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Determining The Level of Commonly Used Pesticides in Soil at Different Ecosystems in Sudan
Case Study: Kassala- Al-Gadarif State

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ABSTRACT:
The Pesticide residues in the environment pose a risk to public health and environmental components, especially soil and water, particularly in agricultural areas that rely on the intensive use of pesticides. Analytical chemistry has played a significant role in using analytical methods and laboratory equipment to estimate these residues such as terbufos, parathion and profenofos. In This study aims to estimate and evaluate the levels of pesticide residues in some agricultural areas of Kassala state from (Kassala locality, newhalfa and algrba) and Gedaref state from(Gadarif locality, Eastern Galabat, Western Galabat, Alfashaga, Algoraesha, Basonda and Alrahad) in eastern Sudan by collecting (10) soil samples . The study relied on the use of gas chromatography and the presentation of results and statistical analysis using the statistical program (SPSS). As the results of the analyses of soil samples showed that terbufos, profenofos and parathion topped the list with average concentrations of (0.462), (0.333), and (0.289) mg/kg, respectively. Through the results of the (T) test for the samples, the pesticide concentrations showed a significant difference ($P<0.05$) in their mean with the specified value (0.001), where a strong correlation was shown between the use of these pesticides and their concentrations in the samples.

Keywords: Pesticide Residues, Estimation, Soil Contamination, Gas Chromatography, Sudan.

INTRODUCTION

Soil represents the final depot for most of the environmental contaminants including pesticides. This topic was extensively reviewed for POps pesticides [1]. According to his review several studies were conducted to investigate the soil residue levels of organ chlorine pesticides in the Sudan [2]. Analyzed samples from 25 different locations representing three types of cropping systems. In all samples the residues were almost exclusively DDT (Dicholoro-Diphenyl-Trichloroethane) and DDE (Dicholoro-Diphenyl-Ethylene). In none of the samples DDD (Defined-Daily-Dose) was detected. They reported that in spite of the large amounts of DDT used in Gizera scheme since 1949, the residues of DDT(Dicholoro-Diphenyl-Trichloroethane) were 0.26 ppm in cotton soil, o.25 ppm in wheat soil and 0.22 ppm in fallow soil [3] conducted systematic comparative investigation of organ chlorine residues in areas of limited and intensive pesticide use in the Sudan. In their first study they collected fifty soil samples from different locations (the irrigated rive rain region of Northern State, mechanized and traditional rain-fed areas in eastern and western Sudan and the semi-desert area northwest Khartoum) representing areas of limited or no pesticide use in the Sudan[4].

Residues in soil

Soil represents the final depot for most of the environmental contaminants including pesticides [5]. This topic was extensively reviewed for POps pesticides by (Abdelbagi 2005). According to his review several studies were conducted to investigate the soil residue levels of organ chlorine pesticides in the Sudan [6]

MATERIALS AND METHODS

Collection of Soil Samples:

- **AL-Gadarif state:-**

Seven samples of soil were collected from different locations of AL- Gadarif state the samples were collected from agricultural fields grown vegetables, fruits and sorghum. The surface was cleaned from plant debris using soil auger up to a depth of 10 cm. The samples were dried and saved at room temperature until analyzed. Dried samples were crushed into fine powder using pestle and mortar and sieved through 2mm sieve Stored samples were used for experimentation[7].

- **Kassala state:-**

Four samples of soil were collected from different locations of Kassala state the samples were collected from agricultural fields grown vegetables, fruits and sorghum. The surface was cleaned from plant debris .Using soil auger up to a depth of 10 cm. The samples were dried and saved at room temperature until analyzed. Dried samples were crushed into fine powder using pestle and mortar and sieved through 2mm sieve Stored samples were used for experimentation[7]

Extraction and Clean-up:-

- **Extraction of Soil Samples:**

Extracts from each sample were transformed into a 500 ml separator funnel. 400 ml of n-hexane and 100 ml of acetone were added .The jar were tightly closed and placed on an end over –end shaker for two hours .they were left to stand for awhile and then filtered through 240-mm filter paper into around bottom flask containing 100 g anhydrous sodium sulfate to absorb the moisture. Extracts were then Flittered, and solvent was stripped off .The solvent was removed to dryness by rotary evaporator operating under vacuum at a temperature of 40°C. Dried extracts were reconstituted in 10 ml hexane and kept in closed vials at 4°C for clean-up and residues analysis. Two drops of KOH were added to each vial to convert DDT (Dicholoro-Diphenyl-Trichloroethane) to DDE (Dicholoro-Diphenyl-Ethylene) prior to the analysis [8]

- **Clean-up of Soil Samples:**

Clean–up was done according to the method of (Winklemam. and Harmburg., 1980). A chromatographic column (20 x 40mm) was plugged from its lower end with glass wool and then about 4 inches of activated silica gel and layer of anhydrous sodium sulphate were added. The column was first rinsed with few ml of hexane. Extracts from each sample were added as soon as the hexane dried in the top of the silica gel layer and eluted with 200 ml of toluene: acetone (19:1) mixture. The elates were concentrated to dryness by rotary evaporation, reconstituted in 10 ml of acetone, transferred to 10 ml volumetric flask and stored at 4°C for subsequent residues analysis[9].

