Proper Treatment and Energy Recovery through Codigestion of Two Phase Olive Mill Waste

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Introduction

Many agro-food industries produce huge amount of waste in a form of biomass as a byproduct. Irregular disposal of this waste can cause a serious harm to the environment. However, such waste has received much attention as a renewable energy source. Recently, protecting the environment through a safe disposal of waste has gained much attention in order to guarantee a sustainable environment and support future needs. Moreover, the utilization of biowaste for energy production will decrease the dependency on the limited non-renewable energy sources.

Olive milling industry is one of the main industries in the Mediterranean region. The short period of olive oil production (November to March) leads to the uncontrolled discharge of the olive mill wastes to the environment. Mekki et al. (2007) reported the phytotoxic effect of olive mill wastewater (OMW) on soil microorganisms. Its negative impact on soil structure and composition has been shown as well. Moreover, the negative effect of OMW on aquatic ecosystem was studied (Karaouzas et al., 2011). Therefore, a safe disposal of this waste is needed.

There was a shift in olive oil production system from 3-phase extraction to 2-phase extraction. This shift reduced the amount of olive mill waste water produced; however, it generates another kind of waste called two phase olive mill waste (2POMW). Since the shift to 2-phase extraction is continuing, this study attempted to find a proper treatment option for the 2POMW.

Anaerobic digestion (AD) is an environmentally sustainable process for the environment and its suitability for treating several wastes such as food waste (Kawai et al., 2014), manure (Rico et al., 2011) and other wastes has been proven. Anaerobic digestion is a biological process in which complex organic materials are broken down into simpler compounds by microbes under anaerobic condition.

In order to achieve an optimum anaerobic digestion of 2POMW, several factors have to be considered. The anaerobic digestion of 2POMW can be inhibited by the presence of phenolic compounds and long chain fatty acids. Moreover, the lingocellulosic structure limits its biodegradability as it slows down the hydrolysis step. Therefore, this study tries to overcome these limitations and the energy recovery from the 2POMW.

In Jordan, Olive milling is one of the main industries. There are over 15 million olive tree producing around 105,000 t of olive. Pressing this olive generates huge amount of olive pomace and olive mill waste water. Up to date
there is no proper disposal way which creates a serious environmental problem in the country. As well, regarding energy Jordan is considered as one of the poorest countries. Utilizing the 2POMW as an input for anaerobic digestion process has a dual advantage in protecting the environment and as an additional source of renewable energy. However the short period of oil production (5 months) is one of the main drawbacks of the process. Therefore, finding a proper way of utilizing the 2POMW in anaerobic digester, while keeping a whole year around reactor operation, will be addressed in this study.

**Objectives of the study**

Since hydrolysis of lignocellulosic material is a rate limiting step for anaerobic digestion, optimizing this step can improve the overall efficiency of the process and increase its economic profitability. The 2POMW contains significant amounts of lignocellulosic material. Several studies have shown that alkaline pretreatment facilitates the degradation of lignocellulosic materials such as wheat and corn and causes a reduction in the lignin and hemicellulose content (Monlau et al., 2012; Liang et al., 2013 and Zhu et al., 2010). Therefore, the objective of this study is to examine the effect of mechanical (size reduction) and alkaline (using NaOH and CaO) pretreatments on biogas production and to investigate the pretreatment conditions for a more effective biogas conversion from the 2POMW.

The anaerobic digestion of 2POMW is limited by the presence of toxic compounds such as oleic acid (a long chain fatty acids). This limits its application as a sole source for anaerobic digesters. Therefore, the second objective of this study is to propose a system in which food waste is treated in a digester that receives 2POMW and to investigate the codigestion conditions mainly the mixing ratio which is required for the design of AD plants.

Our proposal of codigestion process for the 2POMW and food waste was applied to the existing biogas plant in Jordan which receives food waste as case study to show that proper treatment and energy recovery can be achieved for the 2POMW.

**Thesis overview**

The following chapter (chapter 2) gives an insight about the olive oil production and the environmental problems associated with it. The major three processes of olive oil extraction were identified, that will help the reader to better understand the different kinds of waste produced from different olive oil processing techniques. The current disposal practices and the different treatment options were illustrated. An experiment was conducted to determine the effect of mechanical and alkaline pretreatment. That was discussed in chapter 3 which reviewed some papers related to the anaerobic digestion and pretreatment of different lignocellulosic materials. The results of a series of batch experiments of different pretreatment conditions are shown and the effect of different pretreatment
conditions on \( CH_4 \) production was clarified. In **chapter 4** the results from chapter 3 were used to investigate the codigestion conditions of food waste with the NaOH pretreated 2POMW. In **Chapter 5** the applicability of co-digesting 2POMW with food waste in the already existing biogas plant in Jordan was investigated. Finally **Chapter 6** shows the general conclusion of this research.

**Mechanical and alkaline pretreatment of 2POMW for improving methane production**

Like other lignocellulosic biomass, the lignocellulose fraction of the 2POMW is hard to be enzymatically degraded. Cellulose, hemicellulose and lignin, the three main component of lignocellulosic material, all together form lignocellulosic building blocks that prevents its attack and destruction by bacteria and enzymes. This is mainly attributed to (1) the degree of cellulose crystallinity (2) shield effect of lignin. Monlau et al. (2012) found a strong negative correlation between lignin content and the methane production.

