Turning wastes into valuable materials: Valorization of pistachio wastes in agricultural sector

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Abstract:

*Purpose:*In the framework of the LIFE11 ENV/GR/951 project AGROSTRAT, pistachio wastes produced in Aegina island, Greece, were mixed in different ratios with other raw materials (i.e. goat and sheep manure, straw) to produce three composts (A, B and C) and to investigate the potential of wastes valorization in agricultural sector. Clinoptilolite, a natural zeolite, was added to the mixture of one of the three composts to investigate the potential of improving the properties of the final product. *Methods:*The feedstock was composted using widrows and three composts were produced. Clinoptilolite was added to the one of the three composts at a percentage of 5%. When maturity was achieved, the properties of the materials were determined. Germination index was determined as well. Water extracts of the three composts and one of the raw solid pistachio waste were studied as regards their potential phytotoxicity. *Results:* The properties of the three composts were within the acceptable quality limits, however the composts without clinoptilolite had very high Electrical Conductivity (EC). The addition of clinoptilolite reduced the EC at low and acceptable values. Germination index indicates that the zeolite-compost can be used without any limitation for plant growth. Some limitations are existed for the other composts of this study. *Conclusions:*The EC is the main restricted factor for the production of high quality composts from pistachio wastes. The addition of clinoptilolite in the compostable mixture results in a high quality compost that may enhance germination and plant growth.

Keywords: Pistachio wastes, compost, clinoptilolite, natural zeolites, *Pistachia vera* L., germination index.

1. Introduction

Pistacia vera L. (Anacardiaceae), the only one of the 11 species of the genus Pistacia that produces edible nuts, is a small tree grown in Southern Europe and Asia Minor. In Europe the main pistachio producer countries are Greece (10,000 tn in 2012) and Italy (2,850 tn in 2012) while Cyprus and Spain are following. The annual production in Europe during 2012 is estimated at 12,867 tn [1].

Pistachio is the nut of the tree, having an edible green kernel enclosed in a woody shell. In Greece the most famous pistachio (PDO product-Protected Designation of Origin) is produced in Aegina, a small island near Attiki prefecture. Greece, with almost 10,000 tn of pistachio annual production is the European leading producer while ranks 6th in the world production [1]. Apart from Aegina Island, *Pistachia vera* L. is also cultivated in Peloponnese, Central Greece (Fthiotida, Viotia and Evoia) and in North Greece (Chalkidiki). The primary processing of these nuts results in by-product streams (hulls and shells) that are more than 75% of the harvested crop. Traditionally, these by-products are used either as animal feed, fuel for energy generation (mainly in the USA), or they are discarded as waste. It is estimated that almost 7,000 tons of pistachio wastes are produced annually in Greece.

So far little is known regarding the fate of pistachio's waste in Greece and in other Med countries. It is also mentioned that international scientific literature is rather poor, especially in issues regarding pistachio waste management. Contacts with producers from Aegina and other Greek areas provided the authors with some information regarding management of pistachio waste. According to their description and to personal visits at wastes disposal areas, liquid waste (produced mainly during nuts washing) is either disposed on soil, mainly where *Pistachia vera* L. trees or vegetables are cultivated, or is disposed in wells and streams. Solid wastes (i.e. hulls, shells and water) are sometimes left to be naturally composted and further used as supplement to fertilizers or are disposed untreated on soil or in streams.
The existence of many, small, mainly family plants, scattered throughout Mediterranean region is one

additional issue of concern, since it leads to discrete and localized soil contamination, which however may pose threats to human health and to the local environment quality.

Table 1 includes the mean composition of 13 pistachio waste samples collected during 2013 harvesting period in Aegina island by the authors. Considering its very high organic matter and nutrients content, pistachio waste could be an excellent soil additive that will promote C sequestration and nutrients recycling. This is very significant considering that nearly 75% of soil in Southern Europe is low in organic matter content and the need of recycling nutrients in agriculture [2]. However, its high content in polyphenols, nitrates, chlorides and sodium could cause adverse effects on soil and water bodies and thus, restricts its utilization without pretreatment (e.g. composting). Development of specific guidelines considering also other parameters, such as soil properties and cultivation needs will promote sustainable management of this waste type.

