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Geostatistical Investigation of Sequentially Extracted Zn Forms at Field Scale in Highly Calcareous Soils

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Abstract: Evaluation agricultural land management practices require a through knowledge of soil spatial variability and understand their relationship. The objectives of this study was to estimate, the distribution of Zinc (Zn) fractions in calcareous soils in Bajgah district, Fars province, Southern Iran, for recognize the mobility and bioavailability of Zn chemical fractions in the field scale. Five different kinds of chemical forms of Zn in the surface soil of this area analyzed by the method of a combination of geostatistic method and sequential extraction. Soil properties will have a major effect on elements mobility. A sequential extraction procedure was used to fractionate chemical forms of Zn. The forms determined were exchangeable, sorbed, organic, carbonate, residual and sum of forms. The spatial distribution and spatial dependence level varied within location. The range of spatial dependence was found to vary within soil parameters. Generally, exchangeable form had the shortest range of spatial dependence (70.50 m) and residual Zn had the longest (368.10 m).

Key words: Kriging, zinc, chemical fractionation, sequential extraction, geostatical, soil properties

INTRODUCTION

Site-specific management of nutrients gives the farmer the potential to apply the exact requirement of nutrients at each given location in a field. Also, can be viewed as a cyclical process of within field data collection, data analysis, optimum decision making, variable rate application and evaluation. It is generally recognized that information about the physicochemical forms of elements is required for understanding their environmental behavior, including mobility, pathways and bioavailability (Tack and Verloo, 1995). In soils, elements of interest exist in several different forms and are associated with a range of components (Cottenie *et al*., 1982). Recently, fractionation studies of metallic elements in soils using a sequential extraction procedure can provide an understanding of its chemical fractions and potential bioavailability. Usually, the fractions considered are as follows: exchangeable, organically complexes carbonate bound, iron and manganese hydroxides linked and residual fractions (Kabala and Singh, 2001; Kersten and Förstner, 1995). Different fractions of soil metal vary considerably in their chemical reactivity and bioavailability. The water-soluble and exchangeable forms of metals are considered to be the most available to plants (Shuman, 1991) and metals bound to organic matter are also found to be available to plants (Iyengar *et al*., 1981). Zinc is one of the seven micronutrients essential for crop growth. The deficiency of zinc causes shortage of proteins, which results in plant dwarf ness, delay in ripening and ultimate loss in yield. Globally, 30% of cultivated soils, which has implication for plant growth and nutrition and nutritive value of crops. With almost half of word's population at risk of inadequate Zn intake (Brown and Wuehler, 2002), there is need to increase dietary intake in many regions of highly calcareous soils in Iran. The mobility, transport and partitioning of Zn in soils are dependent on various soil chemical properties that include soil pH, the type and contents of clay minerals and the contents of Al and Fe oxides, carbonates and organic matter (Yasrebi *et al*., 1994; Basta *et al*., 2001). In earlier studies, the sequential extractions of soils demonstrated a positive and significant correlation between Zn amounts extracted with DTPA and the sum of exchangeable and carbonate fractions (Li and Shuman, 1996; Obrador *et al*., 2003). Yasrebi *et al*. (1994) found that Zn in calcareous soils was mainly in the residual fraction (88%). Shuman (1985) reported that Zn was mainly in the crystalline iron oxide fraction and the residual fraction. More than 47% of the land in India is deficient in Zn due

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to the fact that 80-96% of the Zn is in the residual fraction (Singh *et al*., 1988). Han and Zhu (1992) reported that zinc in calcareous paddy soils in China was predominantly in the residual fraction (97-99%). Geostatistics have recently been used as a tool for soil investigation, mapping and helpful to study spatial variability in soil-management practices. Spatial dependence has been observed for a wide range of soil physical, chemical and biological properties and processes (Lyons *et al*., 1998; Raun *et al*., 1998). Soil nutrient variability mapping has been reported as an important component for establishing management zones (Castrignano *et al*., 2000). There is little information available in spatial distribution of zinc fractions and the relationship with available Zn in calcareous soils. Geostatistical approach has been popularly applied to analyze spatial structure and spatial distribution of soil heavy metals (Liu *et al*., 2006; Saby *et al*., 2006). Since, limited information is available for description of spatial variability of elements fractionation in the field-scale. The main objectives of this study were to investigate the spatial dependency of Zn forms at field scale in agriculturally calcareous soils in Bajgah district, Southern Iran, estimate the distribution of Zn forms at field scale in agriculturally used soils for recognize the mobility and bioavailability of zinc fractions in the study area.

