**Original Research** 

# Effects of Food Industry Wastewater Treatment Sludge on Corn Plant Development and Soil Properties

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### Abstract

In this study, agricultural usage of food industry wastewater treatment plant sludge was researched. Within the scope of our work the study was carried out in field conditions. Before sowing the maize, increasing levels of sludge (0-30-60 and 90 ton ha<sup>-1</sup>) were mixed with the soil. Three times plants were sampled during that period of development and crop harvest. Changes in soil properties caused by sludge application before planting and after harvest and the effect on corn plant growth and mineral content were determined.

According to the analysis results, the zinc contents of sludge used in this study were identified in terms of the value exceeding the limit specified in the regulations. Increasing amounts of sludge applied to soil increased the plant nutrient content, with positive effects on plant growth. Sludge reduced the pH and increased the EC of the soil before planting and after harvest. Together with the application of sludge, mainly to organic matter content, total N,  $NH_4$ -N,  $NO_3$ -N and available P, K some microelement (Zn) and heavy metal content of the soil increased.

Keywords: sludge, lime stabilization, agricultural use, soil properties, plant

## Introduction

Preservation of the natural environment is increasingly important nowadays. This is possible if the waste causing environmental pollution is disposed of in an environmentally friendly manner. Wastewater sludge is called solid, semi-solid or liquid residues, resulting from the physical, chemical and biological processes of domestic and industrial wastewater [1]. Land application of wastewater sludge is an economically attractive waste management strategy, largely promoted by scientists. Furthermore, it has been a socially accepted practice for decades in many parts of the World [2-4].

The land application of wastewater sludge in developed countries is highly controlled in order to prevent risks to public health and the environment. Sewage sludge must be treated and 'stabilized' to reduce odor, pathogen content and vector attraction, and is usually also mechanically dewatered or airdried to reduce bulk, to generate a product acceptable for beneficial use [5]. Sludge treatment may be through

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either one or a combination of the following generic methods:

- Biological processes (anaerobic or aerobic digestion, composting)
- (2) Chemical processes (lime treatment).
- (3) Physical processes (pasteurization, thermal hydrolysis, thermal drying, air or solar drying).

The US and EU has established federal requirements for the safe treatment, beneficial use, and disposal of biosolids. For wastewater sludge that is to be beneficially used, lime stabilization is one of the technologies identified to meet the requirements addressing pathogens.

Incremental sludge from industrial activities, especially without any stabilization, can contain highto-heavy metals and pathogens, bacteria and protozoans that were both harmful to human health and harmful to the odor [6]. For this reason some stabilization methods were applied to wastewater treatment sludges. Stabilization has as its main objectives: the reduction of fermentability, of odor emissions (through chemical and thermal treatment), and of bacteria and suspended solids (through biological process, aerobic or anaerobic). In chemical stabilization the inhibition of the biological reactions is performed by the increase of the pH of the wastewater sludge, up to values of around 12. The stabilization treatment with lime is carried out by mixing the sludge with lime and water, resulting in the formation of an alkaline environment. At the end of the process, pH values above 10 are achieved for a certain period [7, 8]. High pH brings an unfavorable environment for microorganisms. As a result, the sludge does not decompose, the smell disappears and health problems do not come to an end. The reuse of limestabilized sludge is not limited to use on farmland. These sludges have also been used as a soil substitute for landfill cover, for example. Exceptional quality sludges can also be sold to the public for use as commercial fertilizer or soil conditioner. The agricultural utilization of sludge has been considered as an environmentally acceptable management strategy [9]. SS contains high levels of organic matter (20% and more) and essential plant nutrients that can help/enhance crop production [10-13].

Food-based treatment sludge, which is an industrial waste used by human beings, is thought not to have negative features. It also thought that it may be used in agriculture from the view of plant nutrient elements and organic matter [14]. However, the determination of the characteristics of these wastes before their application to the soil will be more beneficial in terms of agricultural use and environmental impact [15]. Otherwise, it was very difficult to remove the negative effects of wastes containing harmful factors for soil ecosystem and plant growth [16]. In this study we tried to determine the possibility of being used in agriculture by taking into account some agricultural characteristics of lime-applied treatment sludge.

#### Experimental

#### Establishment and Execution

The lime-stabilized sludge (LTS) used in the research was taken from Natura Food Industry Inc. Lime treatment is one of the methods sanctioned in the regulations. To meet vector attraction reduction requirements using lime, the pH must be raised to 12 or higher for 2 hours and subsequently maintained above pH 11,5 for another 22 hours without further alkali addition.

The factorial experiment was arranged in a completely randomized block design with three replications per treatment. The plot size was 3 x  $10 = 30 \text{ m}^2$ . In the experimental treatment, the sludge was applied to the soil at 0 (control), 30 (dose 1), 60 (dose 2) and 90 (dose 3) tonnes ha<sup>-1</sup> according to them based on dry matter and mixed with the soil.

The soil used in the experiment had a clay loam texture and the pH indicated that the soil was slightly alkaline, which is common for soils in Turkey. The soil had low organic matter and salt contents. The soil had adequate levels of N, P, K, Fe, Mn, and Zn, and a low Cu content. The limed sludge was applied to the soil and mixed before sowing. At the end of the one-month incubation period, a silage corn variety was cultivated. Within the scope of the experiment, phenological observations were made in the field, and plant leaf samples were taken in 3 sampling periods (V3: three leaf collars, R1: silking and R5: dent-nearly all kernels are denting) and then harvested. The analysis made on plant and grain samples are given below. In order to determine the effect of applied sludge on soil properties, after sludge application pre-plant and postharvest samples from 0-30 cm depth were duly taken from all the plots for chemical analysis.

