

AN ECONOMIC METHOD OF MICROPLASTIC SEPARATION, EXTRACTION AND IDENTIFICATION IN AGRICULTURAL SOILS

Ibrahim Sa'adu^{1,2}, Andrea Farsang¹

¹ Department of Geoinformatics, Physical and Environmental Geography, University of Szeged, H-6722 Szeged Egyetem str. 2-6. Hungary

² Geography Department, Usmanu Danfodiyo University, P.M.B. 2346 Sokoto, Nigeria
e-mail: sbrahim@geo.u-szeged.hu

Abstract

Plastics has become a major consumable product and alternative in agriculture as a result of its playing role in energy conservation, maintaining of uniform soil temperature, and controls of weeds and fertilizer transport and thereby contaminate the soils. This research aims to provide the cost-effective method for microplastics separation and extraction from the agricultural soils. The soils were randomly collected from the greenhouse farming and conventional agriculture. The plastics used for recovery tests were collected from the field and cut off into pieces. Result from the field shows that density separation with ZnCl₂ using this method has the highest extraction capacity (400 ±100 pieces/Kg) and recovery rate (90%) compare to other floatation solutions. The method was very effective in extracting both low and high densities microplastics. Furthermore, the results infer that NaCl₂ and distilled H₂O were effective in extracting low densities microplastics such as LDPE and PP. This method provides several alternatives depend on the economy and target of users.

Introduction

Plastic is an indispensable tool in agricultural sector because of its role in processing and handling of agricultural products from nursery, planting to post harvest periods. It became a major consumable product and alternative in agriculture owing to its properties of cheapness, impermeability to precipitation and gases, malleability lightweight, maintaining of uniform soil temperature, and controls of weeds (Sussana, 2018; Patel and Tendel, 2017). The horticultural industries are emerging as major potential consumers of the plastics in form of sheets and films for crop protection, energy conservation, diseases, and pest control, water conservation supply and drainage, fertilizer transport, and building and structures (Patel and Tendel, 2017). Global plastic production has increased from 2 million tons in the 1950s to 359 million tons in 2018, the rate of this plastic recycle is very low (plastic Europe, 2019). More than half is used in protective cultivation such as a greenhouse, small tunnel, mulching, etc. Asia accounts for 48.21%, Europe 18.5%, North America 17.7%, Africa 7.1%, Latin America 4% and 2.6% go to CIS countries. China and Japan witnessed drastic growth in the sector and account for more than 30% of plastic production. Similarly, in India 5 tones of plastics is produced annually and 0.35 million tones go to agriculture (Espejo et al, 2012; Patel and Tendel, 2017).

The sources of plastic contaminants in agriculture come from primary sources such as sewage sludge, organic and inorganic fertilizer application, irrigation water application, atmospheric and wind deposition, etc.(Kaweck, et al, 2021; Wu et al, 2021; Yang et al, 2021; Katsumi et al 2021). Also, the sources can be secondary as a result of larger plastic materials disintegration from mulching, greenhouse films, plastic gauze, etc. (Mo et al 2021, Schothorst et al, 2021; Babagyayou et al, 2020; Huang, 2020). The disintegration is caused by the aging of plastic films as a result of climatic, agrochemical use, and environmental pollution factors(Dehbi, 2015; Alhamdan, 2009). These plastic contaminants litter the municipalities, cities, and farmlands because the rate of degradation is very low. Microplastic waste generated can be transferred horizontally and vertically in the soil by wind, water, microorganisms, and leaching.

