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Production of Liquid Organic Fertilizer from Sewage Sludge with Reduced Heavy Metal Toxicity via Chemical and Organic Treatments

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The usage of sewage sludge in agriculture has increased the accumulation of some heavy metals (such as cadmium, copper, lead, etc.) in soil and plants, due to the high concentration of these elements. Therefore, this research was conducted to investigate the best method to reduce the toxicity of heavy metals in sewage sludge while causing less environmental pollution. This approach utilized NaOH and HCl, along with humic and fulvic acids, to precipitate heavy metals from sewage sludge complexed with heavy metals in sludge to reduce the concentration of heavy elements and produce liquid organic fertilizer. In this research, the concentration of heavy metals (As, Cd, Cu, Ni, Pb, and Zn) in the sewage sludge, humic, fulvic, and liquid organic fertilizer were measured. Also, the humic, fulvic, and the produced organic fertilizer were tested on corn plants in terms of two treatments (10%, and 20%) and the heavy metals of those were analyzed. All experiments were conducted in three replications. The results showed the values of heavy metals in the humic, fulvic and liquid organic fertilizer were lower than the acceptable contents of EPA. The content of these elements in corn plants treated with humic, fulvic, and liquid organic fertilizers were lower than the EPA standard. Therefore, this liquid organic fertilizer can be used as two concertation (10% and 20%) for plant nutrition and compared to chemical fertilizers, is not only economically cost-effective, but also does not have the pollution caused by the use of chemical fertilizers.

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1. Introduction

In recent years, the high usage of chemical inputs in agriculture has caused numerous environmental problems, including the pollution of water resources, the decline in the quality of agricultural products, and a reduction of soil fertility (Sharma, 2002). The health of the products produced in the different agricultural systems in terms of the presence of pesticides and chemical residues and their impact on human health and the environment has caused special attention to be paid to the production methods and inputs used. Also, one of the most important issues that affects environmental health and the sustainability of food production is the use of organic fertilizers instead of chemical fertilizers (Neeson, 2004).

Sewage sludge is usually applied in several ways. Sewage sludge can be applied to land surfaces in pastures and meadows, and on the ground, or injected into the subsoils. However, the important point for sludge used as soil fertility (as fertilizer), is its control under the Environmental Protection Agency (EPA) 503 rule which includes heavy metals (Kajitvichyanukul and Arcy, 2022). Heavy metals are elements found in the Earth's crust with a content less than 0.1% (Adriano, 2001). The atomic weight of these elements is between 63.5 and 200.6 grams and their specific gravity is greater than five grams per cubic centimeter (Kalbasi *et al*., 1978). In fact, the term heavy metal refers to any element that has a high density (Lenntech water treatment and air Purification, 2004). Among the 90 elements in nature, 53 of them are heavy metals, and only a few of them are biologically important (Hasan *et al*., 2009). These elements include iron, zinc, copper, manganese, etc. These elements are essential for the life of both plants and animals. The main sources of unnatural entry of heavy elements into the soil are the result of some human activities. For example, activities such as mining, wastewater consumption, sewage sludge, atmospheric sediments, type of irrigation water, domestic and industrial wastewater entering the irrigation system, vehicles, pesticides, animal manure, compost, and chemical fertilizers can cause an abnormal increase in the concentration of these elements in the soil (Weber and Karczewska, 2004). A large part of the agricultural land is affected by using of chemical fertilizers. The areas around large and industrial cities, factories, mines, and main roads are among the regions solids and inorganic where the concentration of heavy metals is high and in most cases in the toxic range (De Meeus *et al*., 2002). Based on previous research, if the toxicity of heavy metals is reduced to an acceptable level by using solutions such as NaOH and HCl with the precipitation of humic and fulvic acid, which have a high affinity for complexing heavy metals, this valuable sewage sludge (in terms of major nutrients such as nitrogen, phosphorus, and potassium, which are effective for plant growth) can be used in the form of organic fertilizer to meet the nutritional needs of plants. Shafiepour *et al*. (2010) studied the effect of using urban sewage sludge in improving soil characteristics in the Kish and found that the concentration of heavy metals such as arsenic, cadmium, chromium, copper, zinc, and nickel in the sludge was within the permissible range compared to the Environmental Protection Agency (EPA), therefore they used sewage sludge mixed with soil to meet part of the plant's needs. Badr *et al*. (2012) used the phytoremediation method to remove the heavy metals from soils that were contaminated with cadmium, copper, lead, zinc, and iron. In this method, heavy metals are not completely removed from the soil and there is a risk of contamination, Also, due to the accumulation of heavy metals in the plant, any burning and burial will return the heavy metals to the soil, which is one of the disadvantages of using this phytoremediation method. Eslami and Nemati (2015) used the bioremediation method to investigate the removal of heavy metals (cadmium, lead, arsenic, and nickel) from wastewater and soils contaminated with them. Considering that using this method requires a specific type of microbes and specific environmental conditions for their growth, it is economically costly and not cost-effective. Barakat (2011) described a method for removing heavy metals from industrial wastewater by using a filter that removed the organic matter, solids, and inorganic contaminants such as heavy metals. According to the size of the remaining particles, this filtration included ultrafiltration, nano-filtration, and osmosis sources, which were effective in the removal of heavy metals from industrial wastewater. In this method, various filters are used to remove the heavy metals, which are determined depending on the size of the particles remaining in the treatment. Providing these filters in large volumes of industrial wastewater based on the size of the remaining particles requires a lot of expense because the size of the remaining particles must be determined and then the type of filter for purifying the heavy metals to be specified.