Gas Liquid Chromatographic (GLC) Analysis

Pesticides in the samples were determined by Hewlett Packard Model 6890 Gas Liquid Chromatography (GLC) equipped with Ni^{63} electron capture detector (ECD) and DB-5 MS capillary column with 30 m length × 0.25 mm internal diameter0.25mm ID. The Stationary phase was composed of an inner coating of 0.25 µm film of five percent pleusl methyl poly siloxane) (8). The column temperature was programmed at an initial oven temperature of 180°C for 2 min., raised at 3°C/min to 220 °C, finally held at 220 °C for 1 min and then raised at 9°C / min to 280 °C where it was held for 7 minutes. Injector and detector temperatures were 280°C and 300°C respectively. Nitrogen was used as carrier gas at a flow rate of 4 ml/min[10].

Data analysis:

The Statistical Package for Social Sciences (SPSS), version 16.0 (SPSS Inc., Chicago, USA) was used for data analyses. Results are presented as frequencies, percentage, Mean ± SD and regression coefficient. The student's t-test was used to compare mean levels between groups. Chi-esquire was used for qualitative data. P-value ≤ 0.05 was considered as the statistical significance [11].

RESULTS

The Results and Discussion of soil samples

The Result of the distribution of pesticide Atrazine concentrations in the (10) soil samples, as shown in Figure (1). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.042** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

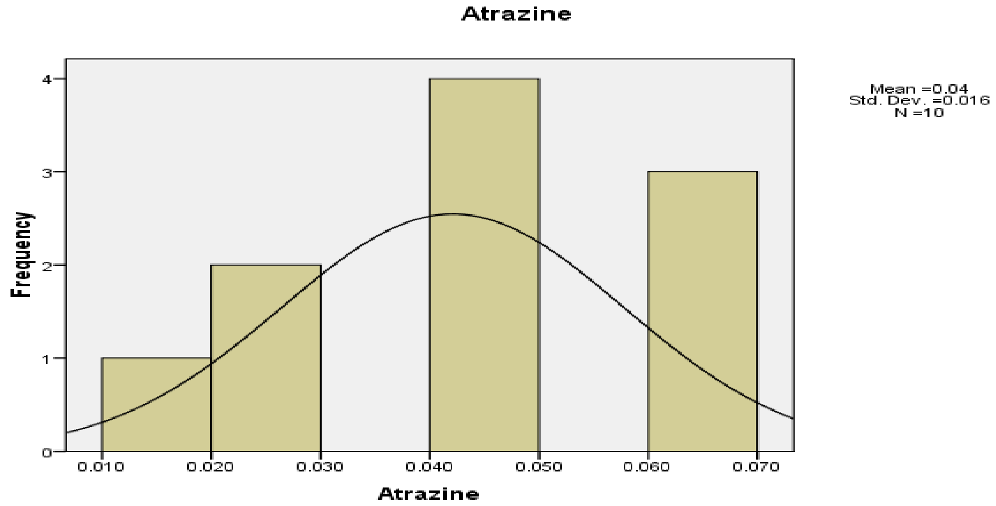

Figure 1 distribution of concentrations (mg/Kg) and percentages of (Atrazine) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Chlorfenvinphos concentrations in the (10) soil samples, as shown in Figure (2). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.166** mg/Kg) suggests that it is the most prevalent in the (10) soil samples