## Limed-Stabilized Sludge, Soil and Plant Analysis

The wastewater sludge sample and soil were analyzed for basic physicochemical properties using standard procedures and results are presented in Table 1. A portion of the sample to be used in the analysis of the treatment sludge used in the trial was sieved from 2 mm after drying at 65°C. In the case of limed treatment sludge, reaction (pH), electrical conductivity (EC) [17, 18], and sludge organic matter content was determined by ashing at 550°C [19]. Total N according to Kjeldahl method; ammonia (NH<sub>4</sub>-N) [19]; nitrate (NO<sub>2</sub>-N) [20]; total P, K, Na, Mg and Ca; and heavy metals (Pb, Cd, Cr, Ni, Cu, Zn, Fe, and Mn) in the solution obtained by wet digestion with HNO<sub>2</sub> in a microwave oven were determined by Perkin Elmer Optima 2100 DV ICP [21]. According to Table 1, the concentrations of heavy metals in this particular sludge are below the limits imposed by the European and National guidelines for the agricultural utilization of sludge (except for Zn).

Properties	Soil	LTS	Anonim 2010
% clay	43,1		
% loam	27,4		
% sand	29,7		
pH	7,73	12,4	
EC, μS cm <sup>-1</sup>	161,8	3,50	
OM, %	1,37	48,8	
N,%	0,12	3,86	
NH <sub>4</sub> -N, mg kg <sup>-1</sup>	59,37	trace	
NO <sub>3</sub> -N, mg kg <sup>-1</sup>	2,69	trace	
P,%	55,93*	1,63**	
К,%	140,14*	0,48**	
Ca, %	301,21*	30,6**	
Mg, %	252,05*	0,69**	
Na,%	76,23*	0,17**	
Pb, mg kg <sup>-1</sup>	trace	12,1**	750
Cd, mg kg <sup>-1</sup>	1,877*	1,36**	10
Cr, mg kg <sup>-1</sup>	0,071*	110,3**	1 000
Ni, mg kg <sup>-1</sup>	1,199*	16,24**	300
Cu, mg kg <sup>-1</sup>	0,032*	42,03**	1 000
Zn, mg kg <sup>-1</sup>	3,871*	12420**	2 500
Fe, %	43,95*	5,142**	
Mn, mg kg <sup>-1</sup>	2,072*	145,6**	

Table 1. Some analytical values of experimental soil and limestabilized sludge (LTS).

\*. Available (DTPA and NH<sub>4</sub>OAc, mg kg<sup>-1</sup>), \*\*. Total (HNO<sub>3</sub>)

In the study, soil samples were taken pre-planting and post-harvest at 0-30 cm depth, soils were dried in air conditions and sieved from 2 mm. Soil pH and EC were measured with a pH meter and conductivity meter using a water ratio of 1:2.5 w/v respectively. Soil texture was determined using the hydrometer method, lime was determined using the calsimetric method, organic matter content was analyzed according to the modified Walkley-Black method, total N was determined using a Buchi K-437/K-350 digestion/distillation unit according to the Kjeldahl method, and ammonium-N and nitrate-N concentrations were determined with 2 M KCl extracts using the indophenol blue method and the salicylic acid method. Available P was determined using a PG Instruments T60 Split Beam UV/VIS model spectrophotometer according to the molybdenum blue method. Exchangeable cations (Na, K, Ca and Mg) were extracted with ammonium acetate at pH 7,0 and measured using an Eppendorf Elex 6361 model Flame Photometer. Available Fe, Cu, Zn, Mn, Cd, Cr, Ni and Pb were extracted with diethylene triamine pentaacetic Plant and grain samples taken during the growing season and dried at  $65^{\circ}$ C to constant weight in a pneumatic dryer and prepared for analysis by grinding in the plant grinding mill. Total nitrogen in plant samples was determined by the Kjeldahl method. Plant samples were digested with a mixture of nitric acid-hydrogen peroxide (HNO<sub>3</sub> + H<sub>2</sub>O<sub>2</sub>) in a Berghof MWS2 model microwave oven. With the obtained solution, phosphorus (P), sodium (Na), potassium (K), calcium (Ca), magnesium (Mg), and heavy metals (Pb, Cd, Cr, Ni, Cu, Fe, Mn and Zn) were determined by a Perkin Elmer Optima 2100 DV ICP OES [23].

The data obtained from the study were statistically evaluated with the JUMP package program, and the LSD test (p<0,05; p<0,01) was used to compare the differences between the averages.

#### **Results and Discussion**

## Effect of Lime-Stabilized Sludge on Soil Properties

In the study, soil samples were collected before planting and after harvesting from the plots applied to determine the effects of the lime-stabilized sludge (LTS) on the soil properties.

Effects of the limed treatment sludge applications before planting and after harvesting include soil properties such as pH, EC, organic matter, total nitrogen, ammonium and nitrate, available phosphorus and DTPA-extractable microelements (Fe, Cu, Zn ve Mn) and heavy metal contents (Cd, Cr, Ni ve Pb). Differences caused by applications were grouped by the LSD test and reported in the relevant tables.

Changes in soil pH, EC and organic matter content caused by the increasing amount of lime sludge are shown in Table 1 and Fig. 1. The lowest pH value was determined as the average of the control (pH 7.27) and the highest pH value was 90 t ha<sup>-1</sup> (pH 7.85). The increase in pH is attributed to the high concentrations of CaCO<sub>2</sub> in the lime-stabilized sludge. (Table 2). The low pH value of the post-harvest soil was related to mineralization and organic acids. The decrease in soil pH is due to organic acids produced during sludge decomposition. Another explanation is the reduction in soil pH probably due to nitrification of NH<sub>4</sub>-N from the sludge [24]. With regard to the change in the soil pH with the treatment sludge application, the sludge reduced the soil pH value of sludge applied to alkaline or neutral soils reported by researchers [25, 26].

