Solvent Extraction as Decontamination Process of Oily Soil in Indonesia

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Abstract. Even though solvent extraction has been known for long time to various of treatment process industry, at soil decontamination area as this process also known as soil washing this treatment process should be studied for further for large scale possibility of implementation. This study described the development of solvent extraction method for treatment some various contaminated soil in Indonesia. Selection of solvents has been developed based on previous research in the earlier stage then continued with optimizing parameters that might give good synergy on removal efficiency as such temperature, moisture, and soil to solvent ratio. Total Petroleum Hydrocarbon (TPH) became main focus on this study, however some other aspects such as metal and heavy metal content using TCLP test were also studied to see its possibility for safely discharged to the environment or having further treatment. Observation statistics were also conducted to see the importance of key parameters during treatment process. Results showed that different characteristics of oily soil might give various TPH removal rate, however key parameters that are significant are original TPH concentration, temperature selection, moisture content, and solid to solvent ratio.

Keywords: *oily soil waste;msolvent extraction; biosurfactant; TPH removal; soil washing*

1 Introduction

Study for seeking methods to treat oily soil waste involving some researchers having intention to capable of removing oil as maximum as possible from the solid matrix. The higher removal that obtained might able to make to soil residue to be discharged to the environment, while the oil content that has been recovered can be utilized as synthetic fuel or any other power source utilities. Solvent extraction is a method that has been implemented widely in industry. Some solvents reported has been utilized to treat the oil sludge. The solvent extraction process is essentially is an adsorption-desorption process of oil between the solid and liquid phases [2]. Biceroglu has been used intermediate hydrocarbon source to be able to extract oil sludge from refinery storage to become lighter hydrocarbonate at several stages of temperature. Temperature that has been selected was from 20ºC to 50ºC [3].

Solvent extraction that utilized Isopar-L (an isoparafin mixture) has been conducted by Trowbridge and Holcombe [4] with the comparison of solvent composition solvent to sludge is 10:1 respectively. Temperature that has been selected is 93ºC for 2 hours. It was reported that 99% of oil has been recovered and residue of solids has been able to discharge to the environment base on USEPA standard. However, heavy metal that contained at solid residue make the sludge can't be declared as non-hazardous waste material. DeFillippi and Markiewicz [5] use propane for conduct extraction process to the oil sludge assisted by moderate pressure. In 2007 Avila-Chavez et.al [6] utilized supercritical ethane and dichloromethane to extract hydrocarbon from oil sludge by using Soxhlet extraction. It was reported that 16%-55% oil has been recovered. Zubaidy and Abouelnasr [7] studied comparative performance of some solvents to extract oil from sludge in room temperature. In the beginning Methyl Ethyl Ketone (MEK) and LPG *condensate* (LPGC) has been utilized with comparison 1:1 until 6:1 between solvent and oil sludge. Solvents has been chosen due to its common availability in every oil and gas refinery plant. LPGC is a product from refinery plant and MEK is chemical that normally used for de-asphalting heavy oil process. Additional solvents that has been studied were heptane, hexane, iso-propanol, and iso-butanol. Oil recovery reported varies from 8% for iso-butanol and 39% for MEK. Generally, MEK contribute highest efficiency and LPGC contribute efficiency that close to MEK. It was also reported that usage of hexane and alcohol produce synthetic fuel that contain high ash content, carbon resider, and asphaltene.

Gazieu, *et al.* [8] reported that usage of turpentine as a solvent has been able to extract 13-53% oil from original mass of oil sludge. Zubaidy and Abuelnasr [7] made comparative study related to the effect of some organic solvents such as methyl ethyl ketone (MEK) dan liquefied petroleum gas condensate (LPGC). They observed that the most efficient comparison between MK and Sludge 4:1. By implementing that, the highest oil recovery that can be reached was 39% for MEK and 32% for LPGC. It was also reported that the usage of MEK has been also able to separate most of ashes, asphaltene, and some other contaminants. Nevertheless, it was found that high sulphur content presence in the carbon residue of recovered oil. Solvent extraction basically conducted with pouring oil sludge with some solvents with certain proportion to separate water, solids, and extracted solvents. The extracted solvents would then to be sent to distillation unit for separating oil and solvent [9]. Further process is required to purify recovered oil to be fuel. El Naggar et. al [10] studied the usage of some solvents such as naphthalene, kerosene cut, n-heptane, toluene and some other solvents. From several experiments, it was reported that toluene has the highest extraction efficiency, which is 75, 94%. Usage of oil solvents that have rings such as (naphtalenics and aromatics) for example catalytic cracking oil has highest efficiency in solving asphaltenics compound in oil sludge. Meanwhile, the solvent that has paraffinic content such as diesel paraffinic found effective to extract oil sludge that has high content of paraphinic [11]. Taiwo [12] also reported that the usage of Hexane and Xylene can also utilized to extract oil sludge. From the experiment, it was found that 67.5% PHC(s) was able to be extracted.

