Sigma Journal of Engineering and Natural Sciences
Web page info: https://sigma.yildiz.edu.tr
DOI: 10.14744/sigma.2023.00007

Research Article

Biodiesel production from biomass by treating textile industry wastewater

Ayşe Elif ATEŞ[1*]

[1]*Department of Environmental Engineering, Istanbul University Cerrahpaşa, Istanbul, 34098, Türkiye*

ARTICLE INFO

Article history
Received: 28 May 2021
Accepted: 09 August 2021

Keywords:
Biodiesel Production; Real Textile Industrial Wastewater Treatment; Energy; Activated Sludge

ABSTRACT

Real textile industry wastewater was treated with the activated sludge process, and biodiesel was produced from activated sludge which was used for the treatment of wastewater. The sequential Batch Reactor was operated at the laboratory scale under ambient conditions that were 25oC. During the treatment, some of the sludge returned to the reactor while the remaining part was harvested and stored at +4°C. To determine the optimum hydraulic residence time in the treatment of textile industry wastewater with the activated sludge process, different hydraulic retention times of 1,2,3,4, and 5 days were compared. In the study, the SBR reactor was operated with and without UV to evaluate the effect of UV on treatment efficiency. While the highest removal efficiency in real textile industry wastewater by activated sludge at HRT-3 was 62% COD and 20% color with UV, the highest removal efficiency was 43% COD and 11% color used without UV. Bio-oil was extracted by Bling and Dyer extraction method from harvested sludge. 82 ml biodiesel and 15 ml glycerine were obtained from 1 L wet activated sludge by the transesterification method.

Cite this article as: Ateş A E. Biodiesel production from biomass by treating textile industry wastewater. Sigma J Eng Nat Sci 2023;41(1):57–63.

INTRODUCTION

Many dyes are toxic to microorganisms and may cause direct destruction of aquatic life due to its high pollutant contents such as nitrogen, phosphorus, many different metals [1]. Dyes, which used to impart color to materials, are dissoluble at some stage of the standard operating procedure in dye production. Some dyes have a synthetic origin, and treatment of synthetic dyes in wastewater is a challenge for researchers because of their complex aromatic molecular structure. Dye production stages include in synthesizing of dyes in a reactor, filtering the synthesized dye and mixing it with many other chemicals. Wastewater of the textile industry using various dyes contains not only many different dyes but also toxic and nonbiodegradable many different additive compounds and their mixtures [2]. Many

***Corresponding author.**
*E-mail address: elifdenizler@istanbul.edu.tr ; ayseelif.ates@iuc.edu.tr

This paper was recommended for publication in revised form by Regional Eyup Debik

Published by Yıldız Technical University Press, İstanbul, Turkey

dyes, 50% of which are used in the textile industries, are produced every year. Significant volumes of water are used during the wet process in the textile industry [3,4]. The dyeing method is one of the most important wet processes in the textile industry and produces wastewater with high chemical oxygen demand (COD). Moreover, textile industry wastewater has a high color content [3]. The discharge of dyes into water source decreases photosynthetic activity, while the increases biological oxygen demand (BOD) and COD levels and change the pH value. The dyes used in the dyeing process are estimated to be existed in textile industry wastewater by about 15% [5]. 70% of the dyes used in textiles are azo dyes consisting of one or more azo bonds (N==N—) connecting aromatic amines [4,6]. Since azo dyes make it difficult to treat industrial wastewater containing dyes, treatment studies are carried out with advanced oxidation methods [7-9].

Treatment system for textile wastewater, generally consist of neutralization, settling tanks, activated carbon adsorption, flocculation-coagulation (generally with iron chloride), biological treatment (aerobic or anaerobic), and oxidation of organics by with UV systems or hydrogen peroxide (H_2O_2) [2]. These methods' main drawbacks are high costs and large quantities of sludge generated. On the contrary, biological therapy is cheaper and eco-friendly [6,10]. If the wastewater of the textile industry is to be treated with a process using light, it should be ensured that the light affects the large area because dyes remaining in wastewater absorb light with wavelengths in the visible region between 350–700 nm [11]. For this reason, in biological treatment studies, UV is selected suitable for the system to increase the treatment efficiency. The complex structure of chemicals and the lack of ability of microorganisms to degrade chemicals in textile wastewater treatment reduce the efficiency of wastewater treatment with biological methods.