U.S. Pat. No. 5,482,528 described a method for purifying sewage sludge, which is used as a useful production product for agricultural land improvement. In this method, the sludge is mixed with acid and a chemical reaction occurs at high temperatures, so the materials are absorbed by soil particles after passing through volcanic ash. This invention is part of U.S. Pat. No. 5,422,015, which described a method for purifying sewage sludge. In this invention, volcanic ash has been used to trap heavy metals. Also, the sludge may not be in contact with the ash while passing through the volcanic ash, and some heavy metals with a high degree of pollution may enter the soil and finally enter the plant. Considering that humic and fulvic are two parts of organic matter that have a great affinity with heavy metals, this research was conducted to investigate the humic and fulvic precipitation of sewage sludge and the use of the remaining solution after precipitation as organic fertilizer as well as investigation its effect on the corn plant.

2. Materials and Methods

2.1. Preparation and analysis of sewage sludge

The content of sewage sludge produced in Urmia city is 13 thousand cubic meters per day, which is dried and transferred to the landfill of Urmia city without any efficiency. In this research, three kg of dried sludge was taken from the sewage sludge (Fig. 1). The prepared sludge was air-dried at 25°C, and passed through a 1 mm sieve. Some properties of sludge include electrical conductivity (EC) and potential of hydrogen (pH) at a 1:5 sludge to water ratio (Uysal *et al*., 2016), organic carbon (OC) and organic matter (OM) by potassium dichromate method (Giovannini *et al*. 1985), nitrogen with Kjeldahl (Bremner, 1996), phosphorus by nitric acid (Kuo 1996), soluble calcium and magnesium by using 2 normal potassium hydroxides and 0.01 normal EDTA (Hendershot *et al*., 2007), soluble sodium and potassium with film photometry (Hendershot *et al*., 2007) were measured. The concentration of As, Cd, Cu, Ni, Pb, and Zn were analyzed with the flame atomic absorption spectrophotometry (FAAS) in air acetylene (Shimadzu-Tokyo-Japan AA-6300) (Mc Bride *et al*., 1997).

Fig. 1. Sludge sample taken from Urmia wastewater treatment plant.