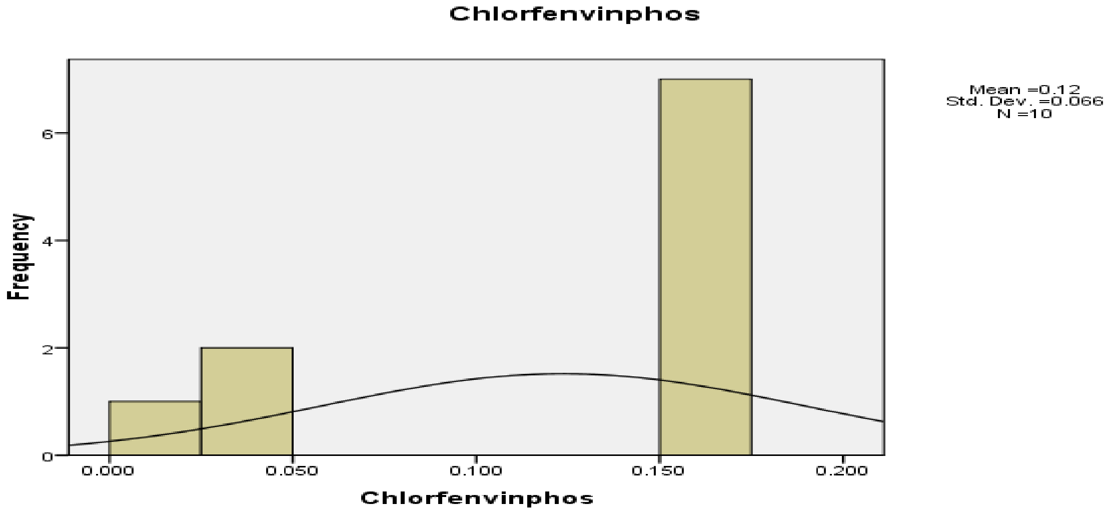

Figure 2 distribution of concentrations (mg/Kg) and percentages of (Chlorfenvinphos) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Chloropyrifos concentrations in the (10) soil samples, as shown in Figure (3).The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.105** mg/Kg) suggests that it is the most prevalent in the (10) soil samples. .

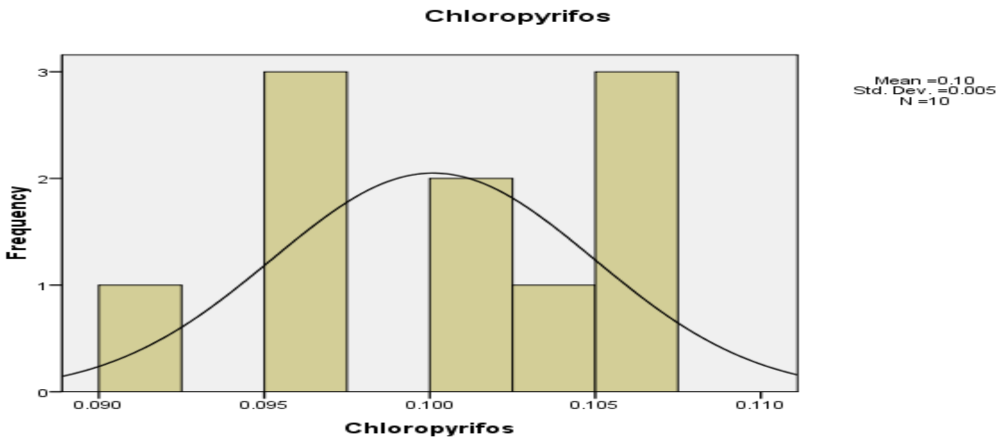

Figure 3 distribution of concentrations (mg/Kg) and percentages of (Chloropyrifos) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Clorfenvinphos concentrations in the (10) soil samples, as shown in Figure (4).The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.042** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

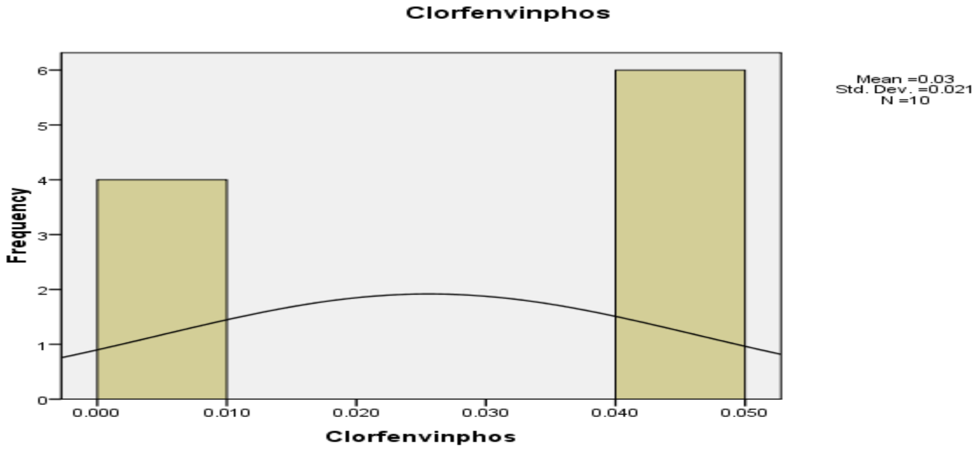

Figure 4 distribution of concentrations (mg/Kg) and percentages of (Clorfenvinphos) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Cyprodinil concentrations in the (10) soil samples, as shown in Figure (5). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.011** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