Biodiesel, as green and environmental-compatible biomass energy, has become quite interesting in recent years. In recent studies, biodiesel production is provided by lipids obtained from waste cooking oil, non-edible seed or biological sludge [12-14]. In recent studies, it has been investigated whether the biomass obtained after biological treatment is an energy source. The key subjects of sustainable society growth are actually energy and environmental conservation. During the method of biological wastewater treatment, biological wastewater systems are used to form microbial communities with varying structures and volumes. The enriched particular microbial population is ultimately precipitated to generate the surplus secondary sludge, and the variation in the sludge microbial culture would presumably impact the fat and oil removed [15].

In this study, biodiesel production trials were carried out from sludge obtained from the textile industry wastewater treatment reactor. In the literature research, it was seen that the effects of UV use on the COD and color removal efficiency in the sequential batch reactor were limited. The effect of UV decomposition of organic dye on the treatment efficiency, when combined with the activated sludge system, was investigated. In addition, biodiesel production from activated sludge was obtained from domestic wastewater treatment plants, and biodiesel production from sludge obtained as a result of the treatment of textile industry wastewater is not found in the literature.

Providing high treatment efficiency or increasing the treatment by modifying the system can encourage water reuse for the textile industry, which has intensive water use. With the system used in the study, energy production with wastewater treatment has advantages such as reduced cost in the industry and the use of biodiesel reduces the formation of emissions that cause air pollution.

MATERIALS AND METHOD

Characteristic of The Raw Textile Industry Wastewater

Textile industry wastewater (TIW), which has high turbidity, COD, and color, is a mixture of wastewaters from processes such as mercerization, desizing, dyeing. Although the real textile industry wastewater characterization was similar to textile industry wastewater characterizations in the literature, its COD and color values were high [16]. The real textile industry wastewater (RTIW) characterization used in the study was given in Table 1. For the determination of wastewater characterization, pH, COD, color, conductivity and suspended solids determinations were made in the laboratory, while the temperature value was used in the balance tank of the industry. When the wastewater was brought to the laboratory, its temperature was between 18-23°C.

COD results were measured using the closed reflux method, while color analysis was performed using the spectrophotometric method. COD and color removal efficiencies were calculated using following formula:

$$RE(\%) = \left(\frac{C_0 - C}{C_0} \right) \times 100$$

RE; removal efficiency (%)
C0; influent concentration ($mg.L^{-1}$)
C; effluent concentration ($mg.L^{-1}$)

Activated Sludge Process

The active sludge used in the study was obtained from the Ataköy Advanced Biological Treatment Plant. Activated

Table 1. Characterization of the RTIW in the study

Parameter	Avarage Value	Parameter	Avarage Value
pH	8	**Color** (Pt-Co)	2900
COD ($mg.L^{-1}$)	6500	**Conductivity** (ms/cm)	20

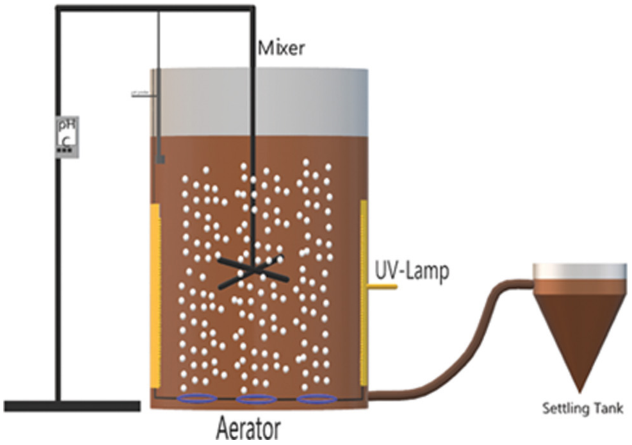

Figure 1. Activated sludge process.

