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COMPARISON OF PHOSPHORUS, MAGNESIUM AND ZINC DETERMINATION METHODS ON HUNGARIAN SOILS

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1. INTRODUCTION AND AIMS AND OBJECTIVES

This thesis aims to summarize the Hungarian soil analysis methods that have a background from decades of work and compare them with other internationally used methods. Our current soil testing system still provides usable results today, but following international trends, the domestic adoption of newer soil testing methods may provide new perspectives in the methodology of Hungarian soil testing. There are several methods used worldwide and each country has its own validated methods, best-suited for its soils. The harmonization of methods, measurements and indicators for the sustainable management and protection of soil resources is increasingly important to comply with the tightening legislation and boundary conditions for sustainable agricultural production. In the harmonization process, it is important to understand the background of our existing methods to work out a methodology that helps to compare and interpret the results of the different methods. The current study was designed to compare the Hungarian soil analysis methods AL (Ammonium lactate), KCl (potassium chloride), KCl-EDTA (potassium chloride ethylenediaminetetraacetic acid) methods with Mehlich 3, water extraction, CoHex (cobalt hexamine) and XRF (X-ray fluorescence) methods. The different nutrient analysis methods were compared for phosphorus, magnesium and zinc measurements with 70 samples from Hungary. Data were first compared for the whole dataset and then, in certain categories of calcium carbonate content, pH, liquid limit according to Arany and clay content.

Aims and objectives:

- The general aim is to compare the Hungarian soil analysis methods with international methods that might open new perspectives for the Hungarian laboratory analysis methodology.

- The study aims to compare the extraction efficiency of some widespread soil analysis methods (AL (Ammonium lactate), KCl (potassium chloride), KCl-EDTA (potassium chloride ethylenediaminetetraacetic acid) methods with Mehlich 3, water extraction, CoHex (cobalt hexamine) and XRF (X-ray fluorescence)) for phosphorus, magnesium and zinc measurements. An additional aim is to quantify the role of soil properties affecting extraction efficiency.

- In addition, to examine the different extraction methods, the impact of the classification of the influencing soil parameters and the statistical analyses (measuring all data or certain classes) were investigated, in the light of how these affect the evaluation of the results of soil P Mg Zn measurements.

2. MATERIALS AND METHODS

Sampling

Seventy geo-referenced soil samples from the 0-20 cm top layer (Figure 1) were taken in Hungary. Factors taken into account in this selection were land use, soil type, climate data, accessibility, and market value.

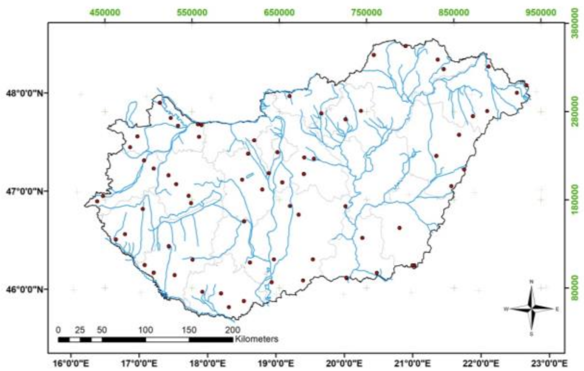


Figure 1: Sampling locations of the soil samples in Hungary

Laboratory analysis

The selection of international analysis methods was a compromise between agricultural relevance/customer expectations and multi-elemental analysis. The most common analytical procedures (extraction methods and analytical equipment) have been selected to determine the different nutrient pools. A list of possible analytical methods for determining the soil parameters was extracted from a number of ISE (International Soil-analytical Exchange Programme) Quarterly Reports (all reports from 2011) produced by the International Soil-Analytical Exchange (ISE) organized by WEPAL (Wageningen Evaluating Programmes for Analytical Laboratories). This ring test is adopted by soil testing laboratories from all over the world, routine and scientific laboratories. The number of participants varies up to about 80 for the most popular procedures.