Solvent extraction process has been conducted by mixing solvent and oil sludge in a reactor with certain retention time so that solvent and oil sludge mixed homogenously and then settled for certain time to make sludge residue settled in the bottom [1]. The mixture is then to be conveyed to solvent distillation reactor. During this process solvent is evaporated and the to be condensed all the way the piping system toward to returned back to mixing reactor of solvent and oil sludge. The residue of the process periodically will be input to distillation reactor that would also purify solvent that still available at the solvent.

There are some studies reporting addition of surfactant to solvent extraction process. Rhamnolipid, Triton X-100, and Triton X-114 are surfactants that has been used during co- solvent extraction process had the highest oil recovery rates (40 – 70%). It was reported that the surfactant concentration is independent to the oil removal rate [13] and the addition of biosurfactant into solvent extraction process can increase 7% from toluene single stage performance by using solvent extraction process [14].

Evaluation the toxicity of oily soil waste residuals from solvent extraction process (washing) four types of oil sludges with five surfactants (Triton X-100 and X-114, Tween 80, sodium dodecyl sulphate-SDS and rhamnolipid) and a co-solvent (cyclohexane) resulted that toxicity index of the 10, 25 and 50% rhamnolipid-oily solid waste having soil residue significantly lower compare to residue from Triton X-100. Ryegrass germination rates were higher than 70% with no apparent phytotoxicity symptoms in the seedlings. These indications gave opportunity for further implementation of biotreatment techniques to treat the oily soil waste residuals [15].

Bao, *et.al* [16] reported a suited binary bio-surfactant mixing system was selected as the washing agents to treat the oily sludge produced from Huabei oilfield by the thermal bio-surfactant washing method. The results showed that in case of the mass ratios of 8:2 the critical micelle concentration (CMC) was dramatically decreased and synergism was the strongest in micellar mole fractions (LT and SL) at bi- mixed surfactant systems. The studied binary mixed bio-surfactant system showed higher washing efficiency for oily sludge than single surfactant system. It was also reported that washing abilities of the selected surfactants not only depend on their mixing ratio and washing conditions but also associate with microstructure and mineral components of oily sludge. This study would give results of some optimizations process, result, and statistical analysis regarding solvent extraction process of several contaminated soil form some places over Indonesia.

2 Material and Method

Selected solvents during the experiments were utilized based on some reference from previous studies of solvent extraction process or also called with soil washing as summarized at the following table.

Table 1 Solvent Extraction Research for Oily Contaminated Solid Waste [14].

Study	Solvent	Oil Content Removal
Gazineu et.al [8]	Turpentine	13-53% oil from original oil sludge mass.
Taiwoo et al [12]	Hexane and Xylene	67.50%
Zubaidy and Abuelnasr [7]	Methyl ethyl ketone (MEK) and liquefied petroleum gas condensate (LPGC).	39% for MEK and 32% for LPGC
El Naggar et al [10]	Toluene	75.94%
Hu et al. [17]	MEK and Ethyl Acetate (EA)	40% for MEK and 60% for EA

Toluene, Acetone and Ethanol that have been selected for the experiments produced by Merck, while Tween 80 is produced by local chemical producer and biosurfactant was produced by Bioscience and Biotechnology laboratory at Institute Technology of Bandung. Burkholderia sp._PAU02 as variance that has been selected capable to reduce surface tension to the point of 47.0 dyne/cm and emulsification index EP24 of 84%.

Solvent extraction process and analysis of parameters has been conducted at ITB and PPLi laboratory at Cileungsi, Bogor. Contaminated soil has been transported from contaminated areas to be prepared accordingly to its homogenous form and be ready to for further treatment. Original sample has variance of TPH concentration that will be elaborated in each section of method and result of studies.

