components, such as methanol and water that evaporate during the drying process in the vacuum evaporator. Therefore, the resulted gas phase leavin the evaporator is introduced into a condenser, where the vapour is cooled and condensed.

To achieve the required quality of the final product according to the EN 14214 standard, the methyl esters must be filtered to remove possibly presented fine mechanical impurities. The value must not exceed 24 mg/kg [16]. The filtration process is conducted using the 2.5 μm filter, which is placed on pump discharge line.

Addition of additives to methyl esters

Fatty acid methyl esters (biodiesel) obtained from sunflower oil show better flow properties at lower temperatures compared to those which are obtained from the oily (e.g. palm oil) and faty feedstocks with a high content of saturated fatty acids [17]. However, independently of the origin feedstock, the biodiesel has higher values of cloud and pour point, compared to the conventional diesel fuel [18]. Therefore, the properties of the biodiesel derived from sunflower oil must be improved by adding appropriate additives (e.g. Wintron®). With these additives, biodiesel could be

transported to storage tanks, even in winter conditions. Also, the addition of antioxidant additives can improve the stability of biodiesel to oxidation and prevent/ clean corrosive deposits in the engine with internal combustion.

The addition of additives to the biodiesel is usually carried out in the helicoial static mixer. The amount of additives is defined according to the measured mass flow of the final biodiesel. Delivering of additives is done in containers, which will be sent back to the supplier after emptying, in order to eliminate the possibility of environmental pollution.

Purification of glycerol layer

The glycerol layer, separated from the crude biodiesel, has an amount of methanol, which should be separated by destilation and reused in the process. If necessary, glycerol is further purifiied, depending of its application. The vertical storage tank for glycerol is located out of the production plant. After refining in a separate facility, glycerol is further used as a raw material in the chemical and cosmetic industry.

From an ecological point of view, glycerol is extremely stable in typical storage conditions (cool and dry place without the presence of heat and ignition sources), compatible with many other chemicals, odorless, practically non-toxic and has no known negative impact on the environment. When glycerol is accidentally spilled on the floor, it is necessary to use the appropriate absorbent, which is, after using, disposed in a proper manner [19].

Storage of the final products

In order to maintain the quality of the final biodiesel product as long as possible at the level defined by the standard EN 14214 (max allowed water content 500 ppm), it is essential to ensure a dry atmosphere in the storage tank. To prevent hydrolysis, oxidation and increase of water content, biodiesel must not come in the contact with the ambient air. Such conditions may be achieved with an inert atmosphere under a pressure of the inert gas (nitrogen) in the storage tank. Transport of the both final products from the storage to the market is carried out with tanker trucks. Loading/unloading installations must be equipped with all necessary technical devices for measuring the amount, temperature and pressure, as well as elements for connecting storage tanks and tanker trucks.

MONITORING PROGRAM FOR ENVIRONMENTAL QUALITY MEASUREMENTS IN THE PRODUCTION OF BIODIESEL

For determining the environmental measures during the continuous biodiesel production process in the presence of quicklime, it is needed to identify the emissions of possible pollutants in air, water and soil, as well as noise emissions and generation of hazardous wastes.

Air quality monitoring is performed on the basis of the act of the competent authority for environmental protection (Law on Air Protection [20]) if it is necessary to determine the level of air pollution in a particular area that is not covered by the official.

According to the Regulation on limit values for emissions of **air** pollutants [21], the measurement of pollutants emissions is conducted by using the corresponding measuring instruments at the measuring points and the prescribed method of measurement. The emergence of unorganized emissions is possible during the technological process, storage of hazardous waste, as well as in possible accidental situations.

Monitoring of **groundwater** quality is carried out by an authorized organization under the Regulation on limit values of pollutants in surface and ground waters and sediments, and deadlines for their achieving [22]. In order to prevent pollution of groundwater and soil, a regular control of parameters is needed. Therefore, the the generated amount of wastes is temporarily stored on cement surfaces. Also, in order to ensure the preservation of the recipient quality within the technological process, it is necessary to install the oil and grease separator for purification of wastewater that can be generated during washing the working area and surface runoffs. The storage of hazardous wastes originated from the production process must be an independent covered and fenced place. The floor must be protected by a special coating that is resistant and impervious to chemicals. An ideal floor protection is concrete coated with an AISI 316 Ti stainless sheet (1 mm thick). For the collection an eventually accidental spill of hazardous waste, it is necessary to build a small pit (5x5x5 dm) in the corner of the floor i.e. "impermeable bund".