Table 1. Composition of solid pistachio wastes. Mean values of 13 wastes samples collected from different producers during 2013 harvesting period.

Parameter	Value	Parameter	Value
Organic matter, %	81.4	Total Na, %	0.41
Total N, %	2.3	Total Cu, mg/kg	115
C/N	23.5	Total Fe, %	0.53
Moisture, %	67.4	Total Zn, mg/kg	72.6
pH	7.48	Total Mn, mg/kg	78.7
EC, mS/cm	4.6	Total B, %	0.75
Total Polyphenols, g/kg	2.7	Cl^-, %	0.71
Total K, %	2.5	NO_3^-, %	0.10
Total P, %	0.55	PO_4^{3-}, %	0.11
Total Ca, %	3.6	SO_4^{2-}, mg/kg	237
Total Mg, %	0.38	NH_4^+, mg/kg	500

Natural zeolites are low cost reactive materials, abundant in European territory and mainly in Mediterranean region (i.e. 1 tn of the natural zeolite clinoptilolite costs only 160€). Deposits are found in southeastern Europe, Turkey, Russia, Mongolia, Korea, China, Mexico, Cuba, South Africa, Tanzania, and Kenya [3]. Only seven types (clinoptilolite, mordenite, chabazite erionite, ferrierite, phillipsite, and analcime) exist in a sufficient quantity and purity to be considered exploitable. The most widely used natural zeolite is clinoptilolite due to its existence in many countries in the world. The subsoil of Greece, Bulgaria, Turkey, Russia, etc. is rich in clinoptilolite [3].

In agriculture, the natural zeolites are widely used as slow releasing carriers of fertilizers [4] as well as, other agrochemicals-insecticides, pesticides [5], antibacterial agents [6], growth stimulators, for improving the fertility and biological activity of the soil, re-cultivation and increasing the production capacity of acid and devastated soils, increasing the nitrogen balance especially in light and sandy soils [4]. Natural zeolites are suitable carriers for fertilizers of various kinds [4]. The nutrients are released gradually, not only in the first year of the vegetation period but also in the second or the following years. Very important is also the water holding capacity of zeolites, which may be used to improve the water balance in soil. In addition, due to their high gas adsorption capacity, zeolites are effective in reducing odors during wastes treatment and composting [7].

Zeolites have been also used as amendments to composts and the soil application shown that zeolite-amended composts are more effective than un-amended [8], while studies, based on results from economic analysis, compost nutrient analysis, temperature results, and crop yield, recommended the use of zeolite in compost at a rate of 5% [9]. Moreover, the addition of natural zeolite to municipal solid waste compost was found to have also beneficial effect on the characteristics of the end product [10].

The present study, which was carried out in the framework of the LIFE11 ENV/GR/951 project entitled “Sustainable strategies for the improvement of seriously degraded agricultural areas: The example of *Pistachia vera* L.- AGROSTRAT”, aims to determine the properties of three composts produced by using pistachio waste and different ratios of goat manure, sheep manure and straw. Clinoptilolite was added to the one of the three composts at a percentage of 5% to investigate the potential beneficial effects as regards compost properties and seeds germination. Considering that the worldwide literature provides rather poor information on the composting of pistachio wastes, this study, although at its initial stage, may significantly contribute to the future valorization and sustainable use of these type of wastes.

2. Materials and methods

2.1 Composts' and wastes' extracts

The three composts were prepared by using solid pistachio wastes from Aegina island, Greece, which were collected during the harvesting period of 2013. The liquid and solid parts of the wastes were separated immediately after their production and the solid part (i.e. raw wastes) was used for the composting. Three mixtures were prepared by using also sheep manure, goat manure, straw and clinoptilolite.

