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# Studies on the Conformity of Three-Nutrient and Micronutrient Fertilisers in Drip Irrigation System

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**Abstract**

As a result of the changes in climatic conditions, especially in precipitation, the water usage methods of the producers have changed in parallel and drip irrigation systems started to be used widely. A very small amount of drip irrigation fertilizers is produced by the Turkish fertilizer industry, and therefore they are mostly imported from abroad. In this study, fertilizers produced by large fertilizer companies and imported by these companies are examined. The main purpose of this study is to determine the conformity of the compound three-nutrients and micronutrient fertilizers used in drip irrigation system, in accordance to the "Regulation on the Surveillance and Inspection of Fertilizers on the Market" (EC fertilizers) of the Ministry of Agriculture and Forestry dated 29.03.2014, number 28956. Some physical and chemical properties as well as heavy metal contents of the fertilizers supplied from the market and fertilizer factories in their original packaging were investigated. As a result of the examination of 56 three-nutrient and 13 micro-nutrient-containing fertilizers according to the relevant regulation, it was seen that 35 of the three-nutrient fertilizers and 5 of the fertilizers containing micro-nutrients were determined to be out of the tolerance values of the regulation, so they were not suitable for the fertigation system. Therefore, it was determined that 40 out of 69 fertilizers were not in compliance with the regulation. If the heavy metal contents are examined on the basis of the tolerance values applied by China and Japan, it can be said that 10 (5xHg, 4xCd and 1xHg) fertilizers do not conform to drip irrigation system. Fertilizers were examined in terms of radioactivity and $^{238}U$ and $^{232}Th$ were not found to be threatening for human health. It is believed that the samples do not contain $^{40}K$ and the defined values are natural. The results of this current study will be useful in making fertigation and fertilization programs to be prepared based on soil and water analyses or whether to use those fertilizers.

**Keywords:** Fertilizer, physical property, chemical property, drip irrigation.

# Üç Besinli ve Mikrobesinli Gübrelerin Damla Sulama Sistemine Uygunluğu Üzerinde Çalışmalar

**Öz**

İklim şartlarında ve özellikle yağış durumlarında meydana gelen değişiklikler nedeni ile üreticilerin su kullanım yöntemlerindeki değişikliklere paralel olarak, damla sulama sistemleri yaygın kullanılmaya başlanmıştır. Damla sulama gübrelerinin çok az bir miktarı ülkemiz gübre sektörü tarafından üretilmekte, genelde ülkemiz dışından ithal edilmektedir. Bu çalışmada, büyük gübre firmaları tarafından üretilen gübreler ile bu firmalar tarafından ithal edilen gübreler incelenmiştir. Çalışmanın ana amacı, damla

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sulama sisteminde kullanılan, üç besinli ve mikro besin elementi içeren gübrelerin Tarım ve Orman Bakanlığının 29.03.2014 tarih ve 28956 sayılı "Gübrelerin Piyasaya Gözetimi ve Denetimi Yönetmeliği" göre (EC fertilizers) değerlendirilerek uygunluk durumlarının belirlenmesidir. Piyasadan ve gübre fabrikalarından orijinal ambalajda sağlanan gübre örneklerinin bazı fiziksel, kimyasal özellikleri yanında ağır metal içerikleri de incelenmiştir. Bulgularımız, analizi yapılan 56 adet üç besinli ve 13 adet mikro besin elementi içeren gübrenin ilgili yönetmeliğe göre incelenmesi sonucunda, üç besinli gübrelerin 35 adedinin ve mikro besin elementi içeren gübrelerin 5 adedinin yönetmeliğin tolerans değerleri dışında saptanması nedeni ile fertigasyon sistemine uygun olmadığını göstermiştir. Dolayısıyla, toplam 69 adet gübreden 40 adedinin yönetmeliğe uygun olmadığı belirlenmiştir. Ağır metal içerikleri Çin ve Japonya'nın uyguladığı tolerans değerlerine göre incelendiğinde, 10 adet (5xHg, 4xCd ve 1xHg) gübrenin de damla sulama sistemine uygun olmadığı ifade edilebilir. Gübreler, radyoaktivite açısından irdelendiğinde, $^{238}U$ ve $^{232}Th$'un insan sağlığı bakımından tehlike oluşturacak seviyede bulunmadığı, örneklerin $^{40}K$ içermediği ve belirlenen bu değerlerin de doğal olduğu düşünülmektedir. Çalışmanın sonuçları, toprak ve su analizlerine bağlı olarak hazırlanacak fertigasyon ile gübreleme programlarının yapılmasında veya o gübrelerin kullanılıp kullanılmaması konusunda yarar sağlayacaktır.