The Presence of plastic contaminants causes imbalance to the ecosystem such as soil, plants, water bodies, aquatic lives, underground water, insects, animals, and human health(Serrano-Ruiz et al, 2021; Zhang et al 2021; Rondoni et al, 2021; Li et al 2021; Mora et al, 2021). However, being the studies of microplastics in the agricultural soil new and emerging(Wang et al, 2021), there is a lack of standard methods on how to identify and quantify the large concentration of microplastics in the soils (Li et al 2019; He et al, 2018). Furthermore, most of the available methods have limitations of use because of their high cost and rigorous nature of preparation stages. Also, some methods (such as Wu et al, 2021; Li et al, 2020; Zhang et al, 2018) consider single polymer type (low-density plastics). This has limitations in the agricultural soils because it comprises different compositions (organic matter, minerals, and clay) and plastic contaminants with different densities. Application of these methods will not be suitable for soils with multiple contaminants of different sizes and densities.

Materials and Methods

Sampling

The validation test was carryout on three different soils from two agricultural farmlands with different land use. The first farmland was subjected to greenhouse farming while the second was subjected to arable farming. The greenhouse farmland was already divided into 15 parcels; each parcel has the same size of 52.30m in length and 9m breadth. Three parcels were randomly selected. At this time each parcel is equally divided into two parts (known as parts A and B). In each part, the soil layer was divided into two layers (0-20cm and 20-40cm). Four samples from the same layers were bulk together and formed one composite sample. The same procedures were followed for the arable farmland. Thus, a total of 20 samples were collected from two different layers of the soils with different land-use type. However, for recovery test, five field plastics contaminants of macroplastics plastics that were use were obtained from the same field. These were cut off to pieces and formed microplastics

Laboratory Analysis

This methodology was implemented base on the improvement of the Liu et al (2019) method. The method was developed because of the high cost of other recently developed method among the other reasons. Briefly, the soils were oven-dried at 40⁰C, sieved with 5mm. A weight of 10g were placed on 250 ml conical flasks, 40 ml of 30% H₂O₂ and 10 mls of Fenton reagent were used for organic matter digestion. The solutions were place of heat sources of 70⁰C until the solutions were dried up or nearly dry. Immersion of the flask containers to cold water and addition of few drops of butyl alcohol reduced the spout out of the samples. 40 ml of 5mol/L ZnCl₂ solution (1.5g/cm³) was used as floatation salt. The solutions were capped with aluminum foil and shaken for 1 hour at 250 rpm in orbital shaker and emptied in 100ml beakers and allowed settling for 24 hours. About 20ml of upper supernatants were pipetted with glass pipette. 20ml of ZnCl₂ were added to the solution and shaken for 30 minutes in the orbital shaker for the second time. This was done in order to effectively remove the microplastics presence in the soils. The upper supernatants were combined with the second one and form a single microplastics extracts. These were later filtered through 20um and 0.45um respectively using vacuum pump. The filters were dried and taken to microscope laboratory for microplastis identification and quantification. The suspected plastic particles were confirmed through; 1. using needle and heat method and 2. Raman spectroscopic analysis.