Clinoptilolite used for the experiments comes from a layer situated in Pentalofos, Thrace, Northern Greece. This specific specimen has been used for adsorption and ion-exchange experiments before [11-13] and, consequently, its properties are well known. According to its chemical composition, it is a Ca-rich clinoptilolite with almost no Fe content ($Na_{0.2}K_{0.6}Mg_{0.7}Ca_{2.0}Al_{6.2}Si_{29.8}O_{72}\bullet 19.6H_2O$). Its characterization as clinoptilolite is further supported by the Si/Al ratio, which is equal to 4.8. The estimated Cation Exchange Capacity (C.E.C.) of clinoptilolite, with respect to its formula, is 2,35 meq/g. The XRD spectrum (data not shown) reveals that clinoptilolite, montmorillonite, illite, cristobalite and albite are present in the tuff, whereas the clinoptilolite content is up to 70% [12]. Table 2 includes physical properties of the clinoptilolite used for the present experiments [11-13].

Table 2. Main properties of the clinoptilolite used in the experiments

	Content/value
% Clinoptilolite	69.4
% Cristobalite	5.1
% Illite	7.0
% Montmorillonite	15.0
% Albite	3.0
% Amorphous	-
BET Specific surface area (SSA), m^2/g	31.0
Monolayer value (cm^3/g)	7.12
Specific porous volume, cm^3/g	$6.23 x 10^{-2}$
Pore diameter, (Å)	< 20
Specific value of adsorbed N_2 (cm^3/g)	54.6

Compost A was prepared by initial mixing 57%, 29% and 14% of pistachio wastes, sheep manure and straw, respectively. Compost B by mixing 53%, 37%, 5% and 5% of pistachio wastes, goat manure, straw and clinoptilolite, respectively. Finally, compost C by mixing 43%, 31% and 26% of pistachio wastes, goat manure and straw, respectively.
Composting took place in windrows and lasted 5 months, from November 2013 until March 2014. Temperature, humidity and O_2 content were measured periodically. Temperatures were within the range of 55–70^o C. The composting process was continued until the stability of the compost. The maturity of the composts was determined with a Solvita Compost Maturity kit [14, 15].

Water extracts of the three composts (A, B, and C) and one extract derived from raw solid pistachio waste were also studied as regards their potential nutritional and phytotoxic status. Water extracts were obtained by shaking 50gr of the mature composts and the raw waste with 100 ml deionized water, for six hours at 25^{o}C. Suspensions were then centrifuged at 8,000 rpm for 20 min before filtering [16].

2.2. Chemical analyses-Germination index (GI)

Water extracts were analyzed for pH, electrical conductivity (EC), K, Ca, Mg, Na, K, Fe, Cu, Mn, Zn, polyphenols and phytotoxicity potential.

The pH was measured with a pH meter and the EC with a conductivity meter. Cu, Fe, Mn, Zn, K, Ca and Mg were measured by a Varian AA-220 Atomic Absorption and Na by a Korning Flame photometer. Phenol compounds in the extracts were quantified by means of the Folin–Ciocalteu colorimetric method [17].

The composts and the raw waste sample were analyzed for moisture content, pH, EC, organic matter (OM), total Kjeldahl N, and total polyphenols. The pH was measured using fresh samples extracted with distilled water at an extraction ratio of 1/5-(v/v). The EC was measured in the same water extract after filtration. The solids (50 g) were dried in an oven at a temperature of 75ºC ± 5ºC to constant weight. Then the oven-dried samples were ground (1 mm) for analysis using a laboratory mill. The OM content was calculated by the loss of mass on ignition at 450ºC for 6 h. The total Kjeldahl N concentration was determined using a modified Kjeldahl method based on a sulphuric acid/potassium sulphate digestion with a copper selenium catalyst, using a Kjeldahl digestion unit and a compact distillation unit [18]. Methanol extractable phenol compounds were quantified by means of the Folin–Ciocalteu colorimetric method [17].

The GI was determined in petri dishes and in solutions prepared by diluting the initial extracts and in five replicates. Dilutions used are 0, 1, 3, 10 and 30% of the initial extract.
A filter paper was placed in each petri dish and 1,00 ml of each of the tested solutions is piped into the dish. Eight cress seeds are placed in each petri dish and incubated at 27ºC for 24 hours in dark. One control series is also prepared by using deionized water.
After incubation, germinated seeds are counted (G) and the root length (L) is measured. The GI is calculated according to the formula

$$GI = \frac{G}{G_o} x \frac{L}{L_o} x100$$

where Go and Lo are the germinated seeds and the root length of the control solution and G and L the germinated seeds and the root length of the diluted extracts.