2.2. Preparing organic fertilizer from sewage sludge

To prepare the organic fertilizer, first sewage sludge was washed with 1N HCl. Then the NaOH solution was added to it with a ratio of 1 to 20, and the whole solution was shaken for 24 hours at high speed. After this period, the solution was centrifuged at 6000 rpm to separate the supernatant solution from the suspended particles. The pH of the solution was adjusted between 1-2 with 1N HCl and stored in the refrigerator for 24 hours. This period caused the humic part of the organic matter that is insoluble in acid to precipitate (Fig. 2). Then, the supernatant solution was separated from the precipitate with a filter paper and its pH was again adjusted to 4-5.5 with NaOH. This action caused the precipitation of the fulvic part of the organic matter, which is insoluble in alkali. The supernatant solution is the desired liquid organic fertilizer (Fig. 2). Also, the concentration of heavy metals (As, Cd, Cu, Ni, Pb and Zn) was measured in the separated supernatant, humic, fulvic, and liquid organic fertilizer.

Fig. 2. Humic, fulvic, and liquid organic fertilizer separated from sewage sludge.

2.3. Using this organic liquid fertilizer on corn plant

To test this organic fertilizer prepared from sewage sludge, it was sprayed on corn plants in three stages during the growing season in two treatments (10% and 20%). Then, these plants were harvested and after air drying and passing through a 0.5 mm sieve, 2 molar HCl was used to extract heavy metals. A flame atomic absorption spectrophotometry (FAAS) in air acetylene (Shimadzu-Tokyo-Japan AA-6300) was used to investigate the concentration of heavy metals (As, Cd, Cu, Ni, Pb and Zn) in the plant samples (Malakouti and Gheibi, 2000).

2.4. Statistical analysis

Also, all experiments related to sewage sludge, fulvic, humic, and liquid organic fertilizer were performed in three replicates, and then the data were categorized, and analyzed in Excel software. So, tables and figures were drawn. All data were analyzed using appropriate statistical software such as SPSS or R. ANOVA was conducted, followed by Duncan's multiple range test at $p<0.05$. Excel was used only for tabulation and visualization.

3. Results and Discussion

3.1. Chemical properties of sewage sludge, fulvic, humic, and liquid organic fertilizer

The chemical characteristics of the sewage sludge, fulvic, humic, and liquid organic fertilizer are presented in Table 1.

Table 1. Mean ± standard deviation values of chemical properties of sewage sludge, humic, fulvic, and liquid organic fertilizer

Parameters	Unit	Sewage sludge	Humic	Fulvic	liquid organic fertilizer
Nitrogen	ppm	9100 ± 0.06a	572 ± 0.76c	611 ± 0.05b	85.2 ± 0.08d
Calcium	%	2.42 ± 0.07a	1.08 ± 1.13c	1.28 ± 0.05b	059 ± 0.02d
Magnesium	%	0.17 ± 0.08a	0.019 ± 1.30c	0.13 ± 0.04b	0.031 ± 0.01d
Sodium	%	9.79 ± 0.05a	0.17 ± 1.31d	1.87 ± 0.03b	0.38 ± 0.02c
Potassium	%	1.66 ± 0.06a	0.63 ± 1.64b	0.57 ± 0.05c	0.29 ± 0.03d
Phosphorus	ppm	4300 ± 0.21a	460 ± 1.80c	750 ± 0.06b	121 ± 0.03d
pH	-	6.8 ± 0.04a	6.3 ± 0.69a	6.6 ± 0.05a	6.5 ± 0.26a
EC	ds/m	2.5 ± 0.02a	1.2 ± 0.48c	1.6 ± 0.01b	0.85 ± 0.10d
Organic carbon	%	2.7 ± 0.02a	1.9 ± 0.27b	2.3 ± 0.09b	1.06 ± 0.10ca
Organic matter	%	3.96 ± 0.02a	2.41 ± 0.16b	3.22 ± 0.03a	1.76 ± 0.03c

Different letters in each row indicate significant differences at $p<0.05$ according to Duncan's test. pH: Potential of hydrogen, EC: electrical conductivity.