Figure 2. Phase separation after extraction.

sludge system was operated as Sequential Batch Reactor (SBR). Designed for activated sludge system plexiglass column volume was 20 L as shown in Figure 1. Ventilation was provided by 4 air pumps which capacity 400 $L.h^{-1}$ and worked continuously. Since the color of the RTIW was pitch black, adjacent lamps were placed around the reactor to provide more light. The UV lamp used in the study was a wavelength of 430 nm and 25 volts. In the pre-studies, experiments were carried out without using UV and at different UV-light durations. In different light trials, 24 hours of light, 16 hours of light - 8 hours of darkness, and 8 hours of light and 16 hours of darkness were conducted. The highest efficiency was obtained as a result of 8 hours of light-16 hours of dark, while the lowest efficiency was obtained as a result of 24 hours of light operation. The lighting in the system was set to 8 hours light and 16 hours dark. The pH of raw wastewater was approximately 8 and the system was fed without changing the pH in the reactor. Textile industry wastewater flow rates were adjusted to have five different hydraulic retention time (HRT) of 1,2, 3,4 and 5 days.

The samples taken from the reactor were kept at 25 °C for 1 hour to settle. While the upper phase was used for COD and color analysis, the obtained sludge was used in the production of biodiesel. The temperature was continuously monitored and kept between 18-22°C to prevent UV lighting from changing the temperature of wastewater. By using the cooling fan, both the lamp and the reactor heating caused by the lamp were prevented.

Extraction Method

Bling and Dyer extraction method, one of the wet extraction methods, was used for activated sludge extraction [17]. The sludge harvested before the wastewater feed was stored at +4°C. No treatment was done for the dewatering of the stored sludge, only settling was applied and the sludge was extracted with its own water. To increase the large-scale production efficiency of biodiesel, pre-extraction experiments were performed. 50 ml of wet sludge was placed in the volumetric flask. 62.5 ml of chloroform and 125 ml of methanol were added to the sample and stirred at 600 rpm for 10 min. Then, 62.5 ml of chloroform was added again and mixed at 600 rpm and 10 min. 62.5 ml of water was added cautiously to allow the phase separation and waited until phase separation was realized. The density of the chloroform is 1.49 g/cm^3 and the density of the chloroform oil mixture after extraction is 1.29 g/cm^3. The residue by the decomposition of the activated sludge with solvent is in the middle phase and the water separates the other residues to the upper water mixture phase, as shown in Figure 2. The solvent-oil mixture was separated with a separating funnel and the solvent was evaporated.

Transesterification

Transesterification reaction pre-experiments were carried out with waste cooking oil because small scale oil would be obtained from activated sludge. 10 ml of oil and 30% by volume of methanol and 1% by weight of NaOH were added in the volumetric flask. The flask was closed that not to be open due to the pressure during the reaction as shown in Figure 3 and the sample was kept at 65°C for 1.5 hours. The produced biodiesel was washed 3 times with distilled water to remove glycerin and methanol residues and dried in 100°C oven for 1 hour.

RESULTS AND DISCUSSION

There was no pre-treatment for RTIW before feeding to the activated sludge process. The upper phase of the wastewater harvested from the SBR during daily wastewater feeding was used for treatment efficiency. Biomass exchange in wastewater at different HRT was investigated, shown in Figure 4. During the 3 days of wastewater feeding, there was an increase in the biomass of the activated sludge.