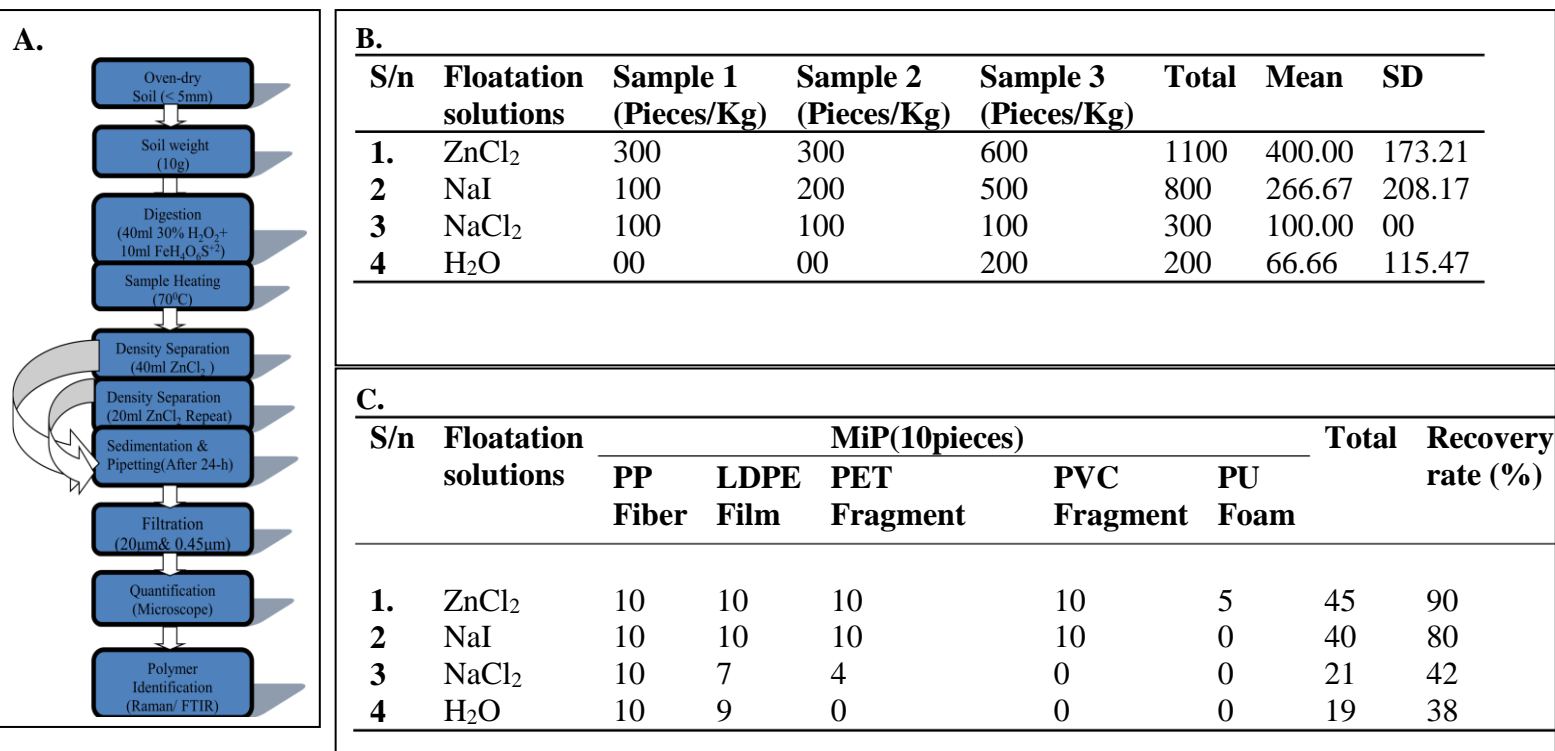


Figure 1. Extraction method and results. (A); Schematic diagram of the method. (B); Validation of the method of 4 floatation solutions. (C); Recovery test using different floatation solution on different microplastics densities

Result and Discussion

Microplastics were detected in all soils tested with different floatation solutions. Table a. Shows that; ZnCl₂ and NaI yielded higher MiP concentration of 400 ± 100 pieces/Kg and 266.67 ± 120 pieces/Kg respectively. Also, NaCl₂ and distilled H₂O recorded the low average concentration of 100 pieces/Kg and 66.66 pieces/Kg respectively. Similar findings were reported in the method developed by Li et al, (2019) where ZnCl₂ and NaI reported to have the excellent yield of microplastics extraction compare to other salts. However, the recovery test by Table b. shows that ZnCl₂ has the highest recovery rate of 90% followed by NaI which has 80%. These recoveries conform to findings of Wu et al, (2021) and Li et al, (2019). Furthermore, the careful observation of the table shows that all the floatation solutions tested good for low density plastics (PP and PE) as all the low densities were recovered in high number. But for the high density plastics (PET, PVC and PU), high recovery rates were only found in the samples treated with ZnCl₂ and NaI solutions. This result confirmed the findings of Zhang et al, (2018) which concludes that density separation with NaCl₂ was efficient in extracting low density plastics such as PP and LDPE.