According to Table 1, the highest and the lowest content of nitrogen was observed in the sewage sludge and liquid organic fertilizer after humic and fulvic precipitation respectively. Nitrogen content in humic was slightly lower than in fulvic. The amount of calcium and magnesium in the four studied treatments were in order: sewage sludge> fulvic> humic> and liquid organic fertilizer. As similar the values of sodium, potassium, and phosphorus were most in the sewage sludge, and the lowest content of them was observed in the liquid organic fertilizer. Also, the electrical conductivity (EC) as well as the potential of hydrogen (pH) in the sewage sludge were higher than the humic, fulvic, and liquid organic fertilizer and reported as in order: sewage sludge> fulvic> humic> and liquid organic fertilizer. The amount of organic carbon and organic matter similar to other chemical parameters measured in this investigation were sewage sludge> fulvic> humic> and liquid organic fertilizer respectively.

3.2. Heavy metals in sewage sludge, humic, fulvic, and liquid organic fertilizer

The content of heavy metals in the sewage sludge, fulvic, humic, and liquid organic fertilizer is shown in Table 2.

The amount of Arsenic in the sewage sludge, humic, fulvic, and liquid organic fertilizer was not more than the standard level and the accumulation of this element in the study treatments was not observed. The cadmium content in the sewage sludge, humic, and fulvic was 3.46, 1.07, and 1.97 $mg.kg^{-1}$ which was lower than the acceptable level (85 $mg.kg^{-1)}$ reported by EPA (2003). Also, the amount of this element in liquid organic fertilizer was 0.002 $mg. L^{-1}$ that was at the permissive level of EPA (2006) (0.01 $mg. L^{-1}$). The copper value in the sewage sludge was more than the humic, fulvic, and liquid organic fertilizer, but this element in all treatments was bellow acceptable level. Also, the contents of nickel, lead, and zinc were similar to the other elements in order: sewage sludge> fulvic> humic> and liquid organic fertilizer, whereas these elements were at the standard values of EPA (2003 and 2006).

Table 2. Mean ± standard deviation contents of heavy metals in sewage sludge, humic, fulvic, and liquid organic fertilizer

Heavy metals	Unit	As	Cd	Cu	Ni	Pb	Zn
Sewage sludge	$mg.kg^{-1}$	28 ± 0.04a	3.46 ± 0.69a	141.27 ± 0.05a	14.75 ± 0.02a	3.36 ± 0.31a	478.23 ± 0.15a
Humic	$mg.kg^{-1}$	0.46 ± 0.02c	1.07 ± 0.48c	2.32 ± 0.01c	2.59 ± 0.10c	1.39 ± 0.02c	30.54 ± 0.35c
Fulvic	$mg.kg^{-1}$	2.8 ± 0.02b	1.97 ± 0.27b	2.59 ± 0.09b	3.48 ±0.10b	1.82 ± 0.02b	42.40 ± 0.21b
EPA (2003) acceptable level	$mg.kg^{-1}$	75	85	4300	420	840	7500
liquid organic fertilizer	$mg.L^{-1}$	0.025 ± 0.05d	0.002 ± 0.16d	0.11 ± 0.03d	0.15 ± 0.03d	0.017 ± 0.02d	0.35 ± 0.40d
EPA (2006) acceptable level	$mg.L^{-1}$	-	0.01	0.2	0.2	5	2

Different letters in each row indicate significant differences at $p<0.05$ according to Duncan's test. As: Arsenic, Cd: Cadmium, Cu: Copper, Ni: Nickel, Pb: Lead, Zn: Zinc.

3.3. The humic, fulvic, and liquid organic fertilizer test on corn plants and investigation of the concentration of their heavy metals

In this study, we used two concentrations (10% and 20%) of humic, fulvic, and liquid organic fertilizer that sprayed them on corn plants respectively. Also, we evaluated the concentration of heavy metals on the leaves of corn plants that were treated with 10% and 20% of humic, fulvic, and liquid organic fertilizer after harvest (Fig. 3).