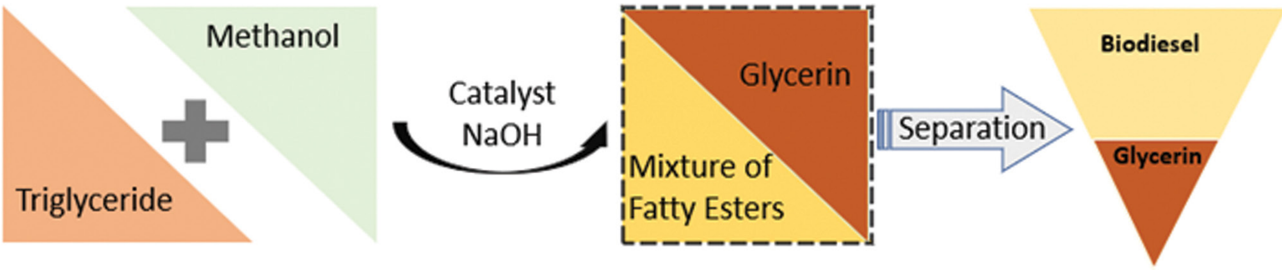

Figure 3. Transesterification reaction.

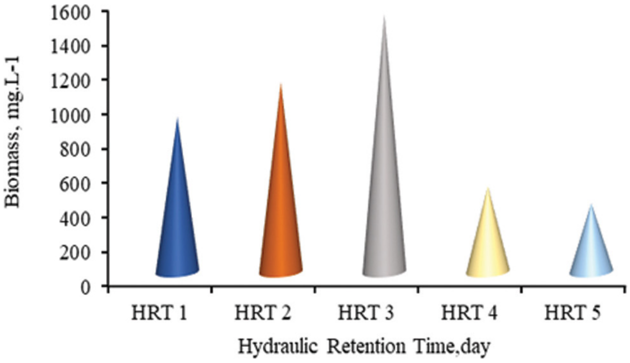

Figure 4. Biomass exchange.

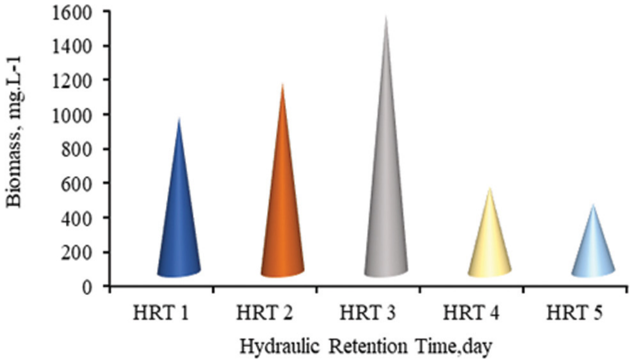

Figure 6. COD removal efficiency without UV.

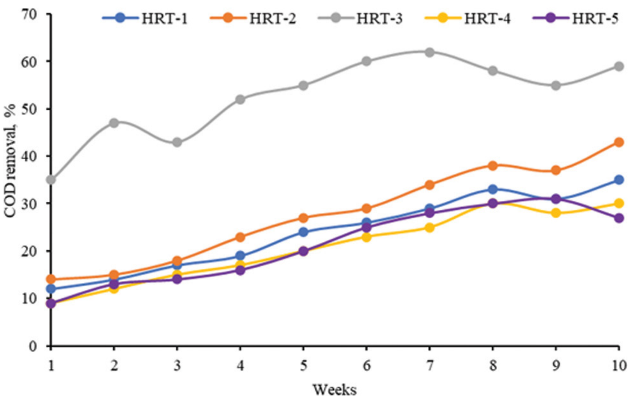

Figure 5. COD removal efficiency with UV.

However, after the 3rd day, the rate of biomass decreased to approximately 400 mg.L^{-1}.

Despite the increase in treatment efficiency in the first weeks, COD treatment efficiency remained constant after the sixth week, shown in Figure 5. The study was completed after 10 weeks because there was no significant increase in treatment efficiency. Due to the high pollution value of RTIW, it should be pre-treated by coagulation or advanced oxidation before being given to biological treatment.