MATERIALS AND METHODS

Study area and soil sampling: The study was conducted in a fallow land in Bajgah, 46.7 ha in northeast of Shiraz, Fars province, Iran (Fig. 1). According to the USDA Soil Taxonomy (Soil Survey Staff, 2006), the soil at the study area was classified as Fine, mixed, mesic, Fluventic Calcixerepts. Soil samples were collected (September, 2007) at approximately 60 m^2 at 0-30 cm depth and coordinates of each of the 100 points were recorded using global positioning system (Fig. 1).

Soil characterization: The soil samples were taken to the laboratory and air-dried over night and passed through a 2 mm sieve. Particle size analysis was performed using hydrometer method (Day, 1965); pH was measured in saturated paste; Cation Exchange Capacity (CEC) was determined using extraction with sodium acetate (Page *et al*., 1987); Electrical Conductivity (ECe) was measured with Electroconductimeter, percentage of Calcium Carbonate Equivalent (CCE) was measured by acid neutralization (Salinity Laboratory Staff, 1954); Organic Matter (OM) content was determined using Walkly-Black (1934) plant-available fraction of Zn was determined by means of atomic absorption spectrophotometer (Lindsay and Norvell, 1978). Aqua regia (mixture of HF, $HClO_4$, HNO_3 and H_2SO_4) was used to determine the total contents of Zn (Ma and Uren, 1997).

Fractionation procedure: The procedure of Sposito *et al*. (1982), was used for this study, is designed to separate zinc into five operationally defined fractions: exchangeable (F1), sorbed (F2), organic (F3), carbonate (F4) and residual fractions (F5). A summary of the procedure is as follows:

F1: Two grams of soil were weighed and placed in a 50 mL polycarbonate centrifuge tube, sample extracted with 25 mL of 0.5 M KNO_3 for 16 h.

F2: Residue from exchangeable fraction extracted with 25 mL of deionized water for 2 h (3 times).

F3: Residue from sorbed fraction extracted with 25 mL 0.5 M NaOH for 16 h.

F4: Residue from organic fraction extracted with M 0.05 Na_2EDTA for 2 h.

F5: Residue from carbonate fraction extracted with M_4HNO_3 for 16 h in 80°C.

Descriptive statistics and geostatistical analysis: Statistical analysis were done in 3 stages. First, the frequency distributions were analyzed and normality was tested using the Kolmogoroph-Smironoph test (SAS, 1996). Secondly, the distribution of data was described using conventional statistics such as mean, maximum, minimum, median, Standard Deviation (SD), Coefficient of Variation (CV), skewness and kurtosis. These analyses were conducted using the STATISTICA software package (StatSoft Inc., 2001). Thirdly, geo-statistical analysis was performed using the GS^+ (Gamma Design Software) to determine the spatial dependency of soil properties and zinc chemical forms. Isotropic semi-variograms for the soil parameters were computed to determine any spatially dependant variance within the field. Experimental semi-variograms were fitted to three models (i.e., exponential, spherical and Gaussian) separately and the best model was selected based on the fit. Block kriging procedure in GS^+ was used to obtain the point estimates of the soil properties and zinc chemical