2.1 Optimization of Mixing for Solvent Extraction Process

Optimization has been conducted to seek better mixing approach for conducting extraction process. During this experiment, ethanol has been selected as solvent to observe optimization of mixing process. Range of contaminated soil that has been selected to be examined were around 10.93%-34.91%. Horizontal shaking and centrifugation have been selected as mechanism of mixing that would provide better efficiency for extraction process. Four types of contaminated soil with each of its characteristics has been examined with horizontal shaker in the speed of 150 rpm for two minutes and centrifugation 1570 g for ten minutes, while ethanol was pro-analisi solvent that has been selected during this optimization process. This result would give baseline information to conduct treatment process for ethanol itself and other solvents that examined in this experiment. The following table would give characteristics of each contaminated soil for mixing optimization study.

Table 2 Waste Characteristics of Mixing Optimization of Solvent Extraction Process.

Sample	pH	Moisture (%)	TPH (%)
1	10.03	2.8	16.52
2	12.64	12.59	25.52
3	6.11	0.55	10.93
4	6.78	13.61	34.91

2.2 Optimization of Temperature for Solvent Extraction Process

Toluene, ethanol, tween 80, and rhamnolipid biosurfactant selected to examine temperature influence to TPH removal rate of solvent extraction process for oily soil. Original TPH of contaminated soil was 6.5 %. Temperatures selected were 24°C, 50°C and 70°C and the application of those three types of temperatures we implemented during centrifugation process.

II.3 Optimization of Soil to Solvent for Solvent Extraction Process and Dependency to Dissociation Coefficient

In this experiment toluene, ethanol, tween 80, and rhamnolipid biosurfactant were also selected to examine effect of soil to solvent ratio for TPH removal rate of solvent extraction process. Ratio of soil to solvent were selected between 1:1 until 1:4. This ratio considered as reasonable ratio to make it implementable for larger scale of implementation for soil decontamination by using this method.

2.3 Emulsification Index Dependency for Solvent Extraction using Statistics Method

Total 1000 grams of soil sample that has original TPH 6.5 % were divided into 16 types of soil that would have different series of treatment as suggested by Taguchi Orthogonal method to obtain optimum treatment based on observed experimental parameters. Moisture content were adjusted by adding certain volume of distilled water into oily soil and being stirred to ensure it would be homogenous. For each series of treatment, 20 grams of contaminated soil were allocated to have 3 main experimental observation factor such as type of selected solvent, soil to solvent comparison, and moisture content. Fitriyani et. al in 2021 were observing some experimental parameters and assessing it based on statistics those three parameters gave significant impact on TPH removal.

There are 16 series of treatment involved horizontal shaking for 2 minutes and continued with 10 minutes of centrifugation. Toluene and ethanol were selected to be observed as synthetic solvents. Tween 80 as synthetic surfactant and rhamnolipid biosurfactant from isolated Bulkholderia-sp were also involved in this treatment process. The extract all together being vacuum filtered by using vacuum filtration so that both liquid and solid separated and both would be checked its characteristics. Solids were analyzed for its TPH content, metal oxide content by using XRF instrument, hydrocarbon compound qualitatively analyzed by using GC-FID, TCLP test for checking its metal and heavy metal content. Liquid were tested for its possibility for solvent recovery. The following **table 3**. is tabulation of observed parameters and treatment.

Table 3 Solvent Extraction Optimization Method Parameters of Experiment.

No	Type of Solvent	Soil to Solvent Ratio	Moisture Content (%)
1	Toluene	1:1	3.39
2	Ethanol	1:2	6.67
3	Tween 80	1:3	13.15
4	Biosurfactant	1:4	16.62

Taguchi Orthogonal Method of Experiment

Sample Number	Type of Solvent	Soil to Solvent Ratio	Moisture Content
1	1	1	1
2	2	1	2
3	3	1	3
4	4	1	4
5	1	2	2
6	2	2	1
7	3	2	4
8	4	2	3
9	1	3	3
10	2	3	4
11	3	3	1
12	4	3	2
13	1	4	4
14	2	4	3
15	3	4	2
16	4	4	1

TPH content was analyzed referring to USEPA 8440 method about total recoverable petroleum hydrocarbons by using infrared spectrophotometry, while metal elements for solid were measured using inductively Coupled Plasma (ICP) analysis base on USEPA 3050 B Method by conducting acid digestion method using HNO_3 and H_2O_2. Solvent extraction method conducted by running horizontal shaker equal to 150 rpm for 15 min then continued by centrifugation SETA Oil Test Centrifuge at 1,000 rpm (1,570 g) for 10 min duration. Extract of aliquot and solid was separated by using vacuum filtration method using Whatman series CAT-1825-047 that has 47 mm of diameter.