A series of batch experiments were performed to evaluate the effect of pretreatment. Pretreatment can overcome this limitation by altering the physical or chemical properties, increasing the surface area, breaking the linkage between cellulose, hemicellulose and lignin, thus facilitating the accessibility of hydrolytic enzymes to the substrate.

The pretreatment conditions performed were (1) mechanical pretreatment by size reduction, (2) alkaline pretreatment with different concentrations of NaOH (2.4, 6, 10, 20 and 30%) and (3) alkaline pretreatment with different concentrations of CaO (2.4, 6, 10, 20 and 30%). Following the pretreatment, anaerobic digestion was conducted in batch mode (using 200 mL vial with effective volume of 100 mL, 37 °C) for 26 days. The effect of pretreatment on the amount of soluble organic compounds (represented as soluble chemical oxygen demand (sCOD)) and methane production was determined. Since mechanical pretreatment had no effect on the sCOD, no improvement in methane production was observed. On the other hand, NaOH was able to solubilize part of the organic material. NaOH increased the concentration of sCOD, while the highest increase in the sCOD was for the 20% NaOH. Regarding methane production, when a loading rate of 0.88 (gVS\text{substrate}/gVS\text{inoculum}) of the NaOH pretreated 2POMW was applied without any pH control, the 6% NaOH pretreatment showed better performance than other treatments. The 20% NaOH pretreatment caused inhibition because of the high pH level inside the reactor (pH > 8.4). If pH is controlled, it is expected that methane production would increase. Degradation of the 2POMW by CaO was not sufficient to increase the sCOD, therefore, methane production from the CaO pretreated 2POMW was less compared with the NaOH pretreatments. Considering a full scale reactor system receiving food waste as a main substrate and the NaOH-treated 2POMW as a co-substrate, the NaOH concentration of 20% might be sufficient regarding the sCOD concentration. It was also expected that too large amount of sCOD in the loading rate might inhibit
the AD process because of the production of volatile fatty acid (VFA).

**Investigating the Co-Digestion Condition of Pretreated Two Phase Olive Mill Waste Mixed with Food Waste**

The treatment of olive mill wastes in a reactor as a sole source is limited by its high concentration of long chain fatty acids (LCFA) and phenolic compounds (Hamdi et al. 1992). Another drawback is the seasonal generation of olive milling wastes which is limited to the winter season (November to March), making the full scale application of olive milling wastes only for a short period economically unfeasible.

Continuous reactor experiment (reactor with effective volume of 6 L, 37 °C, hydraulic retention time of 30 days) for codigesting 2POMW with food waste was conducted. This study focused on investigating the mixing ratio of 2POMW to food waste in order to control the concentration of LCFA inside the reactor for a stable digestion process without inhibition. Mixing ratios of 3%, 4.3%, 5.7% and 8.3% were tested, considering the general total loading rate in COD. With increasing the mixing ratio, the organic loading rate as sCOD is also increasing, causing inhibition as expected before. To reduce the effect of sCOD on the AD process, the 2POMW used was with lower NaOH pretreatment for high mixing ratios, thus the organic loading rate as sCOD that was daily introduced into all reactors will be in the same level. There was no inhibition of methane gas production up to a mixing ratio of 4.3%; however, increasing the mixing ratio lead to higher oleic acid (the main LCFA in 2POMW) concentration and reduced methane gas production. Treatments of 10% NaOH-2POMW with 4.3% mixing ratio and 20% NaOH-2POMW with 3% mixing ratio were shown to be adequate concerning oleic acid concentration and methane gas production. Those treatments caused an increase in methane gas production by 548.5 mL/g-VS and 445.3 mL/g-VS respectively compared with the case of applying only food waste.

**THE APPLICABILITY OF A CODIGESTION SYSTEM TO TREAT 2POMW IN JORDAN**

Our proposal of codigestion process of the 2POMW and food waste was applied to the existing biogas plant in Jordan which receives 60 t/d of food waste from different sources as a case study. This plant has the potential to produce 309 MWh of electricity per month. Applying 10% NaOH pretreated 2POMW in a mixing ratio of 4.3% (which showed the highest methane production in our study) during the five months of olive oil production can produce an additional 72.5 MWh per month, increasing the plant electrical production by 23.5%. This study showed that 2POMW, which has been illegally dumped, can be properly treated and that 2POMW can be a new renewable energy source for the existing biogas plant to recover additional energy significantly.

**Conclusion**
In conclusion, this study showed that NaOH pretreatment is an effective method to solubilize the lignocellulosic fraction of the 2POMW and to enhance methane production. Since the mono-digestion of 2POMW is limited by the high concentration of LCFA, this study proposed a codigestion system of 2POMW with Food waste. This study investigated the mixing ratio of 2POMW with food waste which is the main factor that controls the anaerobic digestion process. This study proposed a practical method for 2POMW to be successfully treated and converted to energy source by a combination of pretreatment and codigestion with food waste.