3. Results and discussion

3.1. Composts

The properties of the raw composting materials are given in Table 3. Although pistachio wastes are very rich in organic matter and the C/N ratio could enhance composting, as being within the range of 25-35, however, composting couldn't start without the addition of other raw materials such as manures.
Considering also the high concentrations of K, Ca, Mg, B and the noticeable concentrations of Fe, Zn and Mn, pistachio wastes could be exploited by composting and recycled in the agricultural sector. However, the high values of the EC and of the polyphenols' and sodium's concentrations may restrict their use.

After the composting completion, the three composts appeared dark brown in colour with an earthy smell, deemed necessary for mature compost [19] while their maturity level according to Solvita test was 7 for composts A and B and 8 for compost C, levels that indicate well matured composts.
Average moisture in compost samples (Table 4) varied from 48.5 to 58.3% percent, which may be placed in the high value range (40 to 50) as suggested by Evanylo [20]. Compost B with zeolite has the higher moisture value, probably due to the high water holding capacity of clinoptilolite [7].

Composts' pH is an important parameter for consideration and it should mainly range between 7.2 and 8.5, according to Watson [21]. Composts A and B are well within the stipulated range, while compost C has a pH value lower than the lower limit of 7.2.

Organic carbon content in all three composts ranged between 27.8 and 40.0%, qualifying not only the criteria for field application (16 to 38) as per the range suggested by USCC [22] but also the standard suggested value of >19.4% [23] for nursery application.

Table 3. Parameters of pistachio waste and raw materials used for the preparation of the three composts (A, B, and C).

Parameter	Pistachio wastes	Goat manure	Sheep manure	Straw
Organic matter, %	88.7	91.0	41.0	97.0
Total N, %	1.56	2.13	0.70	0.77
C/N	31.6	23.7	32.5	70.0
Moisture, %	77.9	17.4	49.1	2.00
pH	7.40	8.20	8.90	
EC, mS/cm	6.0	2.92	1.16	
Total Polyphenols, g/kg	2.20	14.0	5.00	
Total K, %	2.77	0.40	0.72	
Total P, %	0.41			
Total Ca, %	3.10	1.80	2.50	
Total Mg, %	0.38	0.23	0.28	
Total Na, %	0.52	0.09	0.14	
Total Cu, mg/kg	31.0	8.00	57.0	
Total Fe, %	0.10	0.01	1.48	
Total Zn, mg/kg	34.0	6.50	360	
Total Mn, mg/kg	104	32.0	560	
Total B, %	0.26			

Table 4. Properties of the three composts (A, B, and C) prepared by pistachio wastes

Parameter	Compost A	Compost B	Compost C
Organic carbon, %	34.4	27.8	40.0
Total N, %	3.2	4.3	2.91
C/N	10.8	6.46	13.7
Moisture, %	48.5	58.3	52.1
pH	8.10	7.90	6.90
EC, mS/cm	6.3	2.6	4.7
Total Polyphenols, g/kg	0.24	0.34	0.30
Maturity level (Solvita test)	7	7	8

The total nitrogen content ranged between 2.9 and 4.3%, which was well above the reference range (1.0 to 2.0%) as suggested by Alexander [24] and Watson [21]. The highest N content (4.3%) as obtained in case of compost B with zeolite might indicate higher fixation of atmospheric N within compost heap during the composting process [25]. Furthermore, it is anticipated that compost B as having the lowest C/N ratio may supply significant quantities of nitrogen as it decomposes.

Electrical conductivity seems to be the significant restricted factor for the safe use of the composts A and C, as being above the stipulated range (<4.0 mS/cm) for saline toxicity as per USCC [22]. This is an anticipated result considering that the irrigation water used in the pilot area comes from wells and drillholes and is characterized by very high EC (i.e. normally ranged between 3,0 and 15,0 mS/cm). Consequently the use for many years of this water for the irrigation of the pistachio trees enriched soil mainly with Na and Cl^- and increased soil EC, which normally ranges (in Aegina island) between 2 and 4mS/cm for surface soil, but between 5 and 12 mS/cm for deeper soil layers.