As a conclusion of this study the following methods were selected:

- Mehlich 3
- Cobalt hexamine trichloride
- Water extraction
- XRF (X-ray fluorescence)

Mehlich 3 was selected as multielement extraction for the determination of bioavailable pool of nutrients. The Mehlich-3 analysis method is used and accepted worldwide. Furthermore, the accuracy and precision of the method show very low interlaboratory variation compared to different methods. Mehlich 3 method is implemented following Chapter 5 of Recommended Soil Testing Procedures for the Northeastern United States.

Cobalt hexamine trichloride was chosen since this method is relatively simple and allows to determine multiple bases and CEC in one procedure without compromising accuracy. The cobalt hexamine method is implemented following ISO 23470:2007.

Water extraction was chosen mainly to determine pH and EC and to measure the water-soluble forms of each component in the soil.

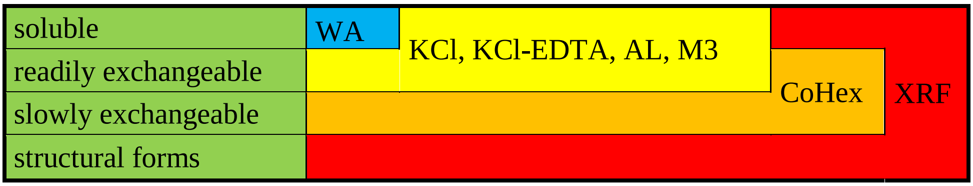
The total amount of nutrients are determined with **XRF** due to method convenience such as relatively low cost, low labour, operator and environmentally friendly. The XRF is a compromise between information that can be obtained, cost, environmental impact and accuracy. The XRF analysis is done following ISO 18227:2014 standard.

The phosphorus content analysis of the soil samples in the Hungarian laboratory was implemented according to the standard MSZ 20135:1999 with **Ammonium lactate (AL) solution.**

The Mg content of the soil samples was analyzed with **Potassium chloride (KCl) extract** using the traditionally accepted standard (MSZ20135:1999).

Zn content is analyzed with **KCl-EDTA method** that is implemented according to the Hungarian standard (MSZ 20135:1999).

Figure 2 shows the studied extractants that differ in their strength. A weak extractant represents the readily available pool of certain nutrients, whereas a very strong reagent represents a more stable pool of that nutrients.



WA – Water extraction, KCl – Potassium Chloride, KCl-EDTA – potassium chloride-EDTA, AL – Ammonium lactate, M3 – Mehlich 3, CoHex – Cobalt hexamine, XRF – X-ray fluorescence

Figure 2: Nutrient forms in soil and extraction methods

pH(KCl) was determined with a potentiometric method according to the Hungarian standard (MSZ-08-0206-2:1978).

The **$CaCO_3$-content** was determined using the gas volumetric method of Scheibler (MSZ-08-0206-2:1978).

The texture index is determined **by Liquid limit according to Arany** method based on the Hungarian Standard (MSZ-08-0205:1978).

Particle size distribution was measured using laser diffractometry (Fritsch Analysette 22 Microtech Plus).

Data analyses of the influencing factors

To evaluate the role of soil properties affecting the phosphorus, magnesium, zinc extraction efficiency samples were grouped according to pH, $CaCO_3$ content, Arany-type texture index, and clay content.

Statistical analysis

Soil properties and the analysis methods are described using descriptive statistics.

Linear regression was used to determine the linear relationship between the P, Mg, Zn determination methods, where R^2 presents a percentage of the variability explained by the model. The chosen level of significance was 5%.

Pearson correlation analysis was used to determine the relationship between the extraction methods and the soil parameters (pH, $CaCO_3$, KA, Clay).

The normality of the data series of the different analysis methods was tested with the Kolmogorov-Smirnov test. If the data of the analysis methods were not normally distributed, then a non-parametric Friedmann ANOVA test was used. If the data of the analysis methods showed normal distribution, then a parametric, Repeated Measures ANOVA test was used.

The Wilcoxon signed-rank test, a non-parametric statistical hypothesis test was used to compare the analysis methods (WA, EDTA, M3, CoHex, KCl, Al) to assess whether their mean ranks differed.

Investigating the pH (KCl), $CaCO_3$-content, liquid limit according to Arany, clay content dependence, pairwise comparison was used.

Box plots diagrams were used to display the variation in the phosphorus, magnesium, zinc determination methods.