The programme of systematic monitoring of **soil** quality is prescribed by the Regulation on systematic soil quality monitoring programme, indicators for risk assessment of soil degradation, and methodology for the development of remediation programmes [23]. Monitoring of soil quality is not carried out at the estate level. The individual soil monitoring at specific locations is implemented in accordance with the decision of the authorized inspection bodies related to the environmental protection. It is implemented by accredited institutions. Spillage or leakage of waste, especially hazardous, can occur during the biodiesel production, when it is necessary to take all envisaged measures, which will minimize their impact on the soil. Depending on the amount of spillage material, it is necessary to test the soil and takes the measures for recovery and remediation.

A legal or physical person who is the owner or user of **noise sources** must provide a single measurement of noise in the prescribed manner, make a report about the noise measurement and pay the cost of noise intensity measuring in the impact zone in accordance with the Law on the Protection of Environmental Noise [24]. During regular operation of the complex, it is necessary

to control and maintain the equipments and devices in order to prevent an increase in noise level and to implement engineering control aimed at the staff compliance to operating instructions, as well as at the damping and isolation of noise sources. In the biodiesel production, the noise sources may be pumps, fans for air transport to the rooms and in the external environment, as well as the resonance of brass channels.

According to the current legislation, each type of **hazardous waste** must be stored in a special container that is clearly marked with an identification sheet in accordance with the Law on Waste Management [25]. As a rule, wastes should not be mixed with each other, and each type of waste must be stored separately in a steel vessel intended and marked for the specific waste. The producer must keep a record of the type, quantity, utilization and liquidation of hazardous waste and packaging. The transportation of hazardous waste is provided through an authorized organization for handling of certain types of wastes. The total annual amount of waste must be registered and an annual report must be submited to the appropriate authority for the protection of the environment within the prescribed period.

CONSLUSIONS

In this paper, the continuous biodiesel pilot production via the methanolysis of sunflower oil using quicklime as a low-cost catalyst was analyzed in order to assess its possible impact on the environment. The main goal was to determine the possibility of negative impacts of certain factors and process conditions on the occupational and living environments. The production process is performed in two alternative packed-bed reactors at moderate operating conditions. Regarding to the catalyst, properly manipulation, transportation and storage willt prevent its harmful environmental impact. A possible critical point in the biodiesel production from the environmental point of view is the quicklime storage. However, the application of the prescribed storage conditions excludes the possibility of any accident. In addition, it is necessary to pay special attention to the storage areas of raw materials and products that must be built according to the safety regulations. Sunflower oil and glycerol do not have any harmful impact on the environment. However, methanol represents a potential danger during the process. To exclude the risk of fire and its emission into the air, the storage tank of methanol should additionally be equipped with the devices for maintaining constant overpressure of the nitrogen atmosphere.

The performed analyze of all parameters that may affect the environment quality, led to the conclusion that there will be no negative effects on the ecosystem if all legal measures of protection during the process are respected and applied.

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BIOGRAPHY

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EKOLOŠKI ASPEKT KONTINUALNE PROIZVODNJE BIODIZELA PRIMENOM HETEROGENO-KATALIZOVANE METANOLIZE ULJA

Ninoslava Popović-Nikolić, Marija Miladinović, Ivana Banković-Ilić, Olivera Stamenković, Vlada Veljković

Apstrakt: *U radu je analiziran ekološki aspekt poluindustrijskog kontinualnog postupka za dobijanje biodizela heterogeno-katalizovanom metanolizom suncokretovog ulja primenom negašenog kreča kao katalizatora. Analizom su obuhvaćene sve faze predloženog postupka, a data je i procenu uticaja pojedinih faktora opasnih po okolinu. Cilj rada je bio da se identifikuju potencijalni negativni uticaji proizvodnje biodizela na životnu sredinu i predvide mere zaštite.*

Ključne reči: biodizel, ekološki aspekt, heterogena kataliza, bezbednost.