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# DETERMINATION OF LABILE POOL OF IRON IN CITRUS SOILS OF ANTALYA REGION BY ISOTOPIC EXCHANGE

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ABSTRACT

This investigation was made to determine the equilibration time and the labile pool (LP) of iron by isotopic exchange and the effect of pH of the extracting mixture on the LP in representative Citrus Soils of Antalya Region. The results obtained can be summarized as follows:

1. Isotopic exchange of $^{59}Fe$ reached a steady state concentration after 48 hours of equilibration.
2. The proportions in soil solution of the isotopically exchangeable Fe with pH 5,7 and 8 were 82.76, 77.18 and 76.34 percent, respectively.
3. DTPA-$CaCl_2$-NaAc mixture at pH 5 resulted higher labile pool of Fe than at pH 7 and 8. LP of Fe was decreased with increasing of pH.

ÖZET

Bu araştırma Antalya Turunçgil Bölgesi Topraklarında isotopik mübadele yöntemi ile, Fe için denge zamanı, labile pool (LP) ve ekstraksiyon çözeltisinin pH'sının LP üzerine olan etkisinin saptanmasını amaçlamaktadır.

Araştırma sonuçları şöyle özetlenebilir:

1. $^{59}Fe$ mübadele yolu ile 48 saat sonra sabit konsantrasyona ulaşmıştır.
2. İsotopik olarak mübadele olabilen Fe, pH 5,7 ve 8'de sırasıyla % 82.76, % 77.18 ve % 76.34 olarak bulunmuştur.
3. pH'sı 5.0 olan DTPA-$CaCl_2$-NaAc ekstraksiyon çözeltisinde pH 7 ve 8'e göre daha yüksek LP değerleri elde edilmiş ve Fe'e ait LP değerleri pH yükseldikçe azalmıştır.

## INTRODUCTION

In the field of soil fertility various methods based on tracer technique have been developed and used in determining of available nutrient contents of soils. The amount of available nutrients in the soil sometimes referred to as labile pool.

Labile pool essentially was defined by Lopez and Graham /1/ as "the amount of nutrient element in the soil solution and solid phase as measured either by chemical equilibrium or plant uptake utilizing isotopic exchange".

Radioisotope usage has done much further to understand nutrients pool. Recently more attention has been directed specially toward the labile pool of micro nutrients and the results of the studies related with this subject showed clearly that radioisotopes may provide useful information for predicting the soils supply of micro nutrients for plants.

Several investigators /1-4/ have been studied labile pool of micro nutrients and they found mainly:

1. The low concentrations of micro nutrients generally presented a problem for accurate determination of the labile pool, particularly in deficient soils.

2. Labile pool of micro nutrients generally varied with pH of the extracting mixture and at pH 5 resulted higher labile pool than at pH 6,7 and 8;

3. After 48 hours of equilibration isotopic exchange reached a steady state concentration.

The objectives of this investigation were respectively to determine the equilibration time and the equilibrium concentration of iron in the soil solution and the solid phase as determined by isotopic exchange (labile pool) and to find out the effect of pH of the extracting mixture on the LP.

## MATERIALS AND METHODS

Eleven representative soil samples taken from 0-20 cm depth were collected from Citrus Soils of Antalya Region. Some important physical and chemical properties of the soils were determined with the methods outlined by Jackson /5/. The results of the analysis showed that the soil were belong to different textural classes and in alkaline reaction. $CaCO_3$ and organic matter contents were varied between 3.76-75.60 % and 2.05-3.83 % respectively. As compared with standard values, all macro

nutrients tested were in sufficient level in all soils. From available micro nutrients Fe, Mn and Cu were sufficient, Zn in soil 4 was deficient, in soils 6, 7, 8 and 9 were in critical level /6/.

**Determination of the equilibration time**

In order to determine equilibration time to reach a steady state concentration of iron, the exctracting mixture with pH 5 was prepared containing 0.005 M DTPA (diethylene triamine pentaacetic acid) + 0.1 M NaAc + 0.01 M $CaCl_2$ . Two different soil samples were carried out in duplicate by equilibrating 10 g of soil with 10 ml of extraction solution. Extraction solution contained 0.01 μ Ci $^{59}Fe$ per ml. Soil mixture was placed in an end-over-end shaker at room temperature. Samples were taken after 2, 6, 12, 24, 32, 48, 56, 72 and 80 hours, centrifuged, filtered, duplicate 5 ml aliquots were taken and counted in a well type scintillation head on a gamma-ray spectrometer. Stabil iron was determined with Pye-Unicam SP 2900 Atomic Absorption Spectrophotometer in the same extract and calculated specific activity.

**Determination of labile pool**

For this purpose an extraction solution was prepared having pH values of 5, 7 and 8. The extracting mixture contained 0.005 M DTPA + 0.1 M NaAc + 0.01 M $CaCl_2$. The extractants contained 0.01 μ Ci $^{59}Fe$ per ml. 1:1 soil:solution ratio and 48 hours equilibration period were used as found above. Triplicate were run on each sample (totally 11 soil samples) and all extractions. After equilibration, the samples were centrifuged and the supernatant filtered through Whatman No 42 filter paper to remove organic debris. From each sample 5 ml aliquot was counted using well type scintillation head on a gamma-ray spectrometer, and the stabil iron were determined using Atomic Absorption Spectrophotometer.

Labile pool (LP) of Fe was calculated from the values of Fe concentration and isotopic measurements using the following equation:

$$LP_x = \frac{\text{Concentration of x in soil solution (ppm)}}{\text{Radioactivity of x in soil solution (\%)}} \times 100$$

where x is the micro nutrient.