Depending on the increasing application LTS levels, the average pre-planting and post-harvest EC value of the soil increased. The lowest EC value was determined as 221,5  $\mu$ S cm<sup>-1</sup> in control plots not applied to sludge, and the highest value was 645,4  $\mu$ S cm<sup>-1</sup> in the 90 t ha<sup>-1</sup>

eatments ton	5		pł	ł		EC $\mu$ S cm <sup>-1</sup>				O.M. %			
ha-1	В	BS	AH	Means	3	BS	AH	Me	eans	BS	AH	Means	
Control 7,29 7,24 7,27 B				*	219,8	223,1	221,	5 C**	1,85	1,64	1,75 B*		
30	7,	7,64 7,68 7,66 A*			*	371,3	340,0	355,7	BC**	2,03	2,14	2,09 B*	
60	7,	,76	7,60	7,68 A'	*	552,0	356,3	454,2	2 B**	2,28	2,32	2,30 B*	
90	7,	7,97 7,73 7,85 A		*	656,7	634,0	645,4	4 A**	2,96	2,90	2,93 A*		
Means	7,	,67	7,56			449,95	388,35			2,28	2,25		
		·		BS: be	efore	sowing, A	H: After ha	irvest					
Dose		0,234				1	34,5		0,559				
riod		nd				nd				nd			
*P	nd					nd				nd			
	ha <sup>-1</sup> Control 30 60 90 Means Dose iod	Control     7       30     7       60     7       90     7       Means     7       Dose	ha <sup>-1</sup> BS       Control     7,29       30     7,64       60     7,76       90     7,97       Means     7,67       Dose     0,2       iod     n	BS     AH       Control     7,29     7,24       30     7,64     7,68       60     7,76     7,60       90     7,97     7,73       Means     7,67     7,56       Dose     0,234       iod     nd	ha-1         BS         AH         Means           Control         7,29         7,24         7,27 B           30         7,64         7,68         7,66 A           60         7,76         7,60         7,68 A           90         7,97         7,73         7,85 A           Means         7,67         7,56         BS: b           Dose         0,234         0,234	BS     AH     Means       BS     AH     Means       Control     7,29     7,24     7,27 B*       30     7,64     7,68     7,66 A*       60     7,76     7,60     7,68 A*       90     7,97     7,73     7,85 A*       Means     7,67     7,56       BS: before       Dose     0,234       iod     nd     1	BS         AH         Means         BS           ha <sup>-1</sup> BS         AH         Means         BS           Control         7,29         7,24         7,27 B*         219,8           30         7,64         7,68         7,66 A*         371,3           60         7,76         7,60         7,68 A*         552,0           90         7,97         7,73         7,85 A*         656,7           Means         7,67         7,56         449,95           BS: before sowing, A         Dose         0,234         11           iod         nd         11         11	ha <sup>-1</sup> BS         AH         Means         BS         AH           Control         7,29         7,24         7,27 B*         219,8         223,1           30         7,64         7,68         7,66 A*         371,3         340,0           60         7,76         7,60         7,68 A*         552,0         356,3           90         7,97         7,73         7,85 A*         656,7         634,0           Means         7,67         7,56         449,95         388,35           BS: before sowing, AH: After ha         Dose         0,234         134,5           iod         nd         nd         nd	BS       AH       Means       BS       AH       Means         BS       AH       Means       BS       AH       Means         Control       7,29       7,24       7,27 B*       219,8       223,1       221,3         30       7,64       7,68       7,66 A*       371,3       340,0       355,7         60       7,76       7,60       7,68 A*       552,0       356,3       454,3         90       7,97       7,73       7,85 A*       656,7       634,0       645,4         90       7,97       7,56       449,95       388,35       BS: before sowing, AH: After harvest         Bose       0,234       134,5       134,5       134,5       134,5	ha <sup>-1</sup> BS       AH       Means       BS       AH       Means         Control       7,29       7,24       7,27 B*       219,8       223,1       221,5 C**         30       7,64       7,68       7,66 A*       371,3       340,0       355,7 BC**         60       7,76       7,60       7,68 A*       552,0       356,3       454,2 B**         90       7,97       7,73       7,85 A*       656,7       634,0       645,4 A**         Means       7,67       7,56       449,95       388,35	ha <sup>-1</sup> BS       AH       Means       BS       AH       Means       BS       AH       Means       BS         Control       7,29       7,24       7,27 B*       219,8       223,1       221,5 C**       1,85         30       7,64       7,68       7,66 A*       371,3       340,0       355,7 BC**       2,03         60       7,76       7,60       7,68 A*       552,0       356,3       454,2 B**       2,28         90       7,97       7,73       7,85 A*       656,7       634,0       645,4 A**       2,96         Means       7,67       7,56       449,95       388,35       2,28         Dose       0,234       134,5       0         iod       nd       nd       144,5       0	ha <sup>-1</sup> BS       AH       Means       BS       AH       Means       BS       AH       Means       BS       AH         Control       7,29       7,24       7,27 B*       219,8       223,1       221,5 C**       1,85       1,64         30       7,64       7,68       7,66 A*       371,3       340,0       355,7 BC**       2,03       2,14         60       7,76       7,60       7,68 A*       552,0       356,3       454,2 B**       2,28       2,32         90       7,97       7,73       7,85 A*       656,7       634,0       645,4 A**       2,96       2,90         Means       7,67       7,56       449,95       388,35       2,28       2,28       2,25         BS: before sowing, AH: After harvest       BS: before sowing, AH: After harvest       0,559       0,559       0,559         iod       nd       nd       nd       nd       nd       nd       134,5       0,559	

Table 2. Effects of lime-stabilized sludge on pH, EC and organic matter content.

application. The increase in EC value due to increasing levels is due to the chemical properties of the sludge and the mineralization due to time. The increase in salinity is one of the most important factors to be considered in agricultural applications [27, 28]. Excessive application of soil may lead to the EC limit of salinity. According to the pre-sowing of the soil

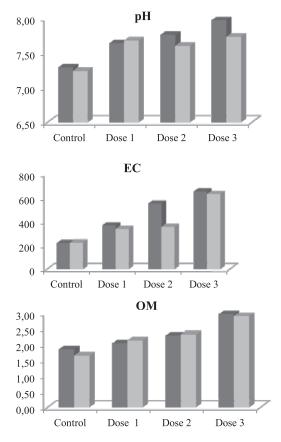


Fig. 1. Effects of lime-stabilized sludge on pH, EC and organic matter content under increasing levels of pre-planting and post-harvest soil.

samples taken after harvest, EC value decreased. This may be related to plant intake.