In the study, studies were carried out with and without UV for COD and color removal efficiency from RTIW with SBR. Among both systems (UV and non-UV), the highest treatment efficiency was achieved in HRT-3, and the COD removal efficiency was 62% in the UV-SBR system and 41% in the non-UV-SBR system, shown in Figure 5 and Figure 6. Due to the high COD in the wastewater, the removal efficiency of the textile industry wastewater was decreased with the activated sludge method. Although the COD concentration of the wastewater used in the study was 6500 mg.L^{-1} and the highest COD removal efficiency was obtained as 62%, in another study with a COD concentration of 3060 mg.L^{-1}, the removal efficiency was 91% in HRT-3[18]. In this study, the lowest removal efficiency was obtained with the HRT-5 (with UV and non-UV). The establishment of a pilot-scale treatment plant with a grid, pH balancing tank, and oxidation ditch provided higher yields [18] compared to the laboratory scale study. In the study, the use of UV in the SBR system increased the COD removal efficiency by approximately 20%. While the use of UV did not harm the activated sludge, oxidation increased both color and COD removal. In the treatment of textile industry wastewater with activated sludge, the treatment efficiency increases with the increase in working time (days, weeks, etc.). In the study, the highest removal efficiency was obtained in the ninth and tenth weeks.

The textile industry wastewater used in the study had intense color content. While the color removal efficiency in 1-5 weeks was quite low, it was increased in other weeks. However, the highest color removal efficiency was 20%.

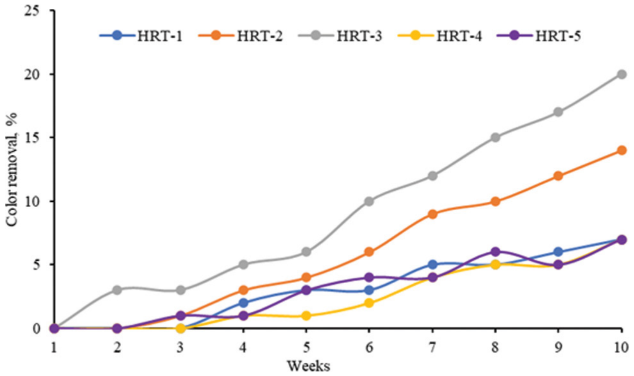

Figure 7. Color removal efficiency with UV.

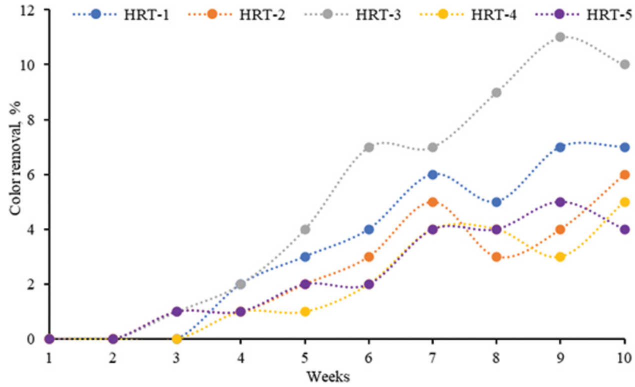

Figure 8. Color removal efficiency without UV.

Table 2. COD and color values after treatment (at 10 weeks)

	COD, mg.L^{-1} (with UV)	COD, mg.L^{-1} (without UV)	Color, Pt-Co (with UV)	Color, Pt-Co (without UV)
HRT-1	4225	4949	2697	2696
HRT-2	3705	4810	2494	2726
HRT-3	2665	3835	2320	2610
HRT-4	4550	5070	2696	2755
HRT-5	4745	5135	2698	2784

Table 3. Europe EN 14214 Biodiesel Quality Standards and Produced Biodiesel Quality [20]

Properties	Europe EN 14214	Produced Biodiesel	Methods
Viscosity, 40° (mm^2/sec)	3.5-5.0	4.2	Optical viscometer
Density(gr/cm^3)	0.86-0.90	0.87	Pycnometer
Flash Point (°C)	120	122	Closed-cup method
Methanol (%m/m)	0.20 max	0.19	Headspace-Gas Chromatography-Flame Ionization Detection