The addition of clinoptilolite in the composting mixture resulted in a compost (compost B) with acceptable EC, which is below the limit of 4 mS/cm. This achievement is mainly owed to the well-known capability of clinoptilolite to retain cations at its inner and outer framework' sites [11, 12]. The cations are retained on these sites and are slowly released when their concentration in soil solution was decreased due to the consumption by the plants/trees. This behavior does not overload soil with cations, instead, it controls cations presence in soil solution on dependence of plants needs.

Polyphenols contents of the three composts were significantly reduced during composting, while the final concentration could be characterized as acceptable.

3.2. Composts' extracts

Although the three composts' extracts were studied mainly for their phytotoxicity potential, by conducting experiments to determine the Germination Index, thus, important results may also be obtained by this study as regards the potential of these extracts to contaminate/overload soil with high concentration of inorganics or/and polyphenols.
While compost has beneficial aspects, such as providing nutrients to plants, increasing soil organic carbon content, thus, the leachate coming out of the compost itself can be environmentally problematic. Therefore, characterization of the nature of the compost leachate is necessary to predict potential harmful effects of compost applications in the environment. When compost is applied as a soil amendment to agricultural fields, gardens, or roadsides, then soluble and dispersible materials can be leached from the compost by rain or irrigation water [26, 27].

As it can be seen in Table 5 and considering that the results represent one compost/water ratio (i.e. 1:2), the EC values of all extracts, except that of compost B with the zeolite, are significantly high, higher even than the extract of the raw pistachio wastes. However, polyphenols are substantially lower in composts' extracts as compared to the extract of the raw waste. Again, compost B has the lower concentration, which could be characterized as acceptable, considering that the mean soil polyphenols concentration should be lower than 40 mg/kg [28]. All extracts are rich in cations and especially in K. Very low concentration of Cu, Mn, Zn and Mn were detected.

Table 5. Parameters of composts' and pistachio waste's water extracts

Parameter	Compost A	Compost B	Compost C	Raw wastes
pH	7.82	7,76	7.86	7.31
EC, mS/cm	8.42	4.22	18.0	6.01
Total Polyphenols, mg/l	77.7	35.8	87.2	282
K, mg/l	1,300	800	1,690	546
Ca, mg/l	190	250	590	280
Mg, mg/l	80	54	350	150
Na, mg/l	120	130	400	300
Cu, mg/l	0.31	nd	0.08	nd
Fe, mg/l	0.69	0.10	nd	0.78
Zn, mg/l	0.24	0.08	0.08	0.09
Mn, mg/l	0.09	0.02	nd	nd

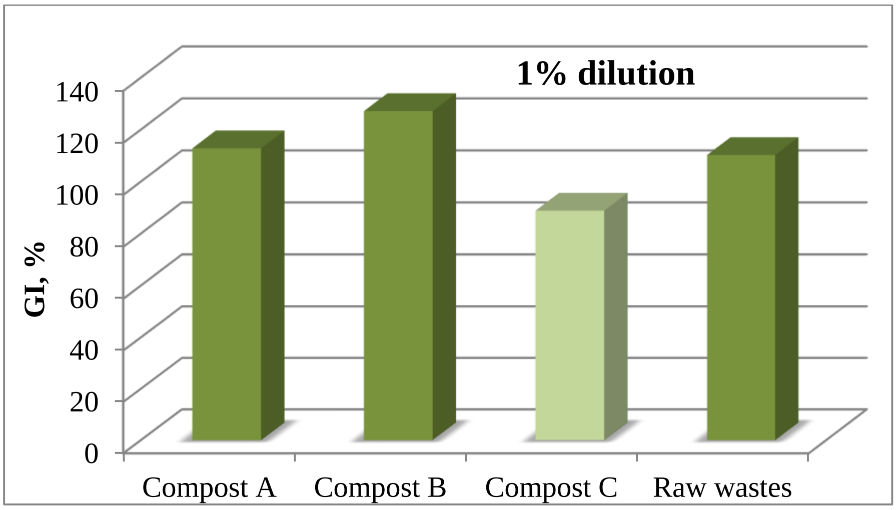

Fig. 1. Germination index at 1% dilution of the composts' and raw waste's water extracts