The soil organic matter value increased as averages before planting and after harvest, depending on the increasing application levels. The lowest organic matter value was 1,75% for the untreated control plot and the highest value was 2,93% for the 90 t ha<sup>-1</sup> application. Increasing levels of sewage sludge have been identified in many studies that increase soil organic matter content. Wastewater treatment sludge can be used to solve organic matter deficiencies, especially due to the high organic matter content [29]. In addition, the mineralization of the more easily degradable forms of sludge organic matter by soil microorganism is rapid, depending upon soil conditions [30].

The changes that the LTS brings to the soil N (%),  $NH_4$ -N (mg kg<sup>-1</sup>) and NO<sub>3</sub>-N (mg kg<sup>-1</sup>) and the available P (mg kg<sup>-1</sup>) value are given in Table 3 and Fig. 2.

The soil N % values increased before and after harvest depending on the increased application levels. The lowest N value was determined in the control plot, and the highest value was found to be 0,17% and 0,28% at 90 t ha<sup>-1</sup> application. The sludge doses and the application of fertilizer to the total N content of soils were statistically significant in that the sludge was applied at increasing rates, increasing the total nitrogen content as well as that the soil was in the organic matter content. Numerous studies have been carried out to investigate the effects of sludge applications on plant nutrients in the soil [31-33].

The average values of the NH<sub>4</sub>-N and NO<sub>3</sub>-N (mg kg<sup>-1</sup>) increased before sowing and after harvest depending on the increasing application levels. The highest values were determined at 90 t ha<sup>-1</sup> application. This increase was related to the chemical properties of the sludge. The increase in the amount of NO<sub>3</sub>-N (mg kg<sup>-1</sup>) in the soil due to the time of incubation was related to nitrification [23]. Soil type, sludge properties and soil pH, temperature, aeration

Treatment	Treatments tons		N, %	NH <sub>4</sub> , mg kg <sup>-1</sup>			NO <sub>3</sub> , mg kg <sup>-1</sup>			P, mg kg <sup>-1</sup>			
ha-1		BS	AH	Means	BS	AH	Means	BS	AH	Means	BS	AH	Means
Contro	Control		0,17	0,17 B*	22,4	30,9	26,7	3,6	3,0	3,3	51,2	49,4	50,3 B*
30		0,20	0,20	0,20 B*	34,4	32,1	33,3	3,6	4,9	4,3	51,0	57,9	54,5 B*
60		0,23	0,20	0,21 B*	59,1	33,0	46,1	3,9	5,4	4,7	62,0	48,3	55,2 B*
90		0,28	0,28	0,28A*	52,2	38,1	45,2	7,7	6,2	7,0	109,2	75,7	92,5 A*
Mean	IS	0,22	0,22		42,0	33,5		4,7	4,9		68,4	57,8	
				BS: b	efore so	owing, A	H: After	harvest					
LSD	Dos	e	0,5	57			nd			nd			29,05
Po	Period		n	d			nd			nd			nd
I	D*P		n	d			nd			nd			nd

Table 3. Effects of lime-stabilized sludge on soil N%, NH<sub>4</sub> NO<sub>3</sub>, available P content.

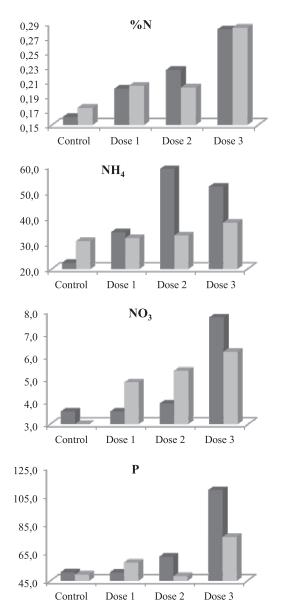


Fig. 2. Effects of lime-stabilized sludge on soil N,  $NH_{4}$ ,  $NO_{3}$  and available P content under increasing levels.

and humidity conditions were especially influential on the final organic nitrogen mineralization of sludge applications. Therefore, it was important to know the organic and inorganic forms of nitrogen in order to determine the nitrogen fertilizer value of the treatment sludge, which was considered to be applied to the soil and to predict how the organic nitrogen will be mineralized during the plant production period [34]. For example, about the subject the rate of agricultural application of biosolids is determined by the nitrogen limited biosolids application rate, where the amount of PAN (plant-available N) applied in biosolids (calculated according to Eq. (PAN<sub>plant available N</sub> =  $NH_4$ -N+[NO<sub>3</sub>- $N+NO_{2}-N$  + mineralizable N) must not exceed the crop N requirement in the year of application. It is important to consider the above formula [35, 36].

Depending on the increased application levels, before planting and after harvest soil was determined at the lowest available P values of 50,3 mg kg<sup>-1</sup> and the highest P value of 92,5 mg kg<sup>-1</sup> at 90 t ha<sup>-1</sup> application. Phosphorus availability in sludge is strongly influenced by the wastewater treatment (WWT) processes [37]. The addition of some type of liming agent to stabilize sludge may result in lower total P. The addition of lime was reported to increase sludge pH and decrease the solubility of P by the formation of recalcitrant caphosphate minerals [38]. Since more stringent N and P discharge limits have been implemented in wastewater treatment plants (WWTP) in environmentally sensitive areas, total P in sludge is expected to increase from current values [39]. Heavy applications of wastewater sludge in order to supply sufficient inorganic N and meet crop N demands will most probably oversupply P [40].