**Anahtar Kelimeler:** Gübre, fiziksel özellik, kimyasal özellik, damla sulama

## INTRODUCTION

Global warming in the recent years significantly changed the precipitation regime of countries. In this regard, the water consumed in cities, industry and agriculture must be used efficiently and in the most economical way. In agricultural production, water harvesting is important, this water is used for irrigation. Studies in this context have shown that drip irrigation is the best method.

Many researchers pointed out that drip irrigation is a very efficient way of irrigation and the fertilizers delivered via this system (fertigation) results in higher quality and high yield, (Bar-Yosef, 1977; Levis et al., 1979).

Fertilization by drip irrigation provides the uptake of nutrients via the roots efficiently (Çolakoğlu, 1990). When chemical fertilizers dissolve in water, they gain an electrical charge and may act as a chemical salt. Producers can check whether they are fertilizing correctly by measuring the EC value of the fertigation system. Therefore, $Na^+$ and $Cl^-$ are not preferred to be included in fertigation fertilizers since they increase salinity. It is desirable that the fertilizers used with the drip irrigation system do not contain additives, have a high solubility in water and do not cause clogging pipes.

The objective of this study was to determine the compliance of 56 macro-nutrient (N, P, K compound fertilizers) and 13 micro-nutrient (B, Co, Cu, Fe, Mn, Mo, Zn,) drip irrigation fertilizers with the Regulations. The other objective was to find some of their heavy metals (As, Cd, Cr, Hg, Ni, Pb) and natural radioactivity ($^{40}K$, $^{238}U$ and $^{232}Th$) contents.

## MATERIAL AND METHODS

In this study, 56 NPK compound fertilizers (Table 1) and 13 single/multi micronutrient fertilizers (Table 2), in total 69 drip fertilizers which are either produced in our country or imported from abroad were the materials that were examined.

**Table 1.** Formulation and number of three nutrient fertilizers
***Çizelge 1.*** *Üç besinli gübrelerin formulasyon ve adedi*

| Fertilizer Formulation | Number | Fertilizer Formulation | Number |
|---|---|---|---|
| 10.5.40 | 1 | 15.30.15 | 6 |
| 28.14.25 | 1 | 18.18.18 | 10 |
| 20.8.20 | 1 | 20.20.20 | 8 |
| 16.8.24 | 2 | 15.5.30 | 5 |
| 3.5.40 | 1 | 18.6.24 | 1 |
| 20.10.20 | 1 | 12.5.40 | 1 |
| 12.5.41 | 2 | 12.40.10 | 1 |
| 14.8.30 | 1 | 14.7.17 | 2 |
| 16.10.17 | 1 | 15.15.15 | 1 |
| 13.40.13 | 1 | 14.6.26 | 1 |
| 5.5.40 | 1 | 12.7.40 | 1 |
| 14.40.5 | 1 | 16.6.31 | 1 |
| 23.5.5 | 1 | 10.30.10 | 1 |
| 15.5.35 | 1 | 16.16.16 | 1 |

**Table 2.** Formulation and number of microelement fertilizers
***Çizelge 2.*** *Mikroelementli gübrelerin formülasyon ve adedi*

| Fertilizer Formulation | Number |
|---|---|
| 13.2% Fe EDTA | 1 |
| 6% Fe EDDHA | 1 |
| 14% Zn EDTA | 1 |
| 3% B 0.4% Cu EDTA 6.5% Fe EDTA 5% Mn EDTA 0.2% Mo 5% Zn EDTA | 1 |
| 24% $MnSO_4 4H_2O$ Mn | 1 |
| 22% $ZnSO_4 7H_2O$ Zn | 3 |
| 0.2% B 0.5% Cu 6% Fe 4% Mn 5% Zn | 1 |
| 0.6% B 1% Cu 5% Fe 3.5% Mn 0.3% Mo 2.4% Zn | 1 |
| 1.5% B 0.6% Cu 4% Fe 3% Mn 0.05% Mo 4% Zn | 1 |
| 0.5% B 1% Cu 6% Fe 4% Mn 8% Zn | 1 |
| 1% Cu 4% Fe 2% Mn 3% Zn | 1 |

After the preliminary preparations were completed in the examination of the fertilizer samples taken from the market and fertilizer factories, for the evaluations the regulation (EC fertilizers 29.03.2014/28956) and AOAC analysis methods were used.