Color removal with activated sludge is achieved by biodegradation. In the study, the highest color removal efficiency is 10% in the system without UV and 20% in the system with UV. Color removal efficiency is at its highest in the ninth and tenth weeks, similar to the COD removal efficiency, shown in Figure 7 and Figure 8. The efficiency in color removal studies from textile industry wastewater with activated sludge is between 0-40%. To increase the color removal efficiency, studies were carried out by adding materials such as polyaluminium chloride (PAC), bentonite, and active clay to the activated sludge system, and the purification efficiency was increased to approximately 70-80% [19].

Biodiesel production was produced only from the sludge obtained from the HRT-3 system. The solvent extraction was carried out after the system balancing and adapting of the sludge. 5 ml of biodiesel and 6 ml of glycerin were obtained from a 50 ml wet sample in pre-trials of biodiesel production. The optimum NaOH and methanol were determined in trials for large scale sample. Finally, after the optimizations of chemicals, 82 ml biodiesel and 15 ml glycerine were obtained from 1 L wet sludge. Quality standards of biodiesel produced are compared to European Standards as shown in Table 3.

In a different study on biodiesel production from activated sludge, different industrial wastewaters were used and the effect of wastewater types on biodiesel production was investigated. Lipid yield from sludge obtained during biological treatment of restaurant wastewater is 40.33 mg/g sludge while the lipid ratio obtained from the municipal wastewater sludge is 28 mg/g sludge [21]. In the study, while lipid ratio was 70 mg/g activated sludge at HRT-5, the highest lipid ratio was 80 mg/g activated sludge at HRT-3. The high oil content of restaurant wastewater was affected the active sludge and decreased the lipid content. In the use of municipal wastewater, the lipid content of activated sludge has decreased due to the pharmaceuticals and toxic substances in the wastewater. Because textile industry wastewater contains domestic wastewater from the factory as well as process water, the lipid ratio is higher than the other wastewater. It was estimated that the continuous aeration in the study and the presence of UV in the system has a positive effect on the activated sludge by providing the treatment of chemicals that may cause toxic effects on the activated sludge.

CONCLUSION

In the study, biodiesel production was carried out from activated sludge and benefit was provided both in terms of treatment and energy. Real textile industry wastewater was treated with activated sludge method. In the study, the most suitable hydraulic retention time was determined as 3 days. In the SBR system, the COD removal efficiency increased by approximately 20% with 8 hours of light and 16 hours of dark with UV. However, the color removal efficiency was the highest 20%. The use of UV increased both COD and color removal efficiency. 82 ml of biodiesel was produced from 1 liter of wet activated sludge.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

REFERENCES

[1] Mahmoud AS, Ghaly AE, Brooks MS. Removal of dye from textile wastewater using plant oils under different pH and temperature conditions. Am J Environ Sci 2007;3:205–218. [CrossRef]

[2] Shindhal T, Rakholiya P, Varjani S, Pandey A, Ngo HH, Guo W, et al. A critical review on advances in the practices and perspectives for the treatment of dye industry wastewater. Bioengineered 2021;12:70–87. [CrossRef]

[3] Peláez-Cid AA, Romero-Hernández V, Herrera-González AM, Bautista-Hernández A, Coreño-Alonso O. Synthesis of activated carbons from black sapote seeds, characterization and application in the elimination of heavy metals and textile dyes. Chin J Chem Eng 2020;28:613–623. [CrossRef]

[4] Popli S, Patel UD. Destruction of azo dyes by anaerobic-aerobic sequential biological treatment: A review. Int J Environ Sci Technol 2015;12:405–420. [CrossRef]

[5] Hai FI, Yamamoto K, Fukushi K. Hybrid treatment systems for dye wastewater. Crit Rev Environ Sci Technol 2007;37:315–377. [CrossRef]