3. RESULTS

Comparison of phosphorus determination methods

Comparing the different percentages of the measured phosphorous that each method could measure from the total amount of phosphorus (XRF), ammonium lactate solution proved to extract the highest amount of phosphorus.

Phosphorus content measured by the six methods resulted in the following order: P-WA< CoHex < P-WA(PO_4) < M3 < AL < XRF

The linear relationship between P content determined by P-WA and M3 methods was significant with the determination coefficients of 0.72 for P-WA(PO_4) vs M3 and 0.67 for P-WA vs M3.

The results of the pairwise analyses of the 5 different P measurement methods based on the percentage that each method could measure from the total amount of P (XRF) showed that M3 is not different from AL just as well as P-WA(PO_4), P-WA and CoHex produced similar values but the two groups (M3 and AL versus P-WA(PO_4), P-WA and CoHex) showed significant differences.

The boxplot analysis of the 5 different phosphorus analysis methods proved that there are two separate groups (M3, AL versus CoHex, P-WA, P-WA(PO_4).

Evaluating the phosphorus contents according to two lime categories based on the Hungarian advisory system showed that higher than 1% $CaCO_3$ content resulted in much higher phosphorus levels in the case of AL method compared to soils with lower than 1% lime content.

The higher the lime content, the higher the amount of fixed phosphates, so it means that AL method extracts not only the available but even the fixed phosphates.

Comparing the results of the AL-phosphorus method (used in Hungary) with the results of P-WA(PO_4), P-WA, CoHex and M3 methods in lime-free – low $CaCO_3$ content soils, showed weak significant linear relationships explained at 39% - 0,57 ($0.39 \leq R^2 \leq 0.57$) variance. In the category of higher lime content soils, the only significant relationship was determined between CoHex vs AL ($p=0.02$) explaining 20% of the variance. All the others were not significant ($R^2<0.2$; $p \geq 0.05$).

The pairwise analyses of the measured phosphorous percentages of the total phosphorous amounts based on the separate analyses of pH, $CaCO_3$, KA and Clay groups showed smaller differences between the methods, but the results were comparable with the pairwise analysis when all data were included. Based on the average of the number of significant results along with the four influencing factors, the highest significant difference was between P-WA and AL methods. P-WA(PO_4) vs P-WA and P-WA vs CoHex were not significantly different from each other.

Evaluating the differences based on all parameters the following order can be made (1: smallest difference 8: biggest difference):

1. P-WA(PO_4) vs P-WA, P-WA vs CoHex
2. P-WA(PO_4) vs CoHex
3. AL vs M3

4. CoHex vs AL
5. CoHex vs M3
6. P-WA(PO_4) vs AL
7. P-WA(PO_4) vs M3, P-WA(PO_4) vs M3
8. P-WA vs AL

Evaluating the differences based on all parameters it can be concluded that P-WA(PO_4) vs P-WA and P-WA vs CoHex were not significantly different from each other. The highest significant difference was between P-WA vs AL method.

Comparison of magnesium determination methods

Physicochemical properties and the chosen classification method affected the evaluation of magnesium measurements.

Mehlich 3 solution demonstrated a greater capacity of extracting Mg from the soil, compared with other extract solutions.

Magnesium content measured by the four methods resulted in the following order: WA < KCl < CoHex < M3 < XRF

The linear regression between all the pairs of Mg content measurement methods are significant, but only 4 of them explain more than 60% of the total variation. The linear relationship between KCl and CoHex methods has the highest determination coefficient (R^2=0.96), followed by WA – M3 (R^2=0.68), M3 – CoHex (R^2=0.66) and M3 – KCl (R^2=0.60).

CoHex vs KCl methods showed an unexpectedly strong relationship. However, these two methods should be more dissimilar from one another as the KCl method “only” measures the soluble and the readily exchangeable part of the Mg in the soil, while the CoHex method can also measure the slowly exchangeable part.

The KCl and M3 methods were expected to produce similar results with a high determination coefficient, but they showed a weaker relationship

(R^2=0.60). The M3 and CoHex methods had a similar low determination coefficient of 0.66.