Radionuclide contents of the fertiliser samples were measured by Gamma spectroscopy method (Yaprak,1995).

## RESULTS AND DISCUSSION

### 1. pH, EC and insoluble matter

The pH values of the N, P, K compound fertilizers examined in the study were found between 3.22 and 6.69; EC between 0.81 and 1.78 mS $cm^{-1}$, humidity between 0.07 and 6.13% and the insoluble matter percentage between 0.01 to 5.62. The pH of the 13 micronutrient fertilizers changed from 3.32 to 8.64; EC values from 0.21 to 1.20 mS $cm^{-1}$; humidity from 0.13 to 2.46% and the insolubility from 0.10 to 2.24%.

The EC values of the 20.20.20 and 15.5.30 grade fertilizers were found to be low. Likewise, the EC values of the chelated fertilizers containing micronutrients were also found to be low. It is considered normal to have lower EC values than fertilizers in the form of mineral salts, since micronutrients bind to chelates such as EDTA or EDDHA and do not generate electrical charge when dissolved in water. The EC values of micro element fertilizers in the form of mineral salts are higher than those in the form of chelate (0.47-1.19 mS $cm^{-1}$). Fertilizers produced with mineral salts sometimes show low levels of EC as those in the form of chelate most probably due to lower nutrient elements than stated on the label. In chelated microelement fertilizers, theoretically, the EC value may be very low or not at all, as in urea fertilizer.

However, when EDTA group is used as the chelating material, EC value can be measured even less

The high moisture content of the three-nutrient compound fertilizer (6.13%) may be due to incorrect packaging or unsuitable storage conditions, as well as the use of chemicals containing crystal water (such as $MgSO_4$) in the production of the fertilizer.

The moisture contents of 8 of the microelements containing fertilizers were determined above the level specified in the relevant regulation (1.50%). Two of these 8 fertilizers are in chelate form. Their moisture contents were found 1.64% and 2.46%, in other words, above the limits, might be caused by inappropriate storage conditions or packaging or low (10%) chelate content. In 6 of the others, the moisture content was between 1.58-1.89% i.e above 1.50%. High humidity in these samples may be due to unsuitable storage conditions. Of the other 2, the moisture content varied between 2.15-9.01%. In the production of the compounds, most probably crystalline water ($CuSO_4$, $FeSO_4$, $ZnSO_4$ and $MnSO_4$) is used.

In 12 of the three-nutrient fertilizers, the rate of insoluble matter in water was more than 1%. The insoluble matter in 8 of these 12 fertilizers was below 2% and at higher levels in the other

4 fertilizers. It is not appropriate to use fertilizers with a high number of water-insoluble matter in fertigation, as this will cause clogging of drip irrigation pipes.

Four of the micronutrient fertilizers were in the form of chelate and their water-insoluble matter was found more than 1%. Of the fertilizers containing micronutrients in mineral form, 4 of them are mono-nutrient and 3 had water-insoluble matter above 1%. Other 5 multi-micronutrient fertilizers can be used in drip irrigation since their water-insoluble matter was found less than 1.1%. Findings of this study indicated that the amount of water-insoluble matter is almost non-existent most of the time.

## 2. N, $P_2O_5$ $K_2O$, (%) Contents of Three-Nutrient Drip Fertilizers

It has been observed that 19 of the 56 drip irrigation fertiliser samples were outside the nitrogen (N) fertilizer tolerance threshold set forth in the "Regulation on Chemical Fertilisers Used in Agriculture" (Figure 1).

Fifty-six fertiliser samples were analysed in terms of their N contents (%). Ten of them had N more than the data on their labels while 27 of them were within the tolerance limits (Figure 1).

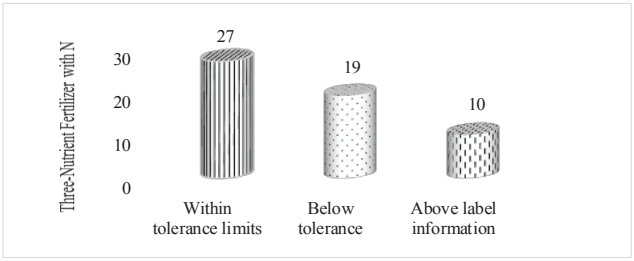


**Figure 1.** Tolerance of three nutrient fertilizers with respect to their N % content
***Şekil 1.*** *Üç besinli gübrelerin % N miktarlarının toleransa uygunluğu*

Results were examined according to their nitrogen forms and found that $NH_2$-N ratio of the total N in 4 fertilizer samples was higher than other N fertilizer types. Such three-nutrient fertilizers are also used in soilless culture technique and in this regard may create a problem in terms of N uptake. Urea-N does not turn into $NH_4$ and $NO_3$-N in soilless environment and since the plant cannot take N in the form of $NH_2$-N, it is recommended that special care should be taken in the use of such fertilizers.