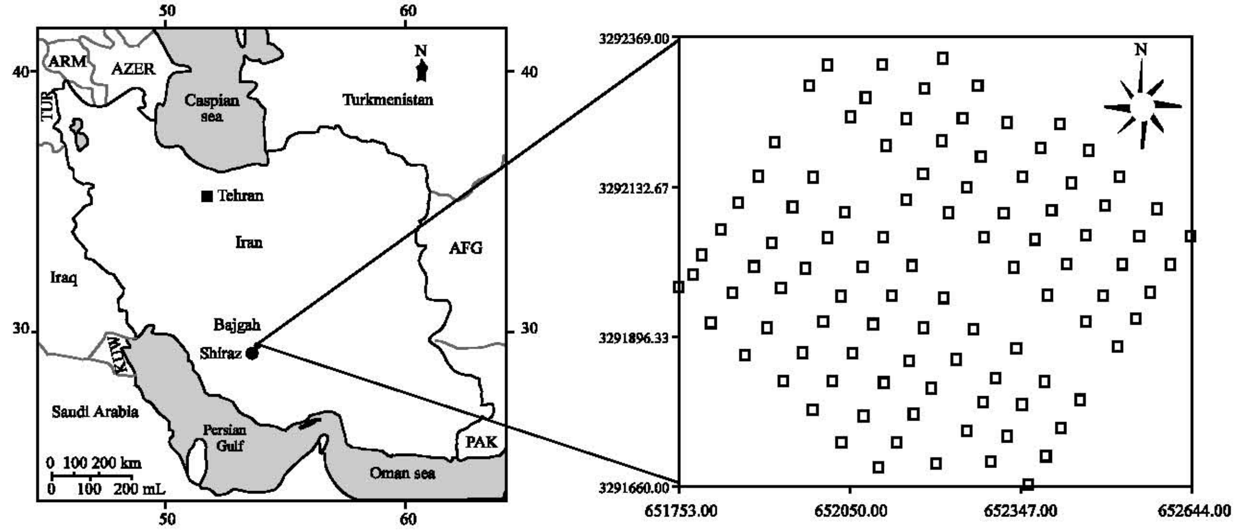

Fig. 1: Location of the study area and sampling pattern

fraction at unsampled locations. Using the model semi-variogram, basic spatial parameters such as nugget variance (C_0), structural variance (C), range (A) and sill ($C + C_0$) was calculated. Nugget variance is the variance at zero distance, sill is the lag distance between measurements at which one value for a variable does not influence neighboring values and range is the distance at which values of one variable become spatially independent of another (Lopez-Granados *et al.*, 2002; Yasrebi *et al.*, 2008).

RESULTS AND DISCUSSION

Soil characteristics: A summery of soil properties is shown in Table 1. The results show that the pH values of the soils ranged from 7.8-8.32, ECe with a range of 0.34-1.20, clay with a rang of 52-59, silt with a rang of 36-43, OM content varied from 0.91-3, CCE and CEC with ranges 47-60 and 15-26, respectively. CV for all of variables was very different; the greatest variation was observed in the OM whereas, the smallest variation was in pH. Silt, pH, clay, CCE and CEC had low variation (CV<15%) whereas, ECe and OM exhibited a medium variation (CV 15-50%) according to the guidelines provided by Warrick (1998) for variability of soil properties.