According to Figure 3, We didn’t observe the arsenic, cadmium, nickel and lead in the two concentrations (10% and 20%) of humic, fulvic, and liquid organic fertilizer. However, we reported the copper and zinc in corn plants, but in very low quantities. In the 10% concentration, the content of copper in the corn plant treated with humic, fulvic, and liquid organic fertilizer was in order: fulvic> humic> liquid organic fertilizer, however in the 20% of them, the concentration of copper in corn plant was fulvic> liquid organic fertilizer> humic respectively. Also, the content of zinc in corn plants treated with the mentioned treatments in the 10 and 20 percent were humic> fulvic> liquid organic fertilizer respectively and the amount of these two elements in the concentration of 20% was greater than the 10%.

The images of crops treated with humic, fulvic, and liquid organic fertilizers are shown in the Figure 4.

According to the obtained results, the contents of measured elements in the sewage sludge were the highest, and in the liquid organic fertilizer were the lowest, however, this liquid organic fertilizer can support all needed elements of the plant well. The organic fertilizer produced from the sewage sludge in this study (in liquid form) contained macro and micronutrients that pollution reduced by precipitation with humic acid and fulvic acid and was able to fully meet the plant's nutritional needs. Due to this fertilizer is organic therefore, it is environmentally friendly and does not pose the risks of chemical fertilizer contamination, which accumulates in the soil and destroys the soil's texture and structure (Kabata-Pendias, 2010). Also, due to the low and acceptable levels of heavy elements in both 10% and 20% organic fertilizer concentrations, it is possible to use both depending on the plant's needs. On the other hand, the average consumption of various types of fertilizers (in terms of nutrients) in the world was 101 kg/ha, Australia 150, Belgium 354, Germany 220, Ireland 527, England 313, India 79, and the European Union 200 kg/ha (Nourbakhsh, 2016). This was while Iran's consumption of chemical fertilizers is estimated at 7.5 million tons per year (Agricultural Organization of West Azerbaijan, 2018). The western Azerbaijan province of Iran has one million hectares of arable land, of which 700,000 to 800,000 hectares are annually allocated to the cultivation of horticultural and agricultural crops (Iran Customs Organization, 2015). The farmers of this region need 158,000 tons of chemical fertilizers annually (Iran Industries and Industrial estates organization, 2008). The produced organic fertilizer is a suitable alternative to chemical fertilizers and significantly reduces the consumption of chemical fertilizers.