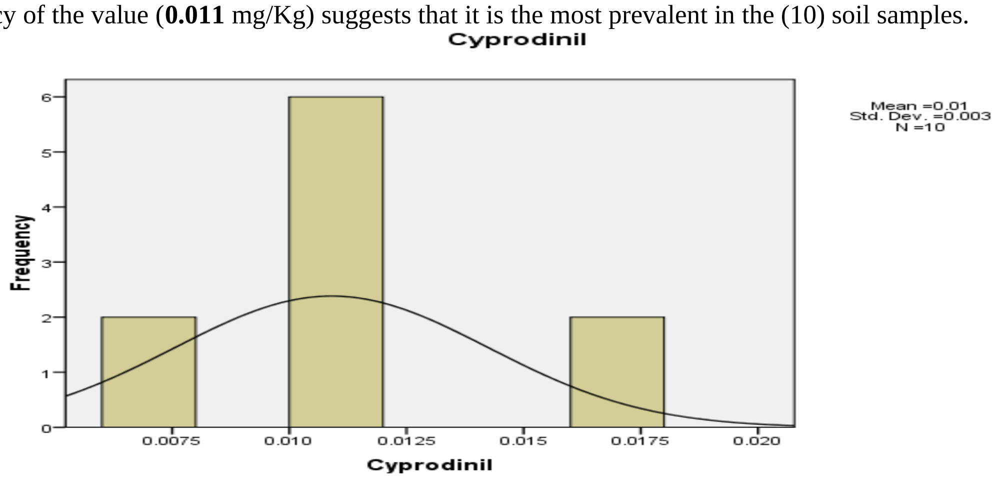

Figure 5 distribution of concentrations (mg/Kg) and percentages of (Cyprodinil) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Diazinon concentrations in the (10) soil samples, as shown in Figure (6). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.014** mg/Kg) and (**0.022** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

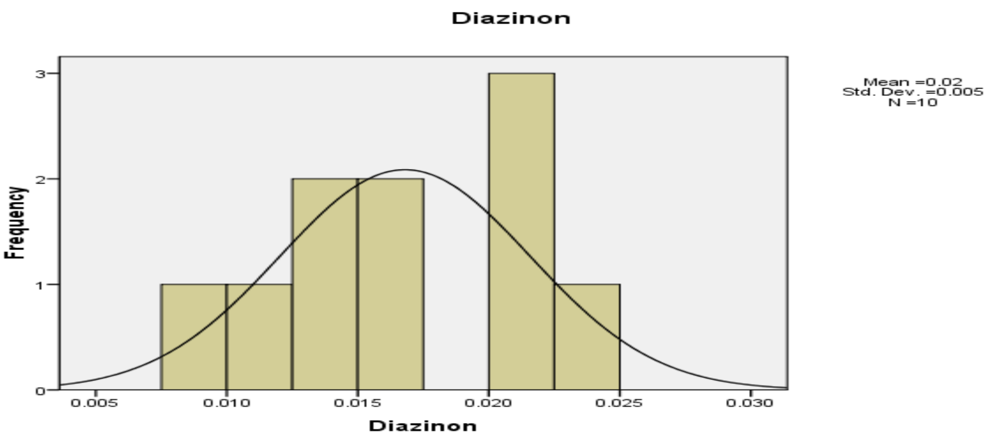

Figure 6 distribution of concentrations (mg/Kg) and percentages of (Diazinon) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Epoxyconazol concentrations in the (10) soil samples, as shown in Figure (7). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.021** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

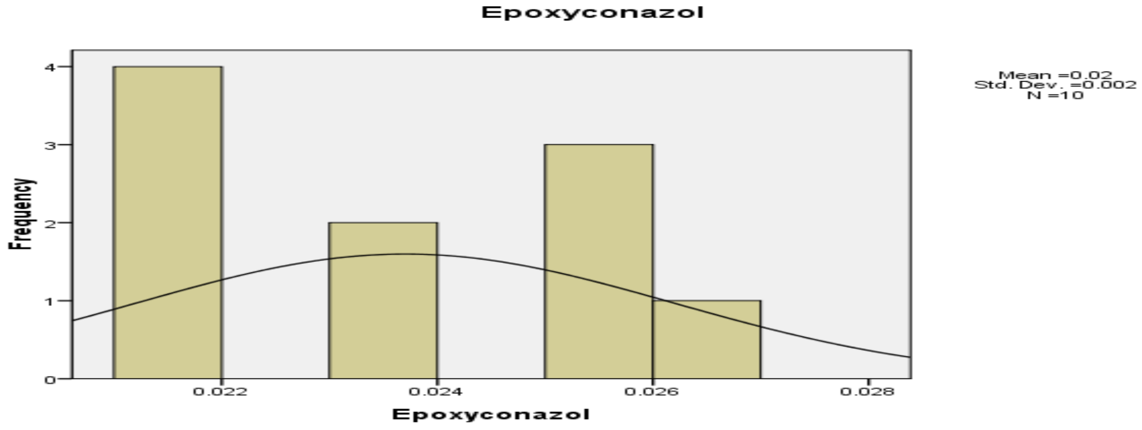

Figure 7 distribution of concentrations (mg/Kg) and percentages of (Epoxyconazol) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Ethoprophos concentrations in the (10) soil samples, as shown in Figure (8). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.062** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