When the $P_2O_5$ contents of 56 drip fertilizers were examined, 5 of the fertilizers were below the tolerance limits of 1.1(%) specified in the relevant regulation. It was determined that 24 fertilizers were within the tolerance limits, and 27 contained more $P_2O_5$ (%) than the label data (Figure 2).

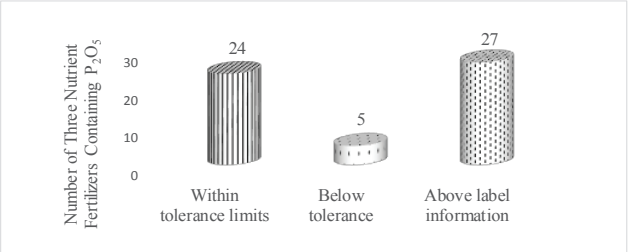


**Figure 2.** Tolerance of three nutrient fertilizers with respect to their $P_2O_5$% contents
***Şekil 1.*** *Üç besinli gübrelerin % $P_2O_5$ miktarlarının toleransa uygunluğu*

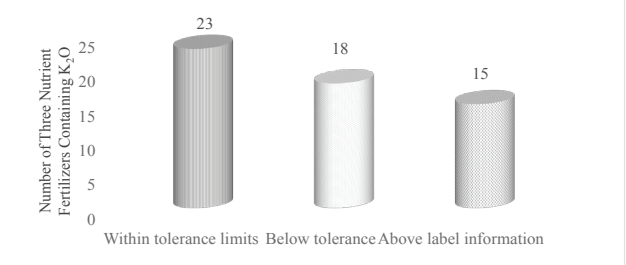


**Figure 3.** . Tolerance of three nutrient fertilizers with respect to their $K_2O$ % contents
***Şekil 3.*** *Üç besinli gübrelerin % $K_2O$ miktarlarının toleransa uygunluğu*

Regulation-compliance status and $K_2O$ (%) ranges of the fertilizers are given in Fig.3. According to the tolerance value (1.1%) for $K_2O$, 23 of the fertilizers were determined to be suitable, 18 of them were out of the tolerance and 15 contained more $K_2O$ than the amount specified on the label.

The results of the compliance status of 56 fertilizers with three nutrients are given in Table 1 shows that 35 fertilizer samples do not comply with the tolerance values of the regulation. Among the fertilizer samples that are not within the tolerances of the relevant regulation, there is only 1 fertilizer that is not suitable for all three plant nutrients. Five of the two nutrient compound fertilizers (NP, NK and PK) are not within the appropriate tolerance values. The number of

fertilizers with inappropriate N (%) content was 14, the number of $P_2O_5$ (%) unsuitable fertilizer was 2, and the $K_2O$ (%) unsuitable fertilizer was 12. It was determined that only 22 fertilizer samples were in compliance with the tolerance limits (1.9).

Therefore, it can be said that the non-compliance with the tolerance values in the chemical fertilizer inspection regulation in NPK fertilizers is mostly caused by N and $K_2O$ as plant nutrients.

Table 3 shows that 14 of the three-nutrient ($N+P_2O_5+K_2O$) fertilizers that do not comply with the tolerance limits are not suitable in terms of N and 13 of them in terms of $K_2O$. One reason for this inconvenience might be the N in the three nutrient compound fertilizers. In these fertilizers, a large part of N (13 of them) are in the form of $NH_2$-N (3.0-11.5% of total N). In terms of K, might be related to the K source. If the Na, Ca and Mg results of three-nutrient fertilizers are examined, it was determined that the sum of these three elements was between 0.27-0.68%.