The results of the pairwise analysis based on the percentage that each method could measure from the total amount of Mg (XRF) proved that all the methods were significantly different except for M3 and CoHex methods.

Pairwise analysis of the measured magnesium percentages compared with the total magnesium amounts (XRF) based on the Arany-type classification showed that in clayey loam texture (42–51 KA), the measured magnesium contents of each method was higher compared to the sandy texture.

Comparing the KCl method that is applied in the Hungarian advisory system with the other methods resulted in different linear relationships between the two texture groups. In the sandy, loamy texture (KA 30-42) texture soil, CoHex and KCl Mg determination methods had the highest determination coefficient (R^2=0.94) explaining 94.1% variance, followed by M3 – CoHex pair with 66.3% and M3 – KCl with 56.3% variance explained. All other pairs had significant linear relationships but with a smaller percentage of explained variance.

The effect of lime content on magnesium measurement methods was also investigated.

Higher lime content resulted in lower extracted magnesium by the four methods.

In all lime categories, the linear relationship between CoHex vs KCl methods was significant, explaining more than 95% variance (R^2>0.95 $p<0.001$).

The relationships between the measurement results of the other methods (WA vs. M3, WA vs. CoHex, WA vs. KCl, M3 vs. CoHex, M3 vs. KCl) were more dependent on the lime category.

The higher $CaCO_3$-content showed a lower determination coefficient in case of M3 vs CoHex and M3 vs KCl methods. The results of WA vs M3, WA vs CoHex and WA vs KCl pairs showed opposite results; in lime-free soils, there was no significant relationship between these methods, but the higher lime content ($CaCO_3$%>0.1) resulted in a significant linear relationship and higher determination coefficient.

The further comparison of the methods based on the influencing factors, such as pH, lime content, texture class, and clay content showed the differences between the different methods.

Linear regression and Pearson correlation analysis showed the strongest correlation between CoHex and KCL.

The pairwise analysis showed other aspects. The pairwise analysis showed that the least significant differences were between the results of M3 vs CoHex and KCl vs M3 methods.

Evaluating the differences based on all parameters the following order can be made (1 - smallest difference 6 - biggest difference):

1. M3 vs CoHex
2. M3 vs KCl
3. CoHex vs KCl
4. WA vs CoHex
5. WA vs KCl
6. WA vs M3

Evaluating the differences based on all parameters it can be concluded that M3 vs CoHex were not significantly different from each other. The highest significant difference was between the results of P-WA vs M3 method.

Comparison of zinc determination methods

Mehlich 3 solution demonstrated a greater capacity of extraction of Zn in comparison to the other extractants.

Zinc content measured by the six methods resulted in the following order: CoHex < WA < EDTA < M3 < XRF.

The relationship between EDTA and M3 Zn determination methods explains 71% variation (R^2=0.71 $p<0.001$), all the other determination coefficients are lower than 0.2 or aren't significant.

Evaluating the zinc contents according to two Arany-type texture categories based on the Hungarian advisory system showed that in loam and clayey loam texture (38-50 KA), the measured zinc from the total was lower for all of the methods compared to the measurements in sandy loam texture.

The linear relationship between the methods was much weaker in the case of loam/clayey loam soils.

In category of KA<38 EDTA and M3 methods of determining Zn have the highest determination coefficient explaining more than 84% of the total variation. EDTA vs WA relationship explains 67.3%, WA vs M3 50.6% and CoHex vs WA explains 47.4% of the total variance. In the category of KA 38-50 the determination coefficients between the pairs were smaller compared to the category of KA<38.

Based on the analyses of all data we can conclude that all methods are different. However, further analyses during the comparison of the methods based on the influencing factors, such as pH, lime content, texture class, and clay content proved that in some of the cases there are similarities among the methods and this way we can get more knowledge on the measurements and the results provided.

Based on the Pearson correlation analysis and the average of the significance levels of all the pairwise analyses of the measurements along the four influencing factors (pH (KCl), $CaCO_3$, Arany-type texture, and clay), it can be concluded that the least different methods for Zn determination are EDTA and M3 analyses methods.