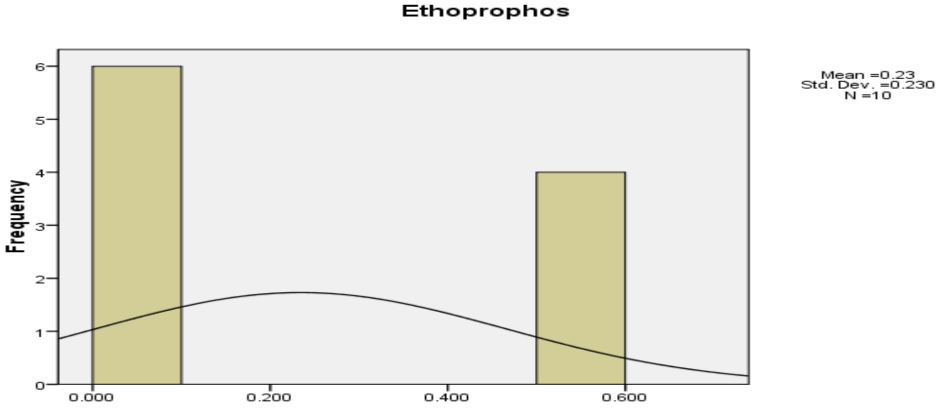

Figure 8 distribution of concentrations (mg/Kg) and percentages of (Ethoprophos) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Malathion concentrations in the (10) soil samples, as shown in Figure (9). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.073** mg/Kg) and (**0.076** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

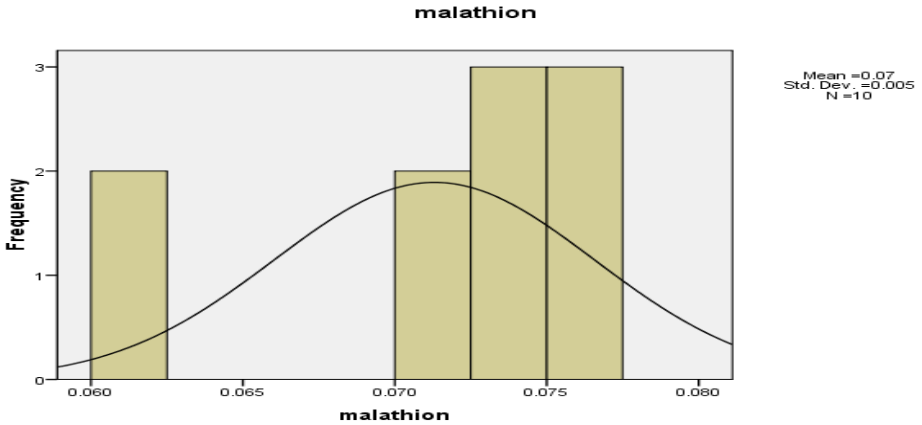

Figure 9 distribution of concentrations (mg/Kg) and percentages of (Malathion) in the soil samples, prepared by the researcher**.**

The Result of the distribution of pesticide Parathionmethyl concentrations in the (10) soil samples, as shown in Figure (10). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.32** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

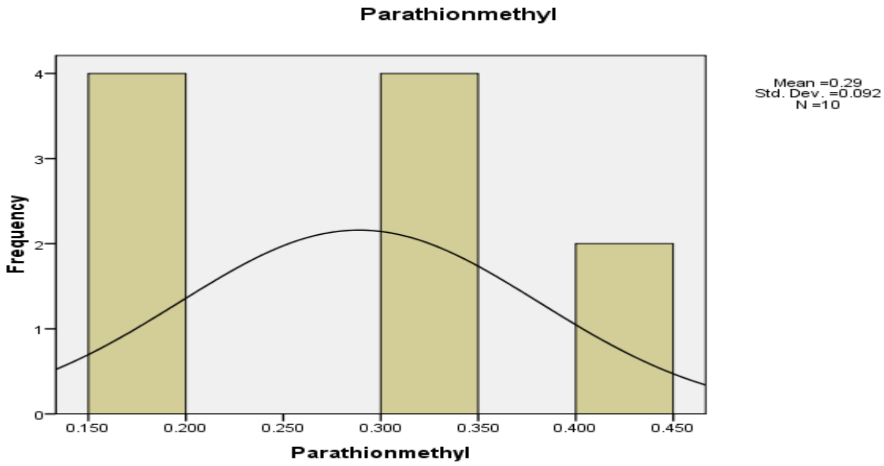

Figure 10 distribution of concentrations (mg/Kg) and percentages of (Parathionmethyl) in the soil samples, prepared by the researcher**.**