Table 1: Descriptive statistics for soil properties within the field grid

Variables	Mean	Median	Min	Max	CV	SD	Skewness	Kurtosis
pH (Log (H+))	8.08	8.08	7.80	8.32	1.30	0.11	-0.03	-0.330
EC (dSm^{-1})	0.60	0.59	0.34	1.20	25.91	0.15	1.09	2.330
Silt (%)	40.36	41.00	36.50	43.00	3.90	1.58	-0.50	-0.630
Clay (%)	55.41	54.60	52.30	58.90	3.42	1.90	0.49	-1.230
OM (%)	1.68	1.55	0.91	3.02	26.06	0.44	0.82	0.030
CCE (%)	53.02	53.40	47.19	59.63	6.57	3.49	0.04	-1.140
CEC (Cmol+ kg^{-1})	18.32	17.63	15.43	25.33	9.43	1.73	1.21	2.340
F1 (mg kg^{-1})	0.53	0.54	0.36	0.64	11.32	0.06	-0.46	-0.269
F2 (mg kg^{-1})	0.31	0.30	0.27	0.44	9.35	0.02	1.21	2.390
F3 (mg kg^{-1})	0.07	0.07	0.06	0.10	48.15	0.06	1.21	2.400
F4 (mg kg^{-1})	8.80	8.90	7.02	10.84	12.63	1.11	-0.76	-0.950
F5 (mg kg^{-1})	63.60	63.39	61.15	73.16	1.78	1.13	-0.08	-0.900
Total zinc (mg kg^{-1})	73.33	73.18	72.56	74.67	0.93	0.68	0.83	0.046
Available zinc (mg kg^{-1})	0.85	0.86	0.03	1.05	12.59	0.10	-0.08	-0.910

Fractionation of zinc: The sequential extraction results in Table 1 shows that zinc is strongly associated with the residual, 86% (with a range of 61-63 mg kg^{-1}) and carbonate fraction, 12% (with a range of 7-10.5 mg kg^{-1}), which agrees with the observation of Yasrebi *et al.* (1994), McGrath and Cegarra (1992) and Han and Zhu (1992). Zn in the exchangeable zinc accounts for 0.7% (with a range of 0.3-0.6 mg kg^{-1}) of the total Zn in the soil. The adsorbed fraction is of minor importance accounts for 0.4% (with a range of 0.2-0.4 mg kg^{-1}) of the total Zn in the soil. The percentage of the organic fraction is very low in soil 0.1% (with a range of 0.06-0.1 mg kg^{-1}). This agrees with those reported by Yasrebi *et al.* (1994) and Luoma and Bryan (1981). Overall, the decreasing order of Zinc fractions generally was: Residual >> carbonate fraction > exchangeable > Adsorbed > Organic fraction. In contrast with the residual zinc fraction, other fractions had great variation in field-scale. The mobility of Zn could be assessed by a Mobility Factor (MF) (Salbu *et al.*, 1998), which could be calculated according to the equation as followed:

$$\mathrm{MF} = \frac{\mathrm{F1} + \mathrm{F2} + \mathrm{F4}}{\mathrm{F1} + \mathrm{F2} + \mathrm{F3} + \mathrm{F4} + \mathrm{F5}} \times 100$$

The mobility factor of zinc in the examined soils varied within the limits of 10.5-16% (with average 13.2%).

Zinc fractions in relation to soil properties: Correlation analysis was conducted between soil properties of the studied samples and Zn distribution in these soils. Table 2 shows that exchangeable fraction of Zn gave significant positive correlation with CEC, Zn-DTPA, OM, Clay content, whereas the changes in part showed significant negative correlations with pH and CCE. Also, this fraction and other fractions were nonsignificantly and positively correlated with Silt. The result shown that adsorbed fraction was positively and significantly correlated with CEC and clay content. The highly correlation between adsorbed fraction and clay content is related to the adsorption and ion exchange of zinc into clay particles. The values of coefficients of correlation of organic fraction with pH, clay and silt content of soil were nonsignificant, only OM and CEC were positively and significantly correlated with this fraction. Xiang *et al*. (1995) also, obtained a significant positive correlation between CEC, organic matter and clay content. The high correlation values between the organic content and the heavy metal is related to the considerable chelating power of the organic matter to the heavy metal as well as to thepower of the surface adsorption of the heavy metals onto the surface of the organic material (Gupta *et al*., 1975). However, this part was nonsignificantly and negatively correlated with CCE. These results are in accordance with the findings of Dhane and Shukla (1995). The strongest correlation for carbonate bound were for CCE and Zn-DTPA, whereas were moderately correlation between this part and CEC and OM. There were no significant correlations between the proportions of this fraction with other soil characters. Residual Zn was correlated with clay content and zn-DTPA. Rivero reported that the residual soil Zn significantly correlated with clay and carbonate content in soil, but not with total Zn content.