Evaluating the differences based on all parameters the following order can be made (1 - smallest difference 6 - biggest difference):

1. EDTA vs M3
2. CoHex vs WA
3. WA vs EDTA
4. WA vs M3
5. CoHex vs EDTA
6. CoHex vs M3

We can summarize that not only the extraction method but also some soil physicochemical properties and the chosen classification method affect the evaluation of zinc measurements.

Based on these results, an important conclusion can be made: analyzing all-inclusive data can result in very strong and significant differences between the applied method but it can be misleading as the in-depth analysis can prove otherwise.

4. CONCLUSIONS

Our current soil testing system still provides usable results today, but following international trends, the domestic adoption of newer soil testing methods may provide new perspectives in the methodology of Hungarian soil testing. As we saw there are several methods used worldwide and the harmonization of methods, measurements and indicators for the sustainable management and protection of soil resources are increasingly important to comply with the tightening legislation and boundary conditions for sustainable agricultural production. In the harmonization procedure it is important to understand the background of our existing methods then to work out a methodology to compare the different methods and then harmonize the results.

The basic dataset of my thesis was seventy geo-referenced soil samples taken in Hungary, differing in soil typology, texture, and pH. These samples were analyzed with different extraction methods in the laboratory. I compared and evaluated the existing Hungarian soil analysis methods, AL, KCl, KCl-EDTA methods with Mehlich 3, Water, Cobalt Hexamine and XRF methods for phosphorus, magnesium and zinc measurements.

The novelty in the comparison is that the amount of P, Mg and Zn measured with different analysis methods were compared to the total contents measured with the XRF method. It was obvious, based on the data analysis that the XRF method measured significantly higher amounts, meaning a thousand times more than other methods.

Data were first compared for the whole dataset and then, in certain categories of $CaCO_3$-content, pH, liquid limit according to Arany and clay content.

It was proved that categorization of the influencing factors resulted in different correlation strengths than the analyses of the overall data, presuming that the increasing trend of clay, liquid limit according to “Arany” and lime content and the pH values were not proportional with the increase or decrease of the efficiency of the measurements.

The analyzed groups can also highlight where the correlation was strong, weak or none, shedding light on the range where the influencing magnitude had significance.

Furthermore, a list of the compared measurement pairs is provided, based on the number of significant differences calculated in all of the analyzed categories of the influencing factors

Based on these results, it was concluded that analyzing all-inclusive data can result in very strong and significant differences between the applied methods. But it can be misleading as the in-depth analysis can prove otherwise.

Comparison of the methods based on the influencing factors proved that in some of the cases there are similarities among the methods and this way we can get more knowledge on the measurements and the results provided.

Despite traditional soil, tests are available in huge numbers, with learning more about soils analysis methods and their interpretations, could lead to a new dimension in Hungarian soil science.

In conclusion not only the well-known extraction methods and the soil but also the chosen classification method of the properties and also, the statistical analysis (measuring all data or certain classes) affect the evaluation of P, Mg, Zn measurements. This comparative analysis study can provide a guide to interpret the different analysis methods on the way of harmonization.

5. NEW SCIENTIFIC FINDINGS

1. It was proved that not only the extraction methods but also the chosen classification of the influencing soil parameters and the statistical analysis (measuring all data or certain classes) affected the evaluation of the results of soil phosphorus, magnesium and zinc Mg Zn measurements.

2. The orders of magnitude of the extraction efficiency of phosphorus, magnesium and zinc measurements are determined based on evaluating the following methods: WA – water extraction, KCl – potassium chloride, KCl-EDTA – potassium

chloride ethylenediaminetetraacetic acid, AL – ammonium lactate, M3 – Mehlich 3, CoHex – cobalt hexamine, XRF – X-ray fluorescence.