The Result of the distribution of pesticide Pirimiphosmethyl concentrations in the (10) soil samples, as shown in Figure (11). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.009** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

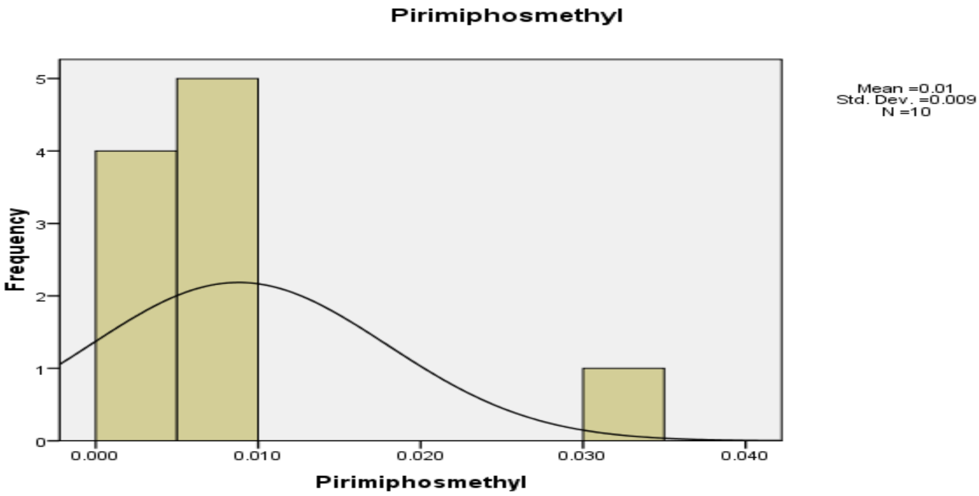

Figure 11 distribution of concentrations (mg/Kg) and percentages of (Pirimiphosmethyl) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Pirimicarb concentrations in the (10) soil samples, as shown in Figure (12). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.038** mg/Kg), (**0.04** mg/Kg) and (**0.424** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

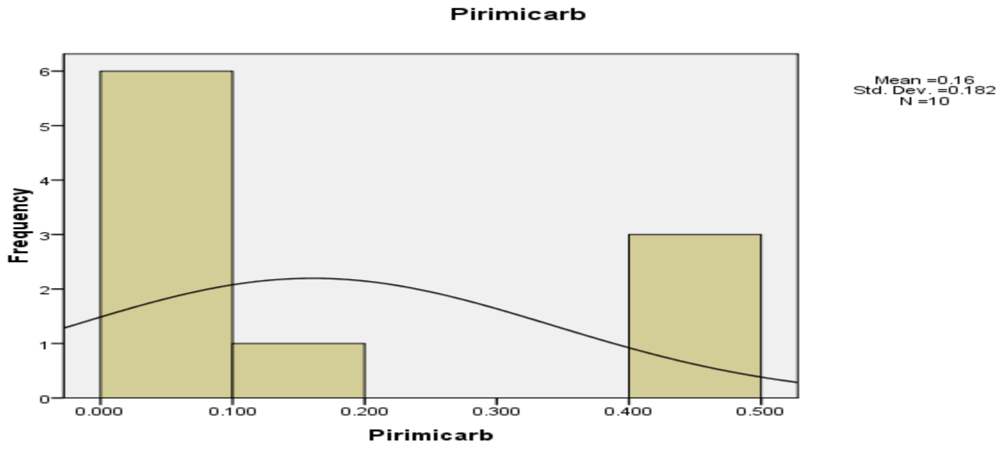

Figure 12 distribution of concentrations (mg/Kg) and percentages of (Pirimicarb) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Profenfos concentrations in the (10) soil samples, as shown in Figure (13). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. It was found that there is equality in the frequencies suggests that it is the most prevalent in the (10) soil samples.