The changes that the lime-stabilized sludge caused to the soil-exchangeable Na, K, Ca and Mg values are given in Table 4 and Fig. 3 The Na, K, Ca and Mg values increased as the before sowing and after harvest averages, depending on the increased application

ents		Na, mg kg <sup>.</sup>	-1		K, mg kg <sup>-1</sup>			Ca, mg kg <sup>-1</sup>				Mg, mg kg <sup>-1</sup>		
na <sup>-1</sup>	BS	AH	Means	BS	AH	Means	BS	AH	Means	BS	AH	Means		
ol	75c*	63c*	691C*	139	203	171B*	287 d*	237 e*	262 C**	264	272	268 B*		
	152b*	84c*	118 B*	143	336	240B*	354 bc*	318 cd*	336 B**	260	276	268 B*		
	248a*	93bc*	170 A*	201	276	238B*	461 a*	338 bc*	399 A**	303	316	309 A*		
	277a*	97bc*	187A*	308	483	395A*	471 a*	369 b*	420 A**	329	346	338 A*		
ıs	188A**	84,2B**		198 B*	324 A*		393 A**	315 B**		289	303			
				BS: bef	ore sowin	g, AH: Afte	r harvest							
Dose			46,59			86,55			33,75			37,86		
od			32,95			61,20			23,86			nd		
þ			65,89			nd			47,73			nd		
	a <sup>-1</sup> ol s Dose od	a <sup>-1</sup> BS       ol     75c*       152b*       248a*       277a*       s     188A**       Dose       od	a <sup>-1</sup> BS         AH           bl         75c*         63c*           152b*         84c*           248a*         93bc*           277a*         97bc*           s         188A**         84,2B**	a <sup>-1</sup> BS         AH         Means           bl         75c*         63c*         691C*           152b*         84c*         118 B*           248a*         93bc*         170 A*           277a*         97bc*         187A*           s         188A**         84,2B**           Dose         46,59           od         32,95	a <sup>-1</sup> BS         AH         Means         BS           bl         75c*         63c*         691C*         139           152b*         84c*         118 B*         143           248a*         93bc*         170 A*         201           277a*         97bc*         187A*         308           s         188A**         84,2B**         198 B*           BS:         bef         058         46,59           od         32,95         100         32,95	a <sup>-1</sup> BS         AH         Means         BS         AH           bl         75c*         63c*         691C*         139         203           152b*         84c*         118 B*         143         336           248a*         93bc*         170 A*         201         276           277a*         97bc*         187A*         308         483           s         188A**         84,2B**         198 B*         324 A*           BS: before sowin           Dose         46,59         46,59           od         32,95         1         308	$a^{-1}$ BS         AH         Means         BS         AH         Means $bl$ 75c*         63c*         691C*         139         203         171B*           152b*         84c*         118 B*         143         336         240B*           248a*         93bc*         170 A*         201         276         238B*           277a*         97bc*         187A*         308         483         395A*           s         188A**         84,2B**         198 B*         324 A*           BS: before sowing, AH: After           Dose         46,59         86,55           od         32,95         61,20	$a^{-1}$ BSAHMeansBSAHMeansBSol75c*63c* <b>691C</b> *139203 <b>171B</b> *287 d*152b*84c* <b>118 B</b> *143336 <b>240B</b> *354 bc*248a*93bc* <b>170 A</b> *201276 <b>238B</b> *461 a*277a*97bc* <b>187A</b> *308483 <b>395A</b> *471 a*s188A**84,2B**198 B*324 A*393 A**BS: before sowing, AH: After harvestDose46,5986,55od32,9561,2061,20	$a^{-1}$ BSAHMeansBSAHMeansBSAHol75c*63c*691C*139203171B*287 d*237 e*152b*84c*118 B*143336240B*354 bc*318 cd*248a*93bc*170 A*201276238B*461 a*338 bc*277a*97bc*187A*308483395A*471 a*369 b*s188A**84,2B**198 B*324 A*393 A**315 B**BS: before sowing, AH: After harvestDose46,5986,55od32,9561,2011	$a^{-1}$ BSAHMeansBSAHMeansBSAHMeansol75c*63c*691C*139203171B*287 d*237 e*262 C**152b*84c*118 B*143336240B*354 bc*318 cd*336 B**248a*93bc*170 A*201276238B*461 a*338 bc*399 A**277a*97bc*187A*308483395A*471 a*369 b*420 A**s188A**84,2B**198 B*324 A*393 A**315 B**56533,75od46,5986,5533,7533,7533,7533,8632,9561,2023,86	$a^{-1}$ BSAHMeansBSAHMeansBSAHMeansBSol75c*63c*691C*139203171B*287 d*237 e*262 C**264152b*84c*118 B*143336240B*354 bc*318 cd*336 B**260248a*93bc*170 A*201276238B*461 a*338 bc*399 A**303277a*97bc*187A*308483395A*471 a*369 b*420 A**329s188A**84,2B**198 B*324 A*393 A**315 B**289BS: before sowing, AH: After harvestDose46,5986,5533,75od32,9561,2061,2023,86	$a^{-1}$ BSAHMeansBSAHMeansBSAHMeansBSAHol75c*63c*691C*139203171B*287 d*237 e*262 C**264272152b*84c*118 B*143336240B*354 bc*318 cd*336 B**260276248a*93bc*170 A*201276238B*461 a*338 bc*399 A**303316277a*97bc*187A*308483395A*471 a*369 b*420 A**329346s188A**84,2B**198 B*324 A*393 A**315 B**289303BS: before sowing, AH: After harvestDose46,5986,5533,750od32,9561,2023,86000		

Table 4. Effect of increasing amounts of LTS on the exchangeable Na, K, Ca and Mg contents.