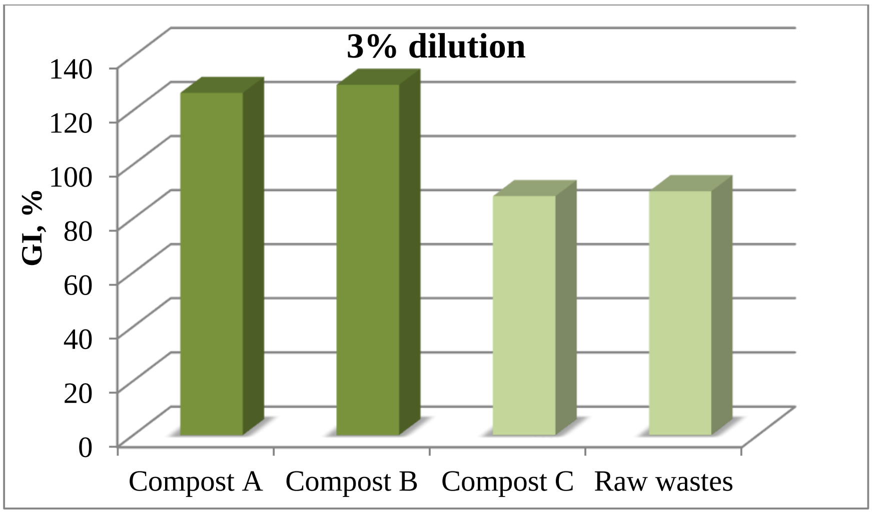

Fig. 2. Germination index at 3% dilution of the composts' and raw waste's water extracts

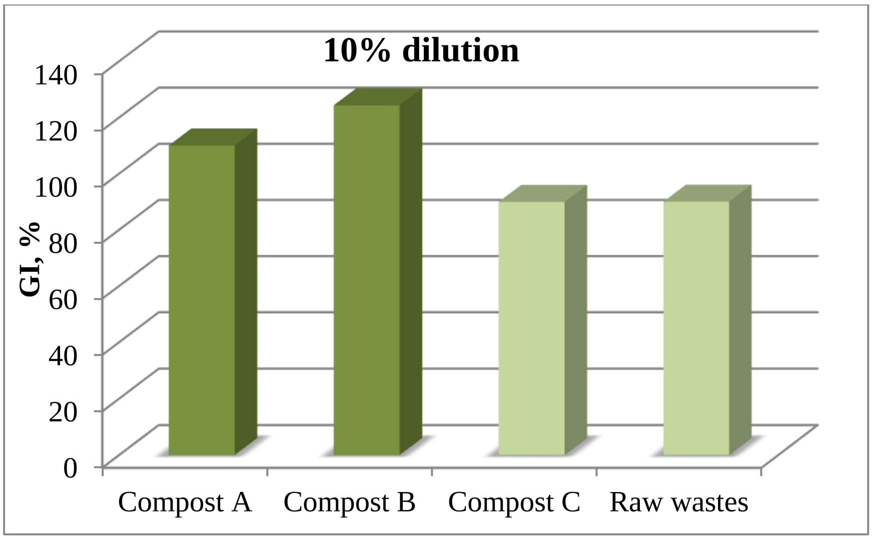

Fig. 3. Germination index at 10% dilution of the composts' and raw waste's water extracts