**Table 3.** Number of fertilizers that exceed the tolerance limits
***Çizelge 3.*** *Damla sulama sistemi gübrelerinin tolerans sınırları dışındaki örnek sayıları*

| Plant nutrients | Number of fertilizers out of limits |
|---|---|
| N, % | 14 |
| $P_2O_5$, % | 2 |
| $K_2O$, % | 13 |
| $N+P_2O_5$, % | 1 |
| $N+K_2O$, % | 3 |
| $P_2O_5+K_2O$, % | 1 |
| $N+P_2O_5+K_2O$, % | 1 |

Most probably Na, Ca and Mg presence, in addition to the N, $P_2O_5$, $K_2O$, in the three- nutrient fertiliser could have led these fertilizers to go beyond the tolerance limits.

### 3. Micronutrient containing drip fertilizers

Thirteen of the fertilizers (B, Cu, Co, Fe, Mn, Mo, Se and Zn) containing micronutrients and used in the drip irrigation system were examined and analysed. Four of these fertilizers are in the form of chelates (EDTA and EDDHA) and the other 9 are mineral salts.

Chelates were determined in 4 micronutrient fertilizers, which are reported as "chelated" on the label. It is found that these fertilizers contained $SO_4$ which shows that they are produced from mineral salts, such as $FeSO_4$, $MnSO_4$, $ZnSO_4$ (Table 4). So, 2 of the 4 fertilizers stated to be chelated are not in compliance with the relevant regulation.

**Table 4.** Chelate and micro elements in the fertilizers (%)
***Çizelge 4.*** *Şelatlı mikro element gübrelerinin içerikleri (%)*

| Elements | 13.2% Fe EDTA | 6% Fe EDDHA | 14% Zn EDTA | 3% B, 0.4% Cu EDTA, 6.5% Fe EDTA, 5% Mn EDTA, 0.2% Mo, 5% Zn EDTA |
|---|---|---|---|---|
| Ca % | - | - | 0.74 | 0.22 |
| Mg % | - | - | - | - |
| S % | 0.56 | - | - | 16.90 |
| Na % | 4.41 | 9.42 | 8.04 | 0.33 |
| B % | - | 0.02 | - | 0.05 |
| Cu % | 0.01 | 0.14 | 0.02 | 0.57 |
| Fe % | 13.18 | 6.04 | - | 4.84 |
| Mn % | - | 0.01 | - | 8.01 |
| Zn % | - | - | 15.59 | 5.59 |
| Se % | - | 0.04 | - | 0.03 |
| Co % | - | - | - | - |
| Mo % | - | - | - | 0.12 |
| Şelat % | 95.42 | 100.00 | 60.90 | 10.00 |

(-): Unidentified

Table 5 shows the results of 9 micronutrient fertilizers which are in the form of mineral salts according to their label. Results indicated that 3 of the 4 fertilizers containing single microelement comply with the relevant regulation, and 3 of the 5 fertilizers containing multiple micronutrients do not comply with the tolerance values specified in the relevant regulation in terms of total micronutrient elements.

Findings in relation to 56 fertilizers containing three nutrients and 13 micronutrients showed that 35 of the three-nutrient fertilizers and 5 of the fertilizers containing micronutrients were out of the tolerance limits of the relevant regulation. Therefore, it has been determined that these fertilizers are not suitable for the fertigation system. It is seen that 39 of the 69 fertilizers are not in compliance with the regulation and contain nutrients other than the label information.

**Table 5.** Micro element fertilizers in mineral salt form (%)
***Çizelge 5.*** *Mineral tuz formundaki mikro element gübrelerinin içerikleri (%)*

| Sample | Fertilization Formulation (Label Information) | Ca % | Mg % | S % | Na % | B % | Cu % | Fe % | Mn % | Zn % | Se % | Co % | Mo % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 24% Mn $MnSO_4.4H_2O$ | 0.17 | - | 18.89 | 0.17 | 0.01 | 0.01 | - | 30.33 | - | 0.09 | - | - |
| 2 | 22% Zn $ZnSO_4.7H_2O$ | - | - | 22.72 | 0.15 | - | 0.01 | - | - | 24.03 | 0.01 | - | - |
| 3 | 22% Zn $ZnSO_4.7H_2O$ | 0.16 | - | 11.64 | 1.06 | 0.01 | 0.02 | - | - | 22.09 | - | - | - |
| 4 | 22% Zn $ZnSO_4.7H_2O$ | - | - | 12.50 | 0.14 | - | 0.02 | 1.00 | - | 23.43 | - | - | - |
| 5 | 0.2% B, 0.5% Cu, 6% Fe, | 0.10 | - | 18.19 | 0.29 | 0.37 | 0.86 | 8.77 | 4.95 | 6.89 | 0.03 | - | - |
| 6 | 0.6% B, 1% Cu, 5% Fe, | 0.32 | - | 10.27 | 0.34 | 0.03 | 1.43 | 4.73 | 2.84 | 2.98 | - | - | - |
| 7 | 1.5% B 0.6% Cu, 4% Fe, | 0.05 | - | 7.79 | 3.63 | 0.35 | 0.86 | 4.99 | 3.60 | 4.90 | 0.01 | - | 0.04 |
| 8 | 0.5% B, 1% Cu, 6% Fe, | 0.60 | - | 19.50 | 0.57 | 0.41 | 1.53 | 9.55 | 6.35 | 11.95 | 0.01 | - | - |
| 9 | 1% Cu, 4% Fe, 2% Mn, | 0.32 | - | 10.27 | 0.34 | 0.03 | 1.43 | 4.73 | 2.84 | 2.98 | - | - | |