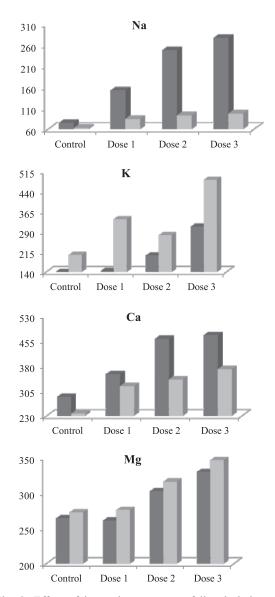


Fig. 3. Effect of increasing amounts of limed sludge on the exchangeable Na, K, Ca and Mg contents of pre-planting and post-harvest soil.

levels. The lowest values were 69.1, 171.8, 262.0 and 268.5 mg kg<sup>-1</sup>, respectively, and the highest values were 187.0, 395.9, 420.4 and 338.4 mg kg<sup>-1</sup> as determined at 90 t ha<sup>-1</sup> application.

Sludges contain N and P as well as more or less K, Ca, Mg and Na plant nutrients [41, 42]. It should be noted that in the course of application to the soil, the amount of these elements, together with the application, was given to the soil and does not disturb the nutrient balance in the soil. In the studies carried out, the low correlation between the amounts of Ca, Mg, Na and K of the soil and the plant was obtained due to the treatment sludge applications [24].

The changes caused by lime-stabilized sludge in soil DTPA-extractable Fe, Zn, Mn and Cu values are shown in Table 5 and Fig. 4. Depending on the increasing application levels, the average content of Fe, Zn, Mn, and Cu can be increased before sowing and after harvesting. The lowest values were determined as 24,8, 5,2, 24,9 and 3,3 mg kg<sup>-1</sup> in control, while the highest values were 28,7, 119,9, 29,2 and 4,1 in 90 tonnes<sup>-1</sup> respectively. When the changes in the microelement content of the soil before sowing and after harvesting are examined, it is seen that the most significant change is in Zn. The reason for this change is due to the high Zn content of the calcareous sludge used (Table 1). Zn content of this food industry treatment sludge is very high [41, 42].

Fig. 5 shows the changes in the amount of DTPAextractable heavy metals (Ni, Cr, Pb, Cd) in the preplanting and post-harvest soil. The change was not statistically significant. This situation was caused by the increase in the organic matter content of the soils depending on the application levels and the formation of complex compounds due to mineralization and the change of the chemical properties of the treated sludge by the lime application. This possibly indicates that the decomposition of organic matter in the soil did not control the availability of DTPA extractable metals,

Treat	ments		Fe, mg l	<b>cg</b> <sup>-1</sup>		Zn, mg kg	l	Ν	In, mg kg	g <sup>-1</sup>		Cu, mg k	g <sup>-1</sup>
tons	ha-1	BS	AH	Means	BS	AH	Means	BS	AH	Means	BS	AH	Means
Cor	ntrol	23,4	26,3	24,8	4,7 c**	5,7c**	5,2 C**	25,0	24,9	24,9	3,35	3,25	3,3 B*
3	30	24,0	25,4	24,7	15,3 c**	48,7 bc**	32,0 BC**	24,9	25,6	25,2	3,68	3,86	3,8 AB*
6	50	28,0	26,3	27,2	43,2 bc**	87,8 b**	65,5 B**	27,4	26,8	27,1	3,72	4,10	3,9 A*
9	90	31,2	26,3	28,7	52,6 bc**	187,1 a**	119,9 A**	32,2	26,2	29,2	4,03	4,16	4,1 A*
Me	eans	26,7	26,1		28,9 B**	82,3 A**		27,4	25,9		3,70	3,84	
					BS: bef	ore sowing,	AH: After ha	rvest					
LSD	Dose			nd		32,39				nd			nd
Pe	eriod			nd		45,81				nd			nd
D	*P			nd		64,78				nd			nd

Table 5. Effect of increasing amounts of limed sludge on Fe, Zn, Mn, and Cu contents of the soil.

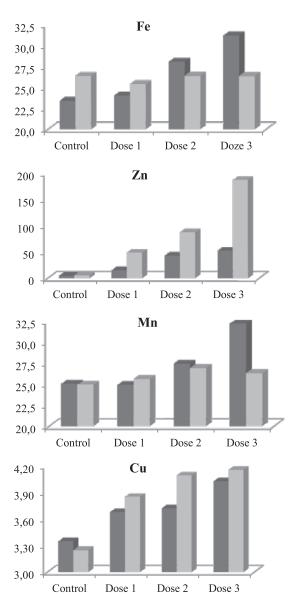


Fig. 4. Effect of increasing amounts of limed sludge on the Fe, Zn, Mn and Cu contents of pre-planting and post-harvest soil.

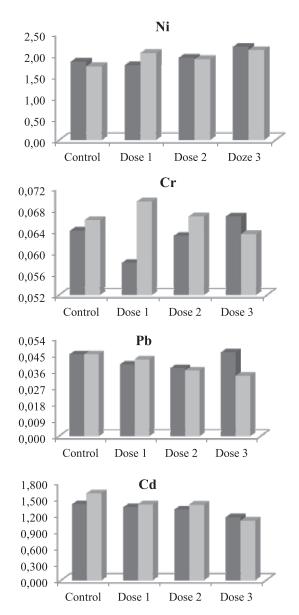


Fig. 5. Effect of increasing amounts of limed sludge on the Ni, Cr, Pb and Cd contents of pre-planting and post-harvest soil.