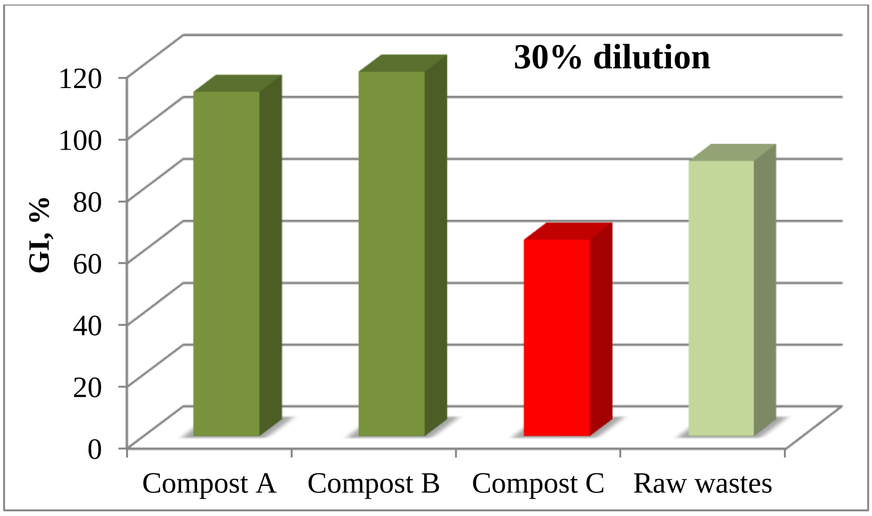

Fig. 4. Germination index at 30% dilution of the composts' and raw waste's water extracts

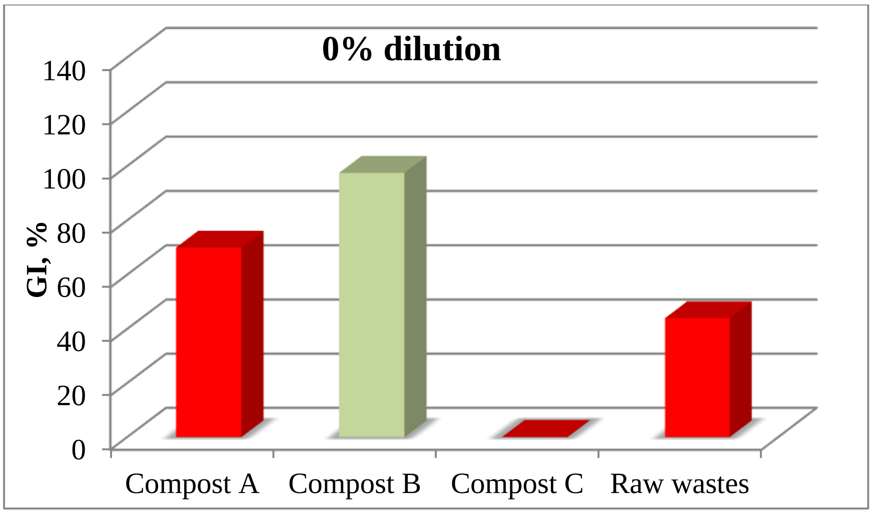

Fig. 5. Germination index at 0% dilution of the composts' and raw waste's water extracts

The composts' extracts were used for the determination of the Germination Index (Figs 1-5). The phytotoxicity bioassay test, as represented by germination index provides a means of measuring the combined toxicity of whatever contaminants may be present [29].

Compost B with the zeolite has the highest GI and can be used as soil amendment without any limitation. For all dilutions, the index was above 100% indicating enhancement in germination and radical growth [30]. Even when the extract was used without dilution, the GI was between 80 and 100% while the index for the other three materials were below 80%, while for compost C the index was almost zero [30]. Compost A shows also promising properties, however the preconditions of its use should be further studied, mainly as regards its high EC. It is worth noting that untreated pistachio wastes have high GI for all dilutions (except when the extract was used undiluted), indicating that they could be used as soil amendments under specific terms, which, however, should be determined. Compost C has the lowest values of GI and thus cannot be proposed to be recycled on soil.

4. Conclusions

Composts produced by mixing pistachio wastes and other raw materials, such as manures and straw are characterized by very good properties as regards their nutritional status and organic matter content. From this point of view, these materials could be excellent soil additives. However, their very high electrical conductivity and also the sodium concentration set some limitations to their use. The addition of 5% clinoptilolite in the compostable mixture results in the production of a high quality compost with low electrical conductivity (i.e. 2.6 mS/cm).

All composts produced during this study had significantly lower polyphenols content in comparison to the untreated pistachio wastes.

The determination of the germination index revealed that the compost which contained 5% clinoptilolite apart from not being phytotoxic, may enhance germination and radical growth. On the contrary, one of the feedstock ratio used for the experiments (compost C) is characterized as potential phytotoxic, while the second one (Compost A) has very good properties as regards its nutritional status and absence of phytotoxic properties, however, its high electrical conductivity may restrict its wide and unlimited use.

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