(-): Unidentified

## 4. Heavy Metals

There are no heavy metal limit values determined for drip irrigation system fertilizers in Turkey. In this study, heavy metal contents in Turkish fertilizers were evaluated according to the limit values (MEP, 2002) in China and Japan.

**Table 6.** Heavy metal limit values in fertilizer (MEP, 2002)
***Çizelge 6.*** *Gübre ağır metal sınır değerleri (MEP, 2002)*

| Heavy metal | Limit values |
|---|---|
| As | ≤75 |
| Cd | ≤10 |
| Cr | ≤150 |
| Hg | ≤5 |
| Pb | ≤100 |

**Table 7.** 69 Minimum and maximum heavy metal contents of the 69 drip irrigation fertilizers (mg $kg^{-1}$ ).
***Çizelge 7.*** *69 Adet damla sulama sistemi gübresine ait minimum ve maksimum ağır metal içerikleri mg $kg^{-1}$)*

| Heavy Metal | Multi-Nutrient | | Micro-Nutrient | |
|---|---|---|---|---|
| | Min. | Max. | Min. | Max. |
| | mg $kg^{-1}$ | | | |
| As | 0.07 | 36.34 | 0.23 | 29.11 |
| Cd | 0.01 | 1.70 | 0.05 | 4962.68 |
| Cr | 0.05 | 27.00 | 0.21 | 86.00 |
| Hg | 0.01 | 2.96 | 0.36 | 13.75 |
| Ni | 0.08 | 6.77 | 1.43 | 110.68 |
| Pb | 0.01 | 7.55 | 1.62 | 100.22 |

Heavy metal contents of the fertilizers are given in Table 6, as minimum and maximum values. The data given in Table 4 have been compared with previous studies on this subject and it can be stated that 10 fertilizers out of 69 fertilizers are not particularly suitable for Hg, Cd and Hg in terms of heavy metal content. Köleli et al. (2005) stated that the Cd in the raw material for P fertilizers and P fertilizers vary between 23-179 mg $kg^{-1}$. According to Rui et al. (2008), the amount of Hg in fertilizers ranges between 5-10 mg $kg^{-1}$.

In conclusion, if the heavy metal findings in this study are compared with the tolerance values used in China and Japan, results put forth that 10 (5xHg, 4xCd and 1xHg) fertilizers are not suitable to be used in drip irrigation systems.

## 5- $^{238}U$, $^{232}Th$, $^{40}K$ Conditions of the Fertilizers

The levels of $^{238}U$ and $^{232}Th$ in three-nutrient drip irrigation fertilizers were found to be below ND (not measurable) and it was concluded that these fertilizers do not contain radionuclide in terms of $^{238}U$ and $^{232}Th$. Depending on the potassium ($K_2O$%) levels, some radioactivity was detected due to the 40K isotope. Indeed, a high correlation (r=0.89) was found between $K_2O$% and $^{40}K$ radionuclide content. This result is important in showing that the level of $^{40}K$ radionuclide increases as $K_2O$% increases. This is due to the presence of 0.0118% of the $^{40}K$ isotope in the K source.

No $^{238}U$, $^{232}Th$ and $^{40}K$ activities have been observed in the micronutrient fertilizers and radionuclide has been observed to be below detectable limits in all samples.

## CONCLUSION

At the end of the research, it was concluded that the fertilizers did not contain radionuclide in terms of $^{238}U$ and $^{232}Th$ and did not contain $^{40}K$ radioactivity that would endanger human health, and that these amounts were natural.

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