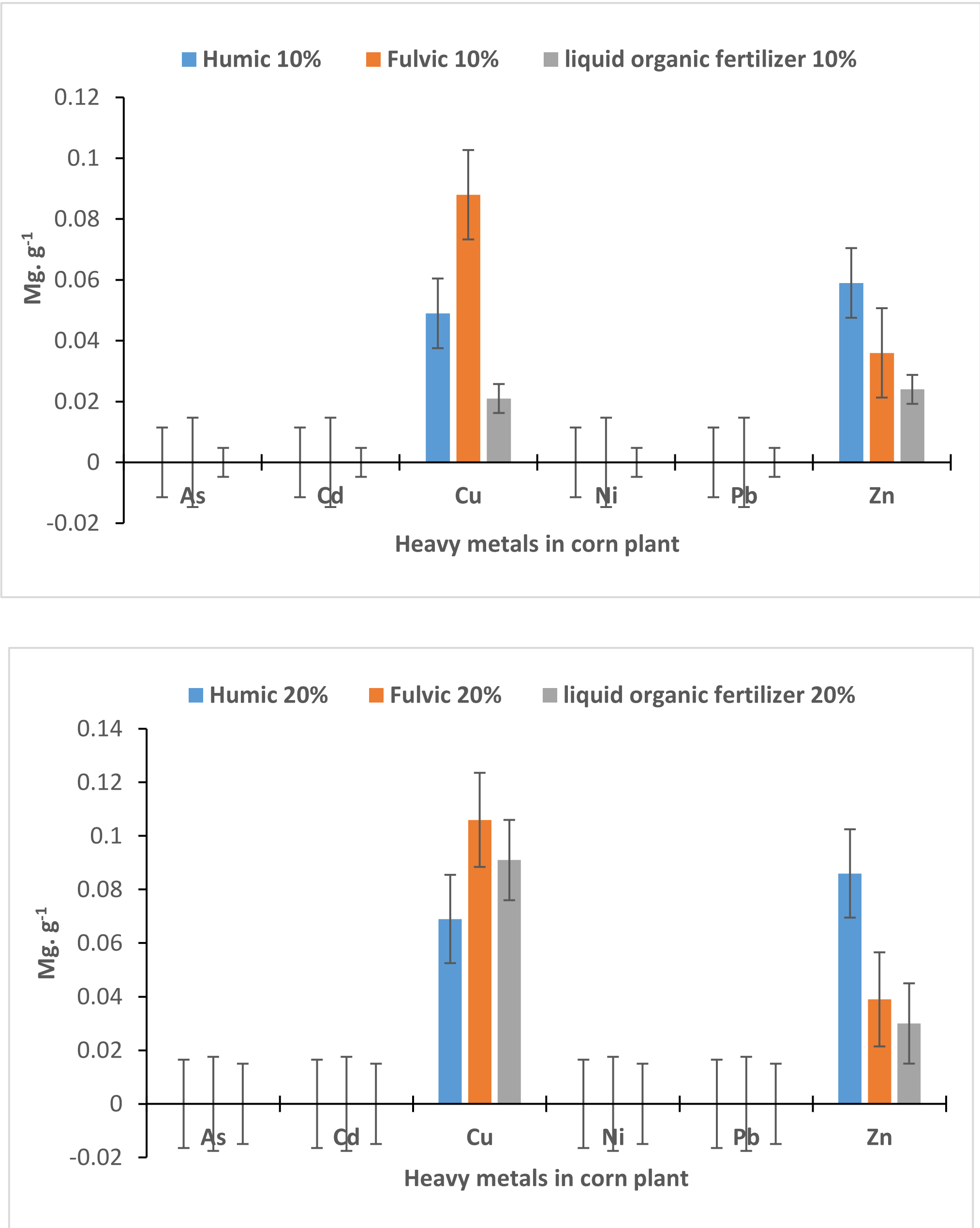

Fig. 3. Heavy metal levels in the corn plants treated with the humic, fulvic, and liquid organic fertilizer.

The cost of chemical fertilizers containing the two elements nitrogen and phosphorus in the country (cooperative price) in 50-kilogram packages is 1,100,000 rials (Iran Customs Organization, 2015).