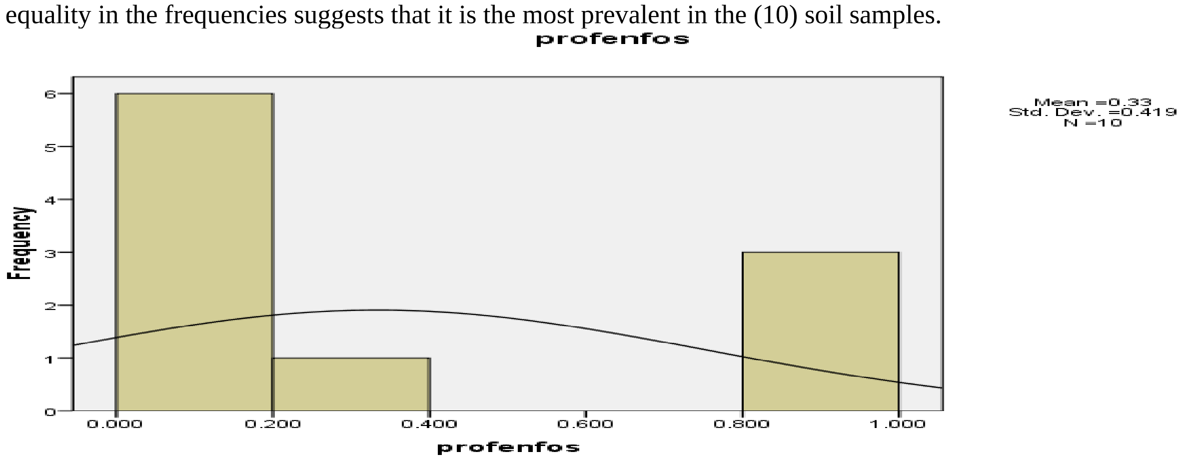

Figure 13 distribution of concentrations (mg/Kg) and percentages of (Profenfos) in the soil samples, prepared by the researcher.

The Result of the distribution of pesticide Terbufos concentrations in the (10) soil samples, as shown in Figure (14). The consistency between the percentage and corrected percentage indicates the absence of missing concentrations. The high frequency of the value (**0.463** mg/Kg) suggests that it is the most prevalent in the (10) soil samples.

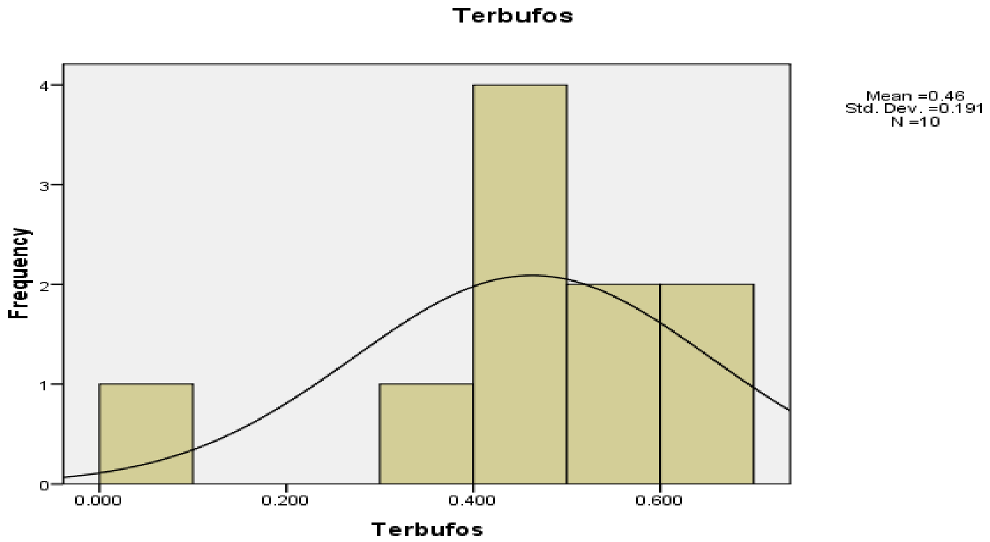

Figure 14 distribution of concentrations (mg/Kg) and percentages of (Terbufos) in the soil samples, prepared by the researcher.

T-Test to soil samples:

Table 1 results of the (T-Test) test for pesticide concentrations in soil samples, prepared by the researcher.

	Test Value = 0.001	
	Sig. (2-tailed)	**Mean Difference**
Atrazine	.000	.041100
Chlorfenvinphos	.000	.122630
Chloropyrifos	.000	.099100
Clorfenvinphos	.005	.024600
Cyprodinil	.000	.009900
Diazinon	.000	.015800
Epoxyconazol	.000	.022700
Ethoprophos	.011	.233400
malathion	.000	.070300
Parathionmethyl	.000	.287900
Pirimiphosmethyl	.024	.007800
Pirimicarb	.021	.159600
profenfos	.034	.332300
Terbufos	.000	.461800

The results showed that all pesticides, except for **Cyprodinil** and **Pirimiphosmethyl**, had a **Sig. (2-tailed)** value less than 0.05, indicating a difference between the sample mean and the assumed mean (0.001 mg/Kg).