However, the recovery tests reveal capacity of floatation solutions on plastic structure. ZnCl₂ and NaI were tested very well in extracting fibers, film, and fragment. But the ZnCl₂ yielded average result (5 pieces) in terms of foam's extractions while the NaI was recorded very low in terms of foam structures. The reason of low recovery of PU (foam) despite its less density compare to PET and PVC might be associated to the nature of foam materials of larger pore space that were occupied by soil particle materials and increases its density. Similarly, for NaCl₂ and distilled H₂O, only fibers and films were recovered at the high rate. This finding also tally

with several findings which concluded that these salts solutions are efficient in removal of fibrous materials (Liu et al 2018; Corradini et al, 2019; Li et al, 2019)

Conclusion

This method tests the extraction capacity of different floatation solutions on low and high density microplastics. The method was developed to minimize the cost of microplastic extraction analysis. In both the validations and recovery tests, the method shows very good result with $ZnCl_2$ and NaI for the separation and extraction of high and low density plastics particles as well as all the plastic structure with the exception of foam. Similarly, for the extraction of low density microplastics as well as structures such as fiber and film, $NaCl_2$ and distilled H_2O can serve as good floatation solutions.

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References

- [1] Alhamdan A. M., and Al-Helal I.M (2009). Mechanical deterioration of polyethylene greenhouses covering under arid conditions. *Journal of materials processing technology* 209Pp. 63–69 doi:10.1016/j.jmatprotec.2008.01.052
- [2] Babaghayou M.I., Mourad A.,I., Ochoa A., Beltran F. and Cherupuraka N(2020). Study on the thermal stability of stabilized and unstabilized low-density polyethylene films. *Polym. Bull.* 78, Pp.5225–5241. <https://doi.org/10.1007/s00289-020-03363-5>
- [3] Chen Y., Leng Y., Liu X., and Wang J. (2020). Microplastic Pollution in Vegetable Farmlands of Suburb Wuhan, Central China. *Environmental Pollution* 257
- [4] Dehbi A., Mourad A I., Djakhdane K. and Hilal-Alnaqbi A. (2015). Degradation of Thermomechanical Performance and Lifetime Estimation of Multilayer Greenhouse Polyethylene Films Under Simulated Climatic Conditions. *Polymer Engineering and Science* 55(2) DOI:10.1002/pen.23895
- [5] Espejo C., Arribas A., Monzó F., Díez P.P (2012). Nanocomposite films with enhanced radiometric properties for greenhouse covering applications. *Sage Journal*. <https://doi.org/10.1177/8756087912439058>
- [6] Giacomelli G.A. and Roberts W.J. (1993). *Greenhouse Covering System*. *Horttechnology*3(1)
- [7] He D., Luo Y., Lu S., Liu M., Song Y., and Lei L.(2018). Microplastics in soils: Analytical Methods, Pollution Characteristics and Ecological Risks. *Trends in Analytical Chemistry* 109 pp 163-172.
- [8] Huang Y., Liu Q , Jia W., Yan W., Wang J. (2020). Agricultural plastic mulching as a source of microplastics in the terrestrial environment. *Environmental Pollution* 260 114096. <https://doi.org/10.1016/j.envpol.2020.114096>
- [9] Katsumi N., Kusube T., Nagao S., Okochi H. (2020). Accumulation of microcapsules derived from coated fertilizer in paddy fields. *Chemosphere*. <https://doi.org/10.1016/j.chemosphere.2020.129185>
- [10] Jiang S., Wang F., Li Q., Sun H., Wang H., and Yao Z (2021). Environment and food safety: a novel integrative review. *Environmental Science and Pollution Research* 28:54511–54530. <https://doi.org/10.1007/s11356-021-16069-6>
- [11] Kawecki D., Goldberg L., and Nowack B.(2020). Material Flow Analysis of Plastic In Organic Waste In Switzerland. *Soil Use Manage.* 1– 12. <https://doi.org/10.1111/sum.12634>