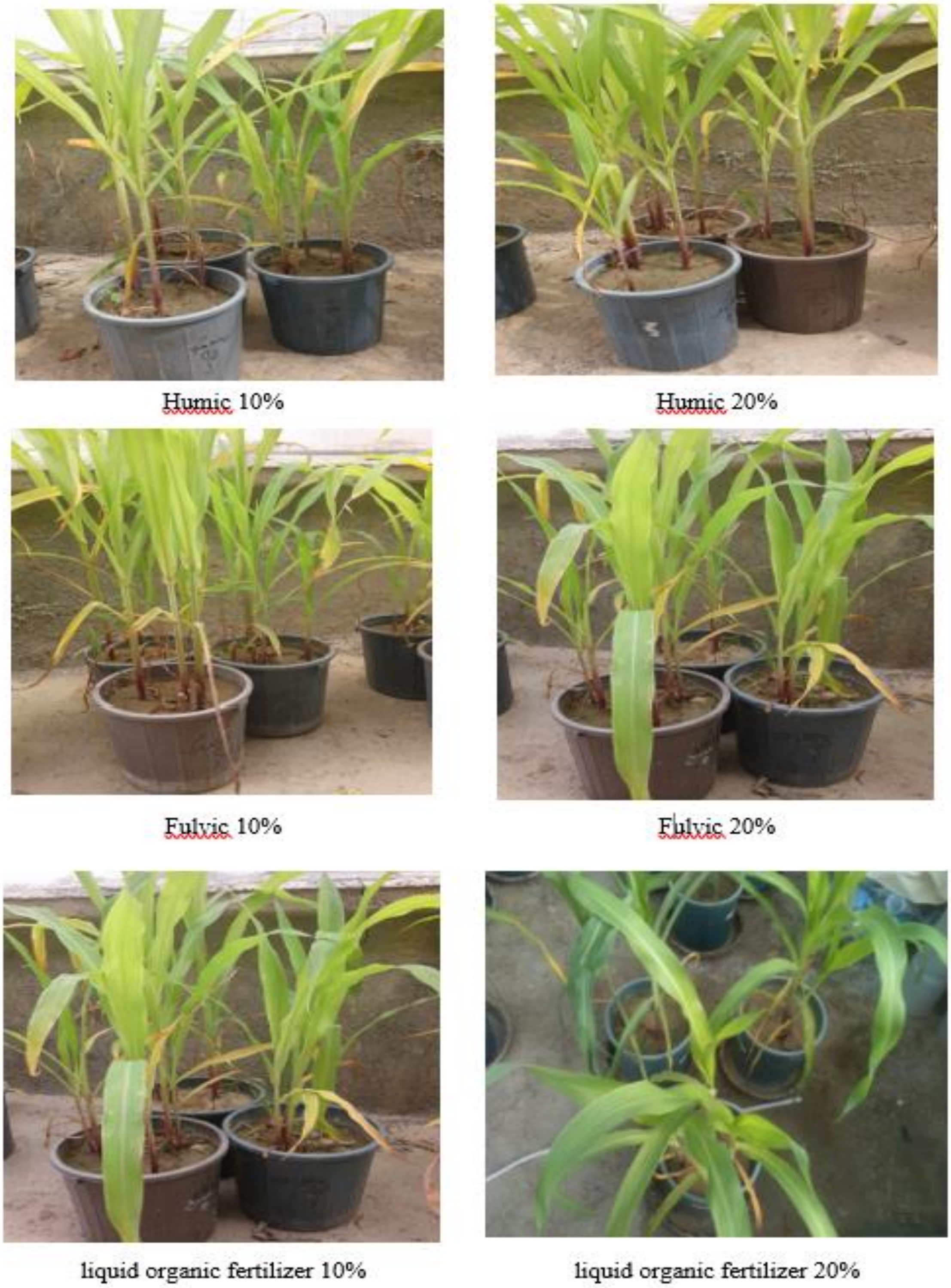

Fig. 4. View of corn plants treated with humic, fulvic, and liquid organic fertilizer.

In recent years, the average of complete fertilizers consumption (solid and liquid) from total fertilizers consumed in the world has been about 18 percent. About 70% of the total amount of complete fertilizer consumed in the world was in solid form and only 30% was in liquid form. However, using of liquid fertilizer does not have the erosion and pollution risks of chemical fertilizers mentioned above and is added to the soil in proportion to the amount needed by the plant along with sprinkler and drip irrigation, and therefore does not cause pollution of the mentioned resources. The current production status of liquid fertilizer is very low at 15-20 percent and the current production rate of this product in the country is 4027 tons per year. The price of one liter of liquid fertilizer containing nitrogen, phosphorus and potassium in the country is 39500 rials (Iran Industries and Industrial estates Organization, 2008). The price of one liter of this organic fertilizer produced in liquid form is 29,000 rials, calculating the price of all the raw materials used to refine heavy metals. Therefore, this fertilizer is very cost-effective and has economic value for the consumer.

4. Conclusion

The organic fertilizer produced in this study is used in liquid form in one-liter packages for drip irrigation, under pressure, or even using special spray containers to meet the nutritional needs of gardens, fruit trees, and crop farmers. Also, using this fertilizer reduces the pollution of chemical fertilizers that destroy soil texture and structure, and using fertilizer in liquid form increases the absorption level and reduces the amount of fertilizer used. Therefore, using this fertilizer is very environmentally friendly and economically affordable for the consumer.

Data Availability Statement

Data is available on request from the author.

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Ethical considerations

The author avoided data fabrication and falsification.

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Conflict of interest

The author declares no conflict of interest.

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