4.3 Discussion of results based on pesticides classification by use: The results will be discussed based on the type of pesticides detected in the soil and water samples, these will be classified into insecticides, herbicides and fungicides. The average total of each pesticide within each group will be compared and a comparative analysis will be conducted to illustrate the most commonly used types of these pesticides in the soil samples under study.

- **Insecticides:**

The study found that the number of insecticides in the soil samples under study is (12) pesticides according to the table below.

Table 2 total average concentrations of pesticides for soil samples.

No	pesticide	total average concentration (mg/Kg)
1	Chlorfenvinphos	0.124
2	Chlorpyrifos	0.100
3	Clorfenvinphos	0.026
4	Diazinon	0.017
5	Epoxyconazol	0.024
6	Ethoprophos	0.234
7	malathion	0.071
8	Parathionmethyl	0.289
9	Pirimiphosmethyl	0.009
10	Pirimicarb	0.161
11	profenfos	0.333
12	Terbufos	0.446

From table No (2), we find that pesticide (**Terbufos**) has the highest concentration with a total average concentration value of (**0.446** mg/Kg), and pesticide (**Pirimiphosmethyl**) has a concentration with a total average concentration value of **(0.009** mg/Kg).

- **Herbicides:**

The study found that the number of herbicides in the soil samples under study is (1) herbicides.

Table 3 total average concentrations of herbicides for soil samples.

No	pesticide	total average concentration (mg/Kg)
1	Atrazine	0.042

From Table No (3), we find that pesticide (**Atrazine**) has the highest concentration with a total average concentration value of (**0.042** mg/Kg).

❖ **Fungicides:**

The study found that the number of fungicides in the soil samples under study is (1) fungicides.

Table 4 total average concentrations of fungicides for soil samples.

No	pesticide	total average concentration (mg/Kg)
1	Cyprodinil	0.011

From Table No (4), we find that pesticide (**Cyprodinil**) has the highest concentration with a total average concentration value of (**0.011** mg/Kg).

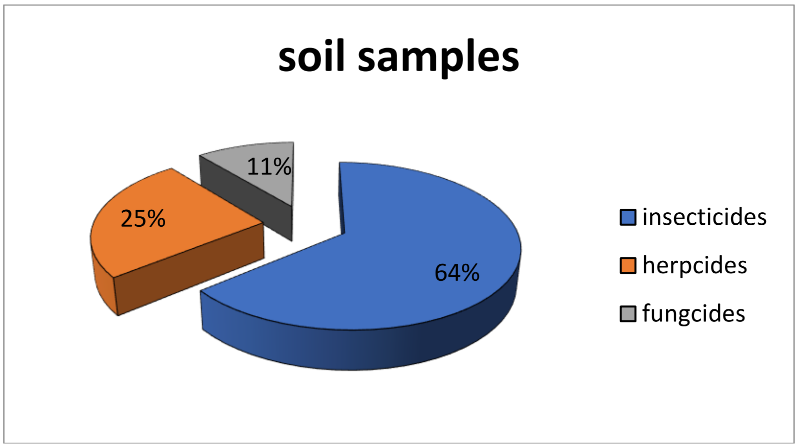

Figure 15 Illustrates the percentage distributions of pesticide types by use in soil samples.

DISCUSSION

-Results of the previous statistical data for soil samples showed the presence of a variety of pesticides depending on the concentrations and the type of pesticide used, which indicates the widespread use of these pesticides in the sample collection areas. The study showed the presence of high concentrations of Terbufos, profenfos, and parathion pesticides **(0.462, 0.333, 0.289 mg/kg) respectively** due to the intensive use of these pesticides in these areas.

- The study found that the pesticides **Ethoprophos**, **Parathion**, and **Terbufos** had a large mean difference, indicating their presence in the samples at concentrations much higher than the assumed value (0.001 mg/Kg).

- Through the classification of pesticides detected in (10) soil samples, the study showed that insecticides are the most dominant, outnumbering herbicides and fungicides. This indicates the overuse of these types of pesticides, due to the nature of the region and the diversity of pests, which led to an increase in the concentrations of these pesticides.

- From the circular diagram showing the distribution of insecticide, herbicide, and fungicide percentages in the soil samples, according to the analysis of the soil samples, the figure shows that the highest percentage is for insecticides, followed by herbicides, and fungicides appear in lower percentages.

- The result of the soil samples analyses in the specified study areas are described as soil contaminated with pesticides residues according to international standards, noting that the pesticides decompose within the safe period defined for each pesticide

CONCLUSION

- The study concluded that there are residual concentration of pesticides used in agricultural area in the states of Kassala and Gedaref
- Insecticides are more prevalent as residual concentrations compared to herbicides and fungicides.
- The study found that Gedarif state has a high intensity of pesticides use compared to Kassala state

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CONFLICT OF INTERESTS

No conflict of interest.

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