- [12] Li Q., Wu J., Zhao X., Gu X., and Ji R.(2019). Separation and identification of microplastics from soil and sewage sludge. *Environmental Pollution* 254 113076. <https://doi.org/10.1016/j.envpol.2019.113076>
- [13] Li W., Wufuer R., Duo J., Wang S., Luo Y.,Zhang D., Pan X (2020). Microplastics in agricultural soils: Extraction and characterization after different periods of polythene film mulching in an arid region. *Science of the Total Environment* 749 141420. <https://doi.org/10.1016/j.scitotenv.2020.141420>
- [14] Liu M., Lu S., Song Y., Lei L., Hu J., Lv W., Zhou W., Cao C., Shi H., Yang X., and He D.(2018). Microplastic and Mesoplastic Pollution in Farmland Soils in Suburbs of Shanghai, China. *Environmental Pollution* 242 pp 855-862
- [15] Mo X., Li H., Lian Y., Zheng B., Dong J., Lu X. (2021). Estimation of soil microplastic input derived from plastic gauze using a simplified model. *Science of the Total Environment* 793 148577 <https://doi.org/10.1016/j.scitotenv.2021.148577>
- [16] Mora A., García-Gamboa M., Sánchez-Luna M.S., Gloria-García L.,Cervantes-Avilés P., Mahlnecht J. (2021). A review of the current environmental status and human health implications of one of the most polluted rivers of Mexico: The Atoyac River, Puebla. *Science of the Total Environment* 782 146788. <https://doi.org/10.1016/j.scitotenv.2021.146788>
- [17] Patel A., and Tandel Y. (2017). Use of Plastics in Horticulture production. *Indian Farmer(Special Issue III)* Pp. 108-112.
- [18] Plastic Europe (2019), Plastic the facts. An analysis of european plasticproduction, demand and waste data. http://www.plasticeurope.org/application/file/9715/71299584/FINAL_web_version_plastics_the_facts2019_14102019.pdf
- [19] Schothorst B., Beriot N., Lwanga E.H and Geissen V. (2021). Sources of Light Density Microplastic Related to Two Agricultural Practices: The Use of Compost and Plastic Mulch. *Environments* 8, 36. <https://doi.org/10.3390/environments8040036>
- [20] Serrano-Ruiz H., Martin-Closas L., Pelacho A.M. (2021). Biodegradable plastic mulches: Impact on the agricultural biotic environment. *Science of the Total Environment* 750 141228
- [21] Susanna G. (2018). Plastic Pollution in Soil. *Interactive Soil Quality Assessment*. Institute for European Environmental Policy Waste in Switzerland. *Soil Use Manage.* 1– 12. <https://doi.org/10.1111/sum.12634>
- [22] Wittwer S.H.(1993). World-wide Use of Plastics in Horticultural Production, *HortTechnology*3(1)
- [23] Wu R., Cai Y., Xing S., Yang Y., Mi J., and Liao X.(2021). A Novel Method for Extraction of Polypropylene Microplastics In Swine Manure. *Environmental Science and Pollution Research* 28:13021–13030. <https://doi.org/10.1007/s11356-020-11111-5>
- [24] Yang J., Li R., Zhou Q., Li L., Li Y., Tu C., Zhao X., Xiong K., Christie P., and Luo Y. (2021). Abundance And Morphology of Microplastics In An Agricultural Soil Following Long-Term Repeated Application of Pig Manure. *Environmental Pollution* 272 116028
- [25] Zhang S., Yang X., Gertsen H., Peters P., Salánki T., and Geissen V. (2008). A Simple Method For The Extraction and Identification of Light Density Microplastics From Soil. *Science of the Total Environment* Pp.1056–1065
- [26] Zhang S., Yang X., Gertsen H., Peters P., Salánki T., and Geissen V(2018). A simple method for the extraction and identification of light density microplastics from soil. *Science of the Total Environment* 616–617 Pp.1056–1065 <https://doi.org/10.1016/j.scitotenv.2017.10.213>