Forecasting evaluation methods: The summary statistics of soil parameters are shown in Table 1. The descriptive statistics of soil data suggested that they were all normally distributed (according to Kolmogrov Smironov test). In order to identify the possible spatial structure of fractions of zinc and soil properties, semi-variograms were calculated and the best models that describe these spatial structures were identified. The results are given in Table 3 and depicted in Fig. 2. The spatial variation depicted by the semivariograms models are shown on Table 3. Spherical, Gaussian and Exponential models were found to fit well the experimental semi-variograms (Fig. 2). The geostatistical analysis presented different spatial distribution models and spatial dependence levels for the soil properties and fractions of zinc. As shown in Table 3, the ranges of spatial dependences show a large variation (from 32 m for carbonate fraction of zinc up to 368/10 m for total zinc). Knowledge of the range of influence for various soil properties and fractions of zinc allows one to construct independent datasets to perform classical statistical analysis. The range values shown considerable variability among the parameters (Table 3). Figure 3 shows the digital maps obtained by kriging for soil properties. The different ranges of spatial correlation for nutrients may be related to the ions mobility in the soil. In the

Table 2: Correlations between soil properties and Zn fractions

Chemical forms	pH	Silt	Clay	OM	CCE	CEC	Zn-DTPA
F1	-0.71*	0.34ns	0.69*	0.72*	-0.81**	0.76**	0.83**
F2	-0.45ns	0.28ns	0.84**	0.43ns	-0.65*	0.88**	0.44ns
F3	0.49ns	0.38ns	0.47ns	0.87**	-0.64*	0.60*	0.53ns
F4	0.51ns	0.41ns	0.54ns	0.65*	0.86**	0.79*	0.84**
F5	0.38ns	0.18ns	0.68*	-0.34	-0.38ns	0.46ns	0.64*

Table 3: Parameters for variogram models for different soil properties

Variables	Model	Nugget	Sill	Range	Spatial ratio (%)	Spatial class	ME	MSE	R^2
pH	Exponential	0.00097	0.01104	109.50	0.08000	S	-0.0006	0.0100	0.35
EC	Gaussian	0.00001	0.01292	51.70	0.00070	S	0.1678	0.0619	0.45
Silt	Gaussian	0.278	2.55200	57.20	0.09000	S	-0.0330	2.2110	0.37
Clay	Exponential	0.000001	0.00142	148.90	0.00070	S	-0.0059	1.2910	0.80
OM	Gaussian	0.0001	0.05700	64.00	0.00175	S	-1.2055	1.5940	0.52
CCE	Spherical	0.0001	0.06190	181.94	0.00170	S	-0.1620	9.4243	0.51
CEC	Gaussian	0.00001	0.00882	91.00	0.00113	S	-0.0049	1.5620	0.72
F1	Exponential	0.00028	0.00228	70.50	10.81600	S	0.0225	0.0354	0.73
F2	Gaussian	0.00001	0.00862	134.92	0.11600	S	-0.0510	7.8950	0.83
F3	Gaussian	0.00001	0.00912	118.99	0.11000	S	0.0710	2.2170	0.88
F4	Spherical	0.04800	1.20700	32.00	3.82500	S	-0.5350	0.9008	0.74
F5	Exponential	0.27000	1.30400	259.50	17.15400	S	0.0064	1.2370	0.68
Total zinc	Exponential	0.00000	0.00074	368.10	0.13500	S	-0.0640	2.9470	0.83
Available zinc	Exponential	0.00001	0.00374	139.25	0.26700	S	0.0280	0.0610	0.92

Spatial ratio = Nugget semivariance/total semivariance; Total semivariance = Nugget + sill