2.4 TPH Removal and Dissociation Coefficient

TPH removal (Ro) from the oily contaminated soil and the oil concentration in the solvent (Co) were calculated as mentioned in the following Eq. 1 and Eq. 2 (Fitriyani et al 2021):

$$\mathbf{Ro} = ((\Gamma\mathbf{0} - \Gamma\mathbf{10}))/(\Gamma\mathbf{0}) \quad (1)$$

$$\mathbf{Co} = ((\Gamma\mathbf{0} - \Gamma\mathbf{10}))/(\mathbf{m/V}) \quad (2)$$

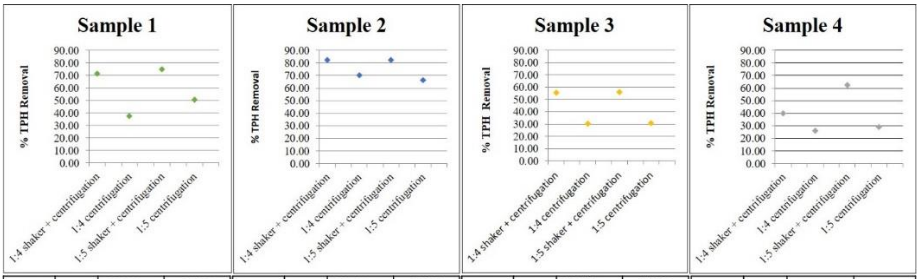
Where Γ_0 and Γ_{10} are the initial and residue oil contents that measured as Total Petroleum Hydrocarbon (TPH) in the contaminated soil (g/g), respectively, m is the mass of soil (g) and V is the solvent volume (mL). TPH removal and removal rate is percentage of Ro that obtained by multiplying it to 100%.

3 Result

Results are discussed as follow related to the optimization process, temperature and solid to solvent ratio influence for solvent extraction process, possibility of biosurfactant usage for single up to multiple stage of extraction, opportunity of recycling. Qualitative analysis using GIC-FID for identifying hydrocarbon composition, analysis of metal and oxide metal of soil by using XRF analyzer and TCLP test result for metal leaching analysis at the residual soil waste that has been treated using solvent extraction.

3.1 Soil to Solvent Ratio and Mixing Method Optimization Treatment Process

The following figure 1. gave description that using centrifugation as mechanism to stir oily soil in ethanol extraction process allowed TPH to be removed in the range between 25-70%, however additional process of horizontal shaker prior would be able to increase the removal until more than 80%. In average 23 % of higher removal rate of TPH gained for horizontal shaker mixing that conducted prior centrifugation process.



Sample	pH	Moisture %	TPH %
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Figure 1 Mixing and Solid to Solvent Optimization.

The observation result found that combination between horizontal shaking and centrifugation provided better performance of TPH removal. Therefore, for the followed solvent extractions process at this study conducted by combining both two mixing processes include its duration.

3.2 Temperature Optimization of Solvent Extraction Process

Toluene, ethanol, tween 80, and biosurfactants has been utilized during this optimization process of solvent extraction. Indication for dependency to temperature showed for toluene and tween 80. However, for ethanol and biosurfactant there are no significant impact of increasing temperature to TPH removal that occurred on process. At the dependency solvent to temperature, toluene and tween-80 showing better performance at temperature 50°C, observed that the removal rate has not significantly increased for additional 20°C into 50°C solvent extraction system.

In the other hand in case of ethanol and biosurfactant there are no significant impact on adding temperature from 24°C up to 50°C. While put temperature higher to 70°C did not give better performance compare to what the system has for lower temperature.