[6] Bonakdarpour B, Vyrides I, Stuckey DC. Comparison of the performance of one stage and two stage sequential anaerobic–aerobic biological processes for the treatment of reactive-azo-dye-containing synthetic wastewaters. Int Biodeterior Biodegradation 2011;65:591–599. [CrossRef]

[7] Muniyasamy A, Sivaporul G, Gopinath A, Lakshmanan R, Altaee A, Achary A, et al. Process development for the degradation of textile azo dyes (mono-, di-, poly-) by advanced oxidation process-Ozonation: Experimental & partial derivative modelling approach. J. Environ Manage 2020;265:110397. [CrossRef]

[8] Hussain SM, Hussain T, Faryad M, Ali Q, Ali S, Rizwan M et al. Emerging aspects of photo-catalysts (TiO2 & ZnO) doped zeolites and advanced oxidation processes for degradation of azo dyes: A review. Curr Anal Chem 2021;17:82–97. [CrossRef]

[9] Alderete BL, da Silva J, Godoi R, da Silva FR, Taffarel SR, da Silva LP, et al. Evaluation of toxicity and mutagenicity of a synthetic effluent containing azo dye after advanced oxidation process treatment. Chemosphere 2021;263:128291. [CrossRef]

[10] Palma C, Carvajal A, Vásquez C, Contreras E. Wastewater treatment for removal of recalcitrant compounds: A hybrid process for decolorization and biodegradation of dyes. Chin J Chem Eng 2011;19:621–625.

[11] Udaiyappan AFM, Abu Hasan H, Takriff MS, Abdullah SRS. A review of the potentials, challenges and current status of microalgae biomass applications in industrial wastewater treatment. J Water Process Eng 2017;20:8–21. [CrossRef]

[12] Sumprasit N, Wagle N, Glanpracha N, Annachhatre AP. Biodiesel and biogas recovery from Spirulina platensis. Int Biodeterior Biodegradation 2017;119:196–204. [CrossRef]

[13] Hotti S, Hebbal O. Biodiesel production and fuel properties from non-edible Champaca (Michelia Champaca) seed oil for use in diesel engine, J Therm Eng 2015;1:330–336. [CrossRef]

[14] Kolakoti A, Mosa PR, Kotaru TG, Mahapatro

M. Optimization of biodiesel production from waste cooking sunflower oil by taguchi and ann techniques. J Therm Eng 2020;6:712–723. [CrossRef]

[15] Mondala A, Liang K, Toghiani H, Hernandez R, French T. Biodiesel production by in situ transesterification of municipal primary and secondary sludges. Bioresour Technol 2009;100:1203–1210. [CrossRef]

[16] Blanco J, Torrades F, De la Varga M, García-Montaño J. Fenton and biological-Fenton coupled processes for textile wastewater treatment and reuse. Desalination 2012;286:394–399. [CrossRef]

[17] Bligh EG, Dyer WJ. A rapid method of total lipid extraction and purification. Can J Biochem Physiol 1959;37:911–917. [CrossRef]

[18] Haddad M, Abid S, Hamdi M, Bouallagui H. Reduction of adsorbed dyes content in the discharged sludge coming from an industrial textile wastewater treatment plant using aerobic activated sludge process. J Environ Manage 2018;223:936–946. [CrossRef]

[19] Pala A, Tokat E. Color removal from cotton textile industry wastewater in an activated sludge system with various additives. Water Res 2002;36:2920–2925. [CrossRef]

[20] ACEA. Biodiesel Guidelines from the Worldwide Fuel Charter Committee. 2009. www.acea.be Accessed on Jul 1, 2019.

[21] Chi X, Li A, Li M, Ma L, Tang Y, Hu B, et al. Influent characteristics affect biodiesel production from waste sludge in biological wastewater treatment systems. Int Biodeterior Biodegradation 2018;132:226–235. [CrossRef]