TABLE-1. Laboratory determination of labile pool of iron DTPA-$CaCl_2$-NaAc solution buffered to pH 5.0, 7.0 and 8.0

| Soil No | pH of equilibrium suspension | | | Iron concentration (ppm) in solution 'A' | | | Percent radioactivity in solution 'B' | | | Labile pool of iron (ppm) (A/B x 100) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 5.0 | 7.0 | 8.0 | 5.0 | 7.0 | 8.0 | 5.0 | 7.0 | 8.0 | 5.0 | 7.0 | 8.0 |
| 1 | 6.95 | 7.10 | 7.45 | 34.27 | 23.57 | 19.03 | 76.99 | 66.31 | 65.77 | 45.51 | 35.55 | 28.93 |
| 2 | 6.75 | 7.25 | 8.10 | 30.6 | 19.89 | 16.15 | 93.99 | 87.52 | 87.11 | 32.56 | 22.73 | 18.54 |
| 3 | 6.75 | 7.25 | 8.35 | 20.66 | 13.05 | 9.96 | 91.78 | 84.64 | 81.81 | 22.51 | 15.42 | 12.17 |
| 4 | 6.90 | 8.00 | 8.65 | 31.26 | 20.47 | 15.51 | 72.46 | 71.46 | 69.30 | 43.14 | 28.65 | 22.38 |
| 5 | 6.90 | 7.65 | 8.15 | 13.12 | 8.89 | 6.59 | 58.07 | 49.08 | 49.48 | 22.59 | 18.11 | 13.32 |
| 6 | 6.95 | 7.90 | 7.95 | 25.78 | 15.69 | 8.43 | 70.63 | 53.15 | 52.32 | 36.50 | 29.52 | 16.11 |
| 7 | 6.60 | 7.95 | 7.95 | 27.49 | 19.10 | 14.85 | 73.16 | 78.40 | 76.27 | 37.58 | 24.36 | 19.47 |
| 8 | 6.95 | 7.70 | 8.20 | 35.41 | 21.72 | 18.63 | 89.67 | 87.54 | 83.79 | 39.49 | 24.81 | 22.23 |
| 9 | 6.95 | 7.65 | 7.80 | 34.78 | 21.67 | 16.68 | 87.28 | 86.06 | 86.26 | 39.85 | 24.18 | 19.34 |
| 10 | 6.95 | 7.65 | 7.75 | 28.24 | 27.92 | 20.15 | 98.81 | 89.08 | 93.20 | 28.58 | 31.33 | 21.62 |
| 11 | 6.75 | 7.55 | 7.75 | 41.81 | 26.94 | 20.33 | 97.54 | 95.74 | 94.43 | 42.86 | 28.14 | 21.53 |

## RESULTS AND DISCUSSION

### Determination of equilibration time

As seen from fig.1 isotopic exchange of $^{59}Fe$ reached a steady state concentration after 48 hours of equilibration. This result was in agreement with the earlier reports of Lopez and Graham /1/ and Rule and Graham /4/.

### Determination of labile pool of Fe

The results obtained from the laboratory determination of labile pool of iron using DTPA-$CaCl_2$-NaAc extracting solution buffered to pH 5,7 and 8 were given in Table-1.

The data in Table-1 shows that the calculated labile pool of Fe at pH 7 and 8 were generally lower than at pH 5, with the exception of soil 10. However labile pool of Fe were found at pH 5 between 22.51ppm-45.51ppm at pH 7 between 15.42 - 35.55 ppm and at pH 8 between 12.17 - 28.93 ppm respectively. Fe percentage in solution was decreased with increasing of pH. The average proportion in soil solution of the isotopically exchangeable Fe at pH 5,7 and 8 were 82.76, 77.18 and 76.34 percent respectively. On the other hand it was reported that only 47 percent of the labile pool in the outside solution of Fe, may be estimated from the most applicable conditions in the soil /1/.

According to the results of variance analysis, the differences found between soils and soil x pH interactions were significant at 1 % level. The results of Duncan's New Multiple Range test showed that the differences found between all pH levels were significant at 5 % level.

Lopez and Graham /1/ have been reported that the decrease in labile pool may be associated with the oxidation-reduction properties of the iron concerned and the resulting of the pH of soil suspension. As it is well known oxidation to higher valence state can be induced by increasing pH of the soil suspension which may result in the formation of insoluble or slightly soluble compounds.

As a conclusion it may be said that by isotopic exchange it is not possible to make accurate estimation of labile pool for iron in citrus soils used for the investigation.

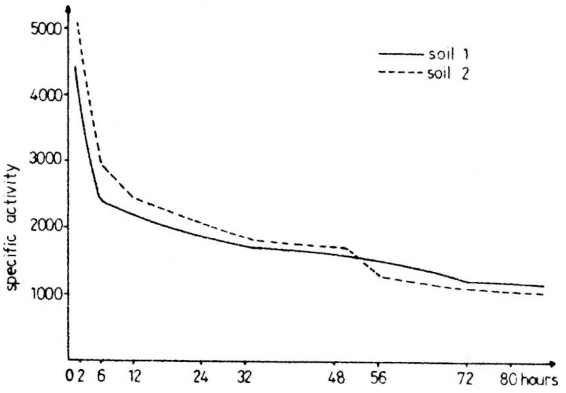



**Fig. 1. Equilibration curves for iron**

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