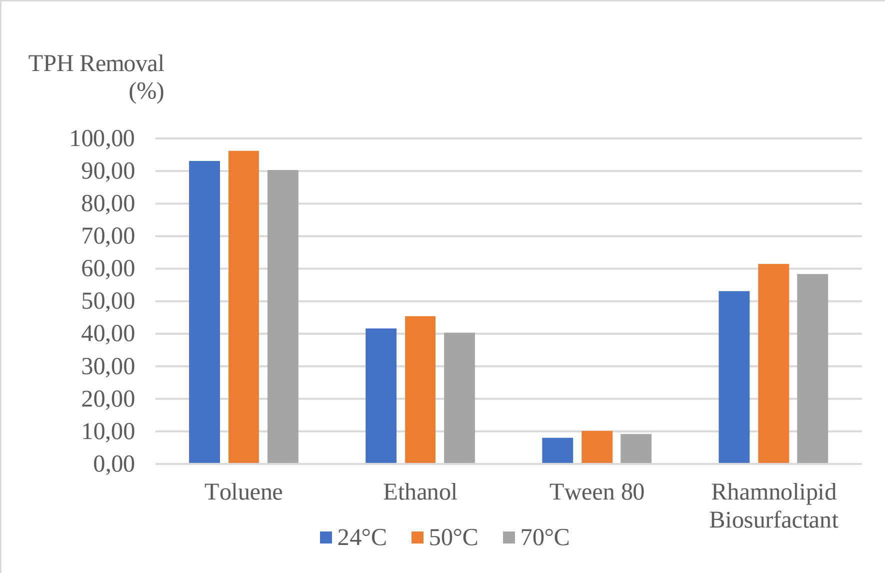

Figure 2 TPH Removal on Soil Residue of Different Temperatures for Solvent Extraction Process.

This phenomenon gave guideline to conduct the upcoming solvent extraction process at room temperature that might give a chance for lower energy utilization for further larger implementation of solvent extraction for decontamination of oily soil.

3.3 Solid to Solvent Optimization of Solvent Extraction Process

Solid to solvent ratio had been one of factor that presumptuous that having high significance to process efficiency in solvent extraction process. Therefore, the following experiment would like to observe it would be dependent to removal rate of TPH of contaminated soil. At the following figure, it can be seen that to solvent indicated dependency to TPH removal for both toluene and toluene. Linear dependency with the increasing ratio of solvent create additional TPH removal efficiency in average 2 %. At the previous research, Fitriyani et,al in 2019 reported the consistency of solid to solvent dependency to TPH removal by using toluene and acetone as solvent despite of the difference of contaminated soil. Seeing the occurrence to tween 80 and rhamnolipid biosurfactant, it was found there are no significant changing of TPH removal by implementing higher volume of both into the solvent extraction process.

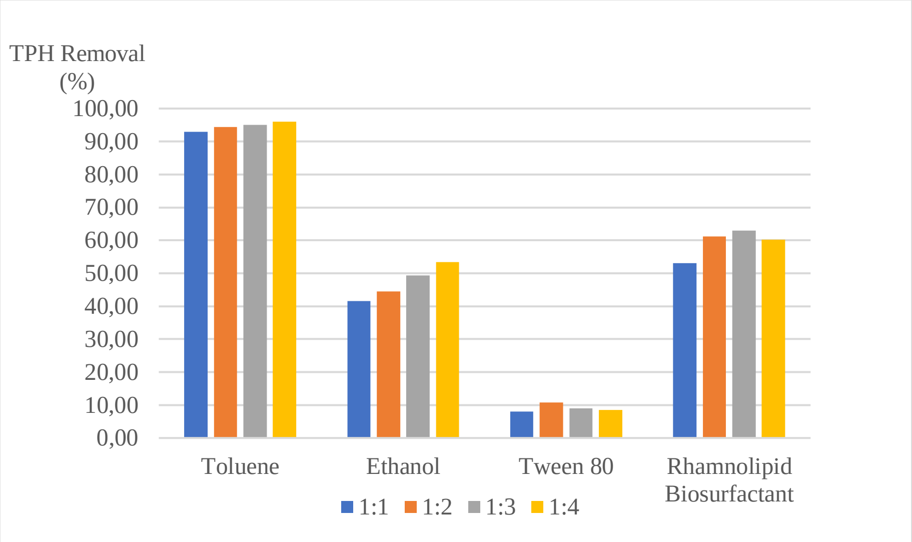

Figure 3 TPH Removal on Soil Residue of Different Temperatures for Solvent Extraction Process.

3.4 Optimization Solvent Extraction Treatment using Taguchi Experimental Method

The Taguchi Orthogonal Experimental method has been selected as a reference to find the optimum condition of parameters to consider the best approach to get optimum removal. Based on the following figure 4. it can be seen that the optimum result of TPH removal was obtained at experiment number nine, which having toluene as solvent and has solved to soil comparison 3:1 and having moisture content 13.15 % removal rate reaching 96.04 %. On the other hand, the utilization of biosurfactant giving a good prospect due to its removal efficiency that reaching 68.85% for solvent to soil comparison 3:1 and moisture content 6.64%.