Treat	ments tons	Ni, mg kg <sup>-1</sup>				Cr, mg kg <sup>-1</sup>			Pb, mg kg <sup>-1</sup>			Cd, mg kg <sup>-1</sup>		
ha-1		BS	AH	Means	BS	AH	Means	BS	AH	Means	BS	AH	Means	
C	Control	1,83	1,72	1,78	0,064	0,066	0,065	1,392	1,595	1,494	0,045	0,045	0,045	
	30	1,75	2,03	1,89	0,058	0,069	0,064	1,338	1,393	1,366	0,039	0,042	0,041	
	60	1,93	1,89	1,91	0,063	0,067	0,065	1,295	1,388	1,342	0,037	0,036	0,037	
	90	2,18	2,10	2,14	0,067	0,063	0,065	1,155	1,089	1,122	0,046	0,033	0,040	
Ν	Means.	1,92	1,94		0,063	0,066		1,295	1,366		0,042	0,039		
				BS:	before so	owing, AF	I: After har	vest						
LSD	Dose			nd			nd			nd			nd	
Period				nd			nd			nd			nd	
D*P				nd			nd			nd			nd	

Table 6. Effect of increasing amounts of limed sludge on the Ni, Cr, Pb and Cd contents of pre-planting and post-harvest soil.

and thus metals were strongly fixed in the inorganic fractions of the sludge, becoming more insoluble due to adsorption/precipitation processes. Strong retention of metals is often attributed to the inorganic fraction of sludge, while the strength and capacity of metal sorption by sludge can be increased through the addition of lime, as reported in previous studies [43].

The release, movement and uptake of heavy metals concomitant with wastewater sludge are influenced by a number of factors such as soil type on which wastewater sludge is applied, pH, soil organic matter, redox potential, and metal bindings [44]. In particular, the amount of extractable heavy metals has significantly decreased in the lime-stabilized sludge. No increase was observed in heavy metal content of soil due to immobilization at increasing levels of sludge applications.

## Effects of Lime-Stabilized Sludge on Plant Growth

The results related to changes caused by lime treatment sludge on plant growth, grain and root nutrients and heavy metal content were given and grouped according to LSD test in Tables 7 and 8. When Table 7 is examined, it is determined that the plant P, K, Mg and Ca contents have increased depending on increasing application levels, but decreased depending on time except Ca. Root nitrogen content also increased due to the increased application level. Increases depending on the application levels are related to the chemical properties of the sludge.

The changes in the nitrogen content of the plant due to increasing levels are insignificant, which may be caused by the applied lime to the sludge, which decreased the availability of nitrogen. Depending on the sampling periods, the decrease in the plant nutrient elements is considered to be due to the fact that the plant nutrient intake is fast at the beginning of development and decreases depending on time and dilution effect.

They reported a rapid decrease in the period of development and fluctuations in N and P in later periods. This change seen in the leaves is consistent with the reports of the study [45, 46]. K reached peak value (2.360%) at the time when photosynthesis and water uptake were highest, then decreased and remained almost stable in other periods. K is not located in the structure of organic matter and is involved in the transport of water, especially of the plant and the transportation of photosynthesis products [47].

It is thought that Ca increases continuously depending on time related to the application of lime to the sludge and increase of Ca content. Calcium is transported to the leaves with transpiration. Although N, P, Mg and K ions can be easily carried in the phloem, some researchers have indicated that the Ca ion is transported very slightly and slowly and it reaches the newly developing meristematic tissues by transpiration, in xylem, As a result of very little or no phloem transport of calcium and the fruits did not transpirate, Ca cannot be moved to newly developing tissues such as fruits, and a continuous increase was observed in the leaves [48].

When Table 8 was examined, the content of plant grain and root Fe increased with increasing application levels. The Cu content of the plant increased depending on time, but an increase also was observed in the Cu content of the root. The most pronounced effect in the study was in the plant Zn content. The Zn content of the plant has increased significantly depending on the application levels and sampling periods. Similarly, it increased with time. it is important to note that the Zn availability in soil shows a good correlation with the amount of this nutrient in the plant shoots [49]. However, Zn content in plants in soil amended with alkaline sewage sludge contrasted with a decrease of Zn with the isolated application of lime was expressed [50].

			Sa	mpling Periods			
			N	itrogen (N), %			
Tons ha-1		V3	R1	R5	Means	Grain	Root
Control		2,598	2,845	2,053	2,499	1,453	1,064C*
30		2,786	2,714	1,966	2,489	1,516	1,167BC*
60		2,95	2,784	1,995	2,576	1,546	1,382AB*
90		2,694	2,789	2,062	2,515	1,596	1,611A*
Means	2	,757A**	2,783A**	2,019B**			
LSD Dose					nd	nd	0,238
Period					0,197		
			Pho	osphorus (P), %		·	
Control		0,196	0,180	0,159	0,178	0,187	0,176
30		0,203	0,183	0,159	0,182	0,193	0,203
60		0,253	0,183	0,160	0,199	0,187	0,199
90		0,258	0,236	0,181	0,225	0,164	0,185
Means		0,228	0,196	0,165			
LSD Dose		-			nd	nd	nd
Period		-			nd		
			Po	tassium (K), %	<u>,                                     </u>	I	
Control		2,197	1,084	1,000	1,427B*	0,151	1,913
30		2,481	1,125	1,076	1,561AB*	0,164	1,977
60		2,378	1,213	1,127	1,573A*	0,163	1,993
90		2,383	1,223	1,277	1,628A*	0,132	2,294
Means	2	,360A**	1,161B**	1,120 B**			
LSD Dose					0,134	nd	nd
Period					0,116		
			Ca	alcium (Ca), %	<u>,                                     </u>	I	
Control		0,282	0,485	0,601	0,456B*	0,012	0,056C*
30		0,366	0,534	0,589	0,496AB*	0,012	0,183B*
60		0,399	0,545	0,624	0,523A*	0,011	0,265B*
90		0,466	0,590	0,626	0,561A*	0,020	0,529A*
Means	0	,378C**	0,539B**	0,610A**			
LSD Dose					0,065	nd	0,121
Period					0,056		
			Mag	nesium (Mg), %	<u>,                                     </u>	I	
Control		0,112	0,074	0,065	0,084	0,087	0,175C**
30		0,125	0,078	0,060	0,088	0,084	0,268B**
60		0,128	0,075	0,070	0,091	0,079	0,328A**
90		0,141	0,055	0,068	0,088	0,066	0,247B**
Means	0	,127A**	0,071B**	0,066 B**			
LSD	Dose				nd	nd	0,025
Peri	od	1			0,021		

Table 7. Effect of lime treatment sludge on macro elements in corn plants.