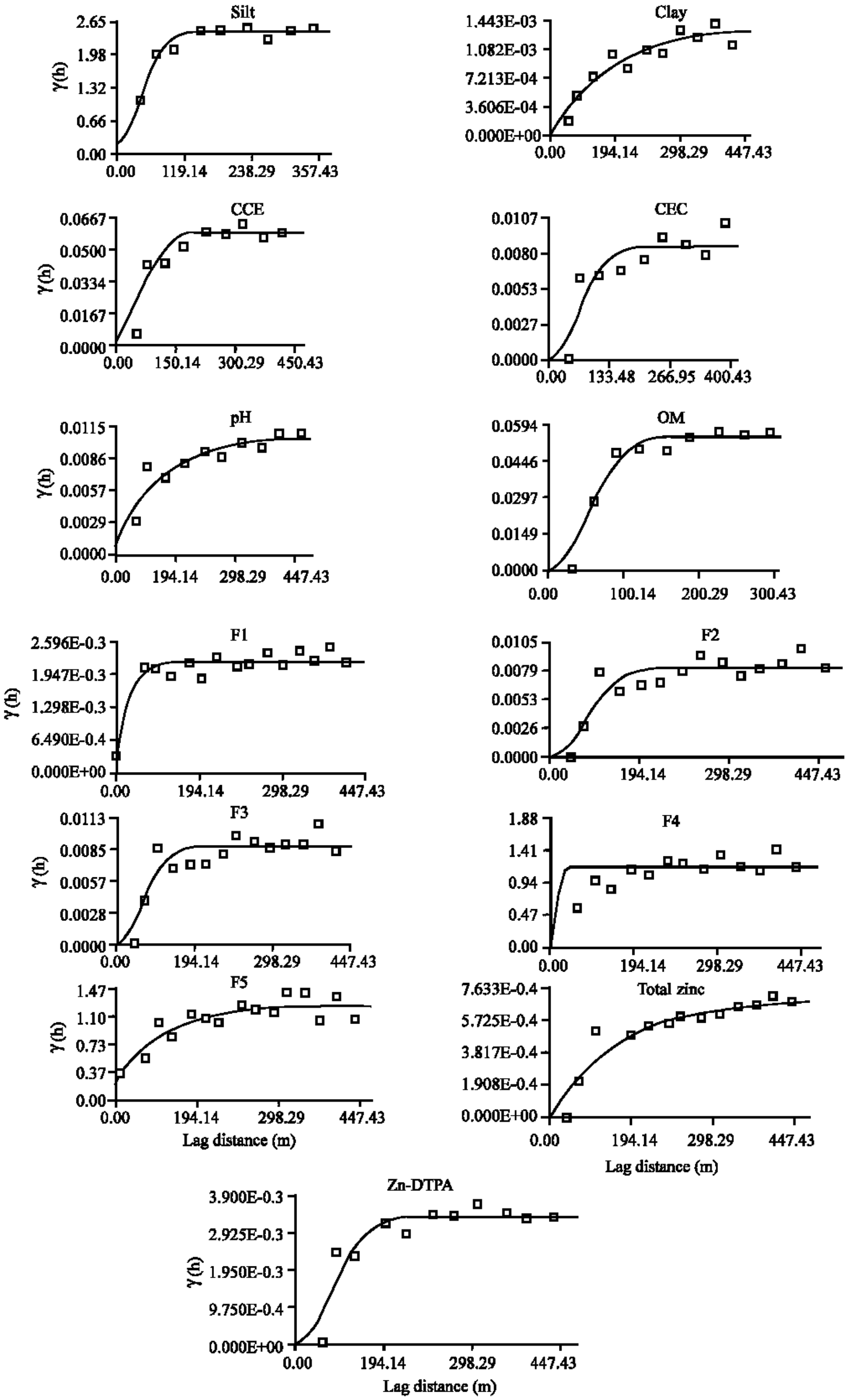

Fig. 2: Omnidirectional semivariogram

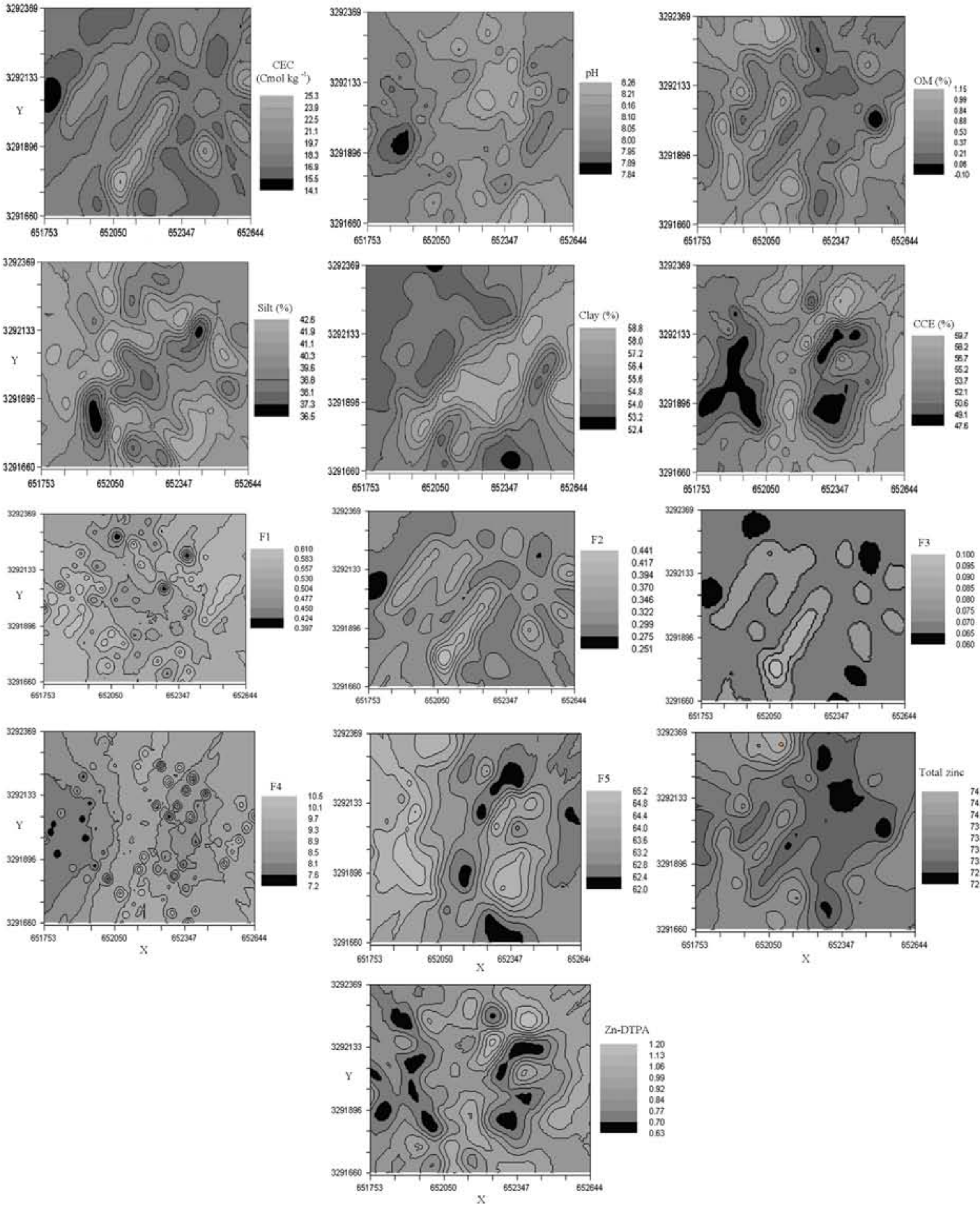

Fig. 3: Digital maps (prepared by ordinary kriging)

present study, spatial distribution of available zinc appeared to be correlated to that of sorbed fraction. The variogram ranges of available zinc and sorbed fraction are the same in studied area (Table 3). A large range indicates that observed values of a soil variable are influenced by other values of this variable over greater distances than soil variables, which have smaller ranges (Lopez-Granados *et al*., 2002).

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