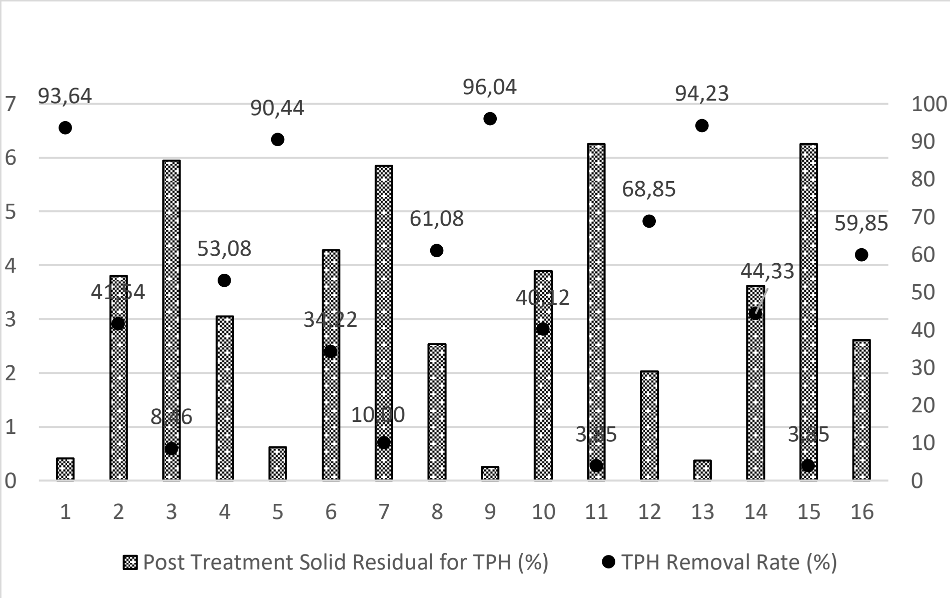

Figure 4 Solvent Extraction Experimental Result.

A promising result also showed by the usage of biosurfactant on single stage of extraction. As its EP24 index showing 87% and capable to reach TPH removal rate 68.85% for the first stage of extraction and reach 86% of TPH removal for the second stage. This biosurfactant observed gave promising implementation for low toxicity implementation of extractor. Parameter that needed to be considered to reach highest performance of biosurfactant usage were having soil to biosurfactant ratio 1:3, while moisture content was 6.67. If we compare to the original moisture content of contaminated soil that content 0.8% of moisture, the addition of moisture and adjustment with soil to biosurfactant ration would give better performance. Another adjustment that shall also be considered is experiment number 8 which indicated that at the ratio soil to solvent 1:2 and having more moisture content which is 13.35% TPH removed 61.08% at the first stage.

To seek how far the efficiency of the process of biosurfactant implementation as single extractor for more than one stage of extraction, double stage of extraction has been implemented into oily soil. Every stage of extraction result was described in the following figure 5.