			Sampling Periods			
	V3	R1	R5	Means	Grain	Root
			Iron (Fe), mg kg <sup>-1</sup>			
Control	102,41	103,69	106,90	104,33B*	14,023B*	6277B*
30	108,70	107,93	107,97	108,20B*	14,547B*	7262B*
60	107,14	121,50	110,08	112,91B*	15,253B*	7292B*
90	119,40	128,93	112,90	120,41A*	19,483A*	10358A*
Means	109,41	115,51	109,46			
LSD Dose				10,576	9,570	2325,3765
Period				nd		
			Copper (Cu), mg kg	-1		
Control	9,135	9,358	10,434	9,642	0,917	13,597
30	9,850	10,870	10,151	10,290	1,269	14,793
60	8,651	10,316	11,023	9,997	1,139	15,280
90	8,407	10,363	11,238	10,003	1,389	16,457
Means	9,012B*	10,227A*	10,712A*			
LSD Dose				nd	nd	nd
Period				1,104		
		1	Zinc (Zn), mg kg-1			
Control	35,420	47,680	56,830	46,643 B**	18,937	43,08C**
30	34,257	48,843	59,703	47,601 B**	21,243	128,65B**
60	40,713	62,923	80,660	61,432 A**	21,277	205,20B**
90	51,737	68,667	61,217	60,540 A**	20,677	494,33A**
Means	40,531C**	57,028B**	64,603A**			
LSD Dose				8,463	nd	80,068
Period				7,329		
	1	Ma	anganese (Mn), mg	kg-1		1
Control	41,017	68,277	86,843	65,379	7,810	165,97B**
30	40,560	80,623	97,200	72,794	7,968	187,27A**
60	41,533	85,780	99,673	75,662	6,725	177,70AB**
90	45,133	80,063	81,857	69,018	6,213	162,77B**
Means	42,061C**	78,686B**	91,393A**			
LSD Dose				nd	nd	17,219
Period				8,552		
	1	C	admium (Cd), mg k	ςg <sup>-1</sup>		
Control	0,052	0,027	0,030	0,036	0,053	0,138
30	0,052	0,034	0,049	0,045	0,061	0,102
60	0,051	0,051	0,054	0,052	0,076	0,139
90	0,064	0,052	0,063	0,060	0,104	0,138
Means	0,055	0,041	0,049			

ne o. Continueu.						,
LSD Dose				nd	nd	nd
Period				nd		
		Cl	hromium (Cr), mg	kg <sup>-1</sup>		
Control	2,340	4,591	3,620	3,517C*	0,427	56,053
30	2,676	3,314	3,639	3,210BC*	0,436	40,193
60	2,795	3,976	3,343	3,37AB*	0,445	43,627
90	3,662	3,893	3,629	3,728A*	0,577	52,563
Means	2,868	3,944	3,558			
LSD Dose				nd	nd	nd
Period				0,451		
			Nickel (Ni), mg kg	-1		•
Control	1,229	1,353	1,247	1,276	0,675B*	35,833
30	1,263	1,065	1,095	1,141	1,573A*	43,337
60	1,288	1,323	1,153	1,255	1,851A*	33,940
90	1,458	1,348	0,958	1,255	1,947A*	39,917
Means	1,310A*	1,272A*	1,113B*			
LSD Dose				nd	0,640	nd
Period				0,149		
			Lead (Pb), mg kg-	1		
Control	0,609	0,580	0,649	0,613A*	0,690	4,578C*
30	0,383	0,521	0,623	0,509AB*	0,844	5,657B*
60	0,580	0,56	0,137	0,426BC*	0,887	4,647C*
90	0,471	0,576	0,034	0,360C*	0,880	6,652A*
Means	0,511	0,559	0,361			
LSD Dose				0,111	nd	0,845
Period				nd		

Table 8. Continued.

\*. p<0,05, \*\*. p<0,01

The changes that occurred in the Cd content of the plant depending on the applications and sampling periods were not significant. The Cr content of the plant increased with both the application levels and the sampling periods. Ni and Pb content decreased with sampling periods and increasing levels of application. However, the amount of Ni in grain increased with increasing application levels. Increases in the amount of plant root heavy metal were observed in the study, depending on the increasing levels.

It is well documented that wastewater sludge application to soils substantially increases nutrient content and crop growth [51] as well as improves soil properties. [52] noticed that the accumulation of heavy metals in the treated crops were under allowable limits. On the contrary, [53] noticed that heavy metals were increased above allowable limits in soil and plants by increasing sludge applications. Certain plant mechanisms render the decrease in uptake at high metal loadings [54]. These mechanisms include exclusion of metals, limited translocation from root to shoot and saturation of the metal transport channels at high metal concentrations [55]. Negative effects of sludge application such as elevated heavy metal content in parts of plants resulting from the use of lime-stabilized sludge must also be taken into consideration.

#### Conclusions

It was known that when wastewater treatment sludges were applied to the soil for agricultural purposes, the soil salinity and heavy metal content increases in particular. This may lead to increases in heavy metal content due to increased application levels. According to the results obtained within the scope of the study; applied food-based and lime-applied sludge, increased soil organic matter, N and P contents at the point of improvement of soil properties. The most notable change has come to the zinc content of the soil and the plant. It was known that this is an important problem in agricultural productivity in which zinc was a plant nutrient and the zinc content of agricultural soils was generally low and fertilizing was needed. It can be concluded that the so-called treatment sludge, which was thought to have no adverse properties, can be applied in soil with calcareous and low zinc content.

In general, lime stabilization decreased the total extractability of heavy metals, indicating a reduced bioavailability of these elements. This is particularly important for safe utilization in agricultural soils in order to reduce the transfer of heavy metals to the food chain. However, at the point of these applications it was necessary to determine the optimum application level for different soil characteristics and plants.

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### **Conflict of Interest**

The authors declare no conflict of interest.

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