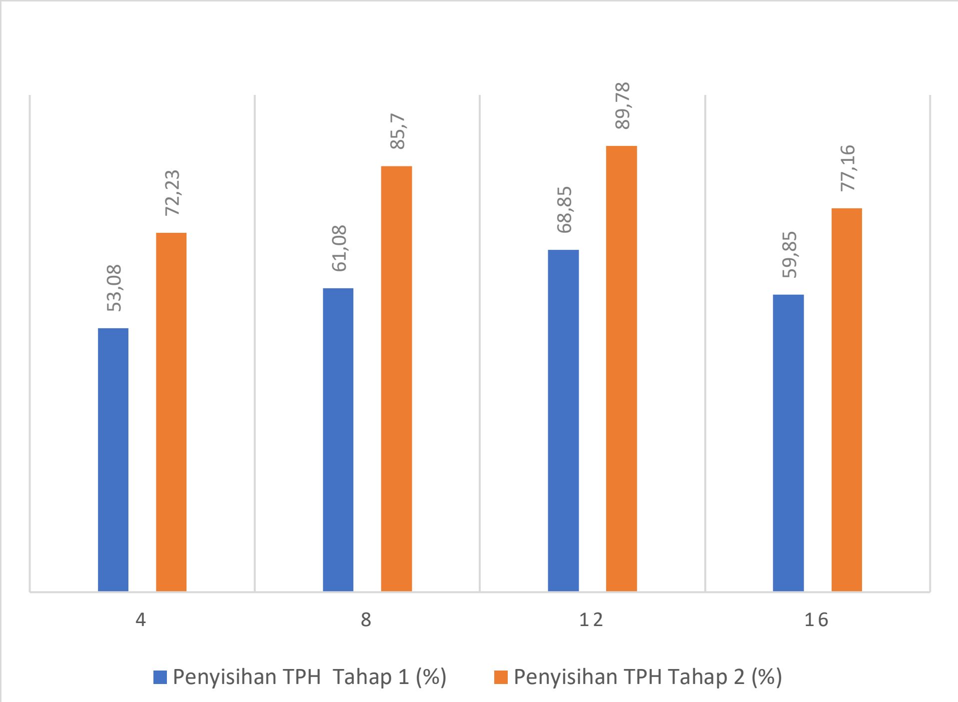

Figure 5 Double Stage Extraction of Biosurfactant Implementation for Biosurfactant Implementation.

Double stage of extraction provided higher efficiency compare to single extraction process. This process has been implemented for biosurfactant to see the performance of biosurfactant compare to chemical solvents. Result gave information that double extraction of biosurfactant on contaminated soil performed removal in average 81.21% and the most efficient dosage can achieve overall 89.78%. This is giving an expectation for further implementation of biosurfactant as oily soil decontamination agent that has more benign characteristics compare to chemical solvents such as toluene that also considered as BTEX group of contaminants.

3.5 Emulsification Index Dependency to TPH Removal by Using Statistics Method

Toluene at any of type combinations of moisture and solid to solvent gave more than 90% of TPH removal of the single stage of extraction, therefore for the reason of resource efficiency using less solvent and less water to provide enough moisture shall be preferred for further implementation. Usage of toluene, besides having some source limitations and having a higher toxicity for its released to the environment has also an opportunity to be implemented further with implementation of recovery and reuse of solvent. A test for reuse and recycle toluene has been performed showing that by using vacuum distillation unit solvent recovery could reach 84%.

4 Conclusion and Recommendation

1. Development of a solvent extraction method for decontamination of oily soil in Indonesia is a process that needed to be studied further domestically due to its benefit of decontamination an also opportunity of recycling. Some conclusions of method, optimization, statistical approach was mentioned below as part of developmental study.
2. Regarding to mixing method, combination between horizontal shaking and Centrifugation provide better performance compare conducting centrifugation as the only one mixing method.
3. Parameters that contribute to the solvent extraction process are temperature, moisture, original TPH of oily soil.
4. Toluene and Acetone as solvent provide better performance for higher TPH of oily soil (17.87%), while biosurfactant perform promising result at lower concentration of TPH (6.5%).
5. Based on Taguchi Orthogonal experimental method, toluene obtained highest TPH removal at 96.4% by adjusting soil to solvent ratio and moisture content. Biosurfactant usage performed higher result compare to synthetic surfactant. At single stage biosurfactant could reach 68.85 % TPH removal, while at double stage the overall TPH removal obtained 88.96%. Test also conducted to limit usage of resource for biosurfactant and water to adjust moisture content. It has been observed that TPH removal could achieve 81% TPH removal for double stage of extraction. Biosurfactant also performed better removal at lower TPH content of oily soil waste. Implementation of biosurfactant at the solvent extraction process might give opportunity to become pre-treatment prior large-scale implementation of bioremediation due to its capability to reduce complexity of hydrocarbon structures.
6. Recovery opportunity observed capable to be performed on chemical solvent as extractors and oxide metal recovery for both solvents and surfactants.
7. Post treatments, soil residue passed TLV of regulated TCLP result Indonesia, therefore there is a possibility to be able to discharge it safely to the environment. In addition, there is availability of the prospects for recovery of solvents and metal that present at the oily soil and has been removed along with the extractor during the solvent extraction process.
8. Further studies related ecotoxicology of post treated oily soil needed to be conducted to ensure its availability to be discharged to the environment include the possibility to conduct large scale implementation of bioremediation by implementing biosurfactant in solvent extraction process for pre-treatment of oily soil contamination.

5 Conflict of Interest Statement

The authors whose names are listed immediately below certify that they have no affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript.

6 Data Availability

The datasets generated during and/or analyzed during the current study are included in this published article.

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