Based on the results, the phosphorus content measured by the six methods resulted in the following order of measured magnitude:

P-WA< CoHex < P-WA(PO_4) < M3 < AL < XRF

Based on the results, the magnesium content measured by the six methods resulted in the following order of measured magnitude:

WA < KCl < CoHex < M3 < XRF

Based on the results, zinc content measured by the six methods resulted in the following order of measured magnitude:

CoHex < WA < EDTA < M3 < XRF

3. Evaluating the differences in P determination methods based on all parameters the following order can be made:

    1. P-WA(PO_4) vs P-WA, P-WA vs CoHex
    2. P-WA(PO_4) vs CoHex
    3. AL vs M3
    4. CoHex vs AL
    5. CoHex vs M3
    6. P-WA(PO_4) vs AL
    7. P-WA(PO_4) vs M3, P-WA(PO_4) vs M3
    8. P-WA vs AL

Evaluating the differences in Mg determination methods based on all parameters the following order can be made:

1. M3 vs CoHex
2. M3 vs KCl
3. CoHex vs KCl
4. WA vs CoHex
5. WA vs KCl
6. WA vs M3

Evaluating the differences in Zn determination methods based on all parameters the following order can be made:

1. EDTA vs M3
2. CoHex vs WA
3. WA vs EDTA
4. WA vs M3
5. CoHex vs EDTA
6. CoHex vs M3

4. The linear relationship between P content determined by P-WA and M3 methods was significant with the determination coefficients of 0.72 for P-WA(PO_4) versus M3 and 0.67 for P-WA versus M3.

5. Comparing the results of AL-phosphorus method with the results of P-WA(PO_4), P-WA , CoHex and M3 methods in lime-free – low lime content soils ($CaCO_3$ w/w % < 1), showed weak significant linear relationships explaining at 39–57% variance ($0.39 \leq R^2 \leq 0.57$). In the category of higher lime content soils ($CaCO_3$ w/w % >1) the only significant relationship was

determined between CoHex versus AL ($p=0.02$) explaining 20% of the variance.

6. The linear relationship between KCl and CoHex methods had the highest significant determination coefficient, ($R^2=0.96$ $p<0.001$) then followed by WA versus M3 ($R^2=0.68$, $p<0.001$), M3 versus CoHex ($R^2=0.66$, $p<0.001$) and M3 versus KCl ($R^2=0.60$, $p<0.001$).

7. In all lime categories, the linear relationship between CoHex versus KCl methods was significant, explaining more than 95% variance ($R^2>0.95$ $p<0.001$).

   However, the higher $CaCO_3$-content ($CaCO_3$ w/w % > 0.1) showed a lower determination coefficient in case of M3 versus CoHex and M3 versus KCl methods.

   The results of WA vs M3, WA vs CoHex and WA vs KCl pairs showed opposite results; in lime-free soils there was no significant relationship between these methods, but the higher lime ($CaCO_3$ w/w % > 0.1) content resulted in a significant linear relationship and higher determination coefficient.

8. The linear relationship between Zn content determined by KCl-EDTA and M3 methods is significant, with 0.71 determination coefficient ($R^2=0.71$, $p<0.001$).

6. LIST OF MY SCIENTIFIC PUBLICATIONS ON THE TOPIC OF THE DISSERTATION

Peer-reviewed papers in Hungarian

VONA, V., BAKOS, I. A., GICZI, ZS., KALOCSAI, R., VONA, M., KULMÁNY, I. M., CENTERI, CS. (2020): MÚLT-JELEN-JÖVŐ a hazai mezőgazdasági talajvizsgálatokban. Agrokémia és Talajtan.69.1-2: 127-151.

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DORKA-VONA, V., KALOCSAI, R., SARJANT, S., GICZI, ZS., VAN, ERP P., KOVÁCS, A. J. (2018): Talajvizsgálat Magyarországon: az infravörös és röntgen-fluoreszcensz analízis alkalmazásának lehetőségei. Acta Agronomica Óváriensis.59.2: 32-43.

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VONA, V., CENTERI, CS., GICZI, ZS., KALOCSAI, R., BIRO, ZS., JAKAB, G., MILICS, G., KOVACS, A. J. (2020): Comparison of magnesium determination methods on Hungarian soils. Soil and Water Research.15.3:173-180.

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Papers published in full conference proceedings

VONA, V., SARJANT, S., LULEVA, M., KULMÁNY, I. M., VONA, M. (Can local samples improve the accuracy of mid-infrared (MIR) and X-ray fluorescence (XRF)-based spectral prediction models? Precision Agriculture '21 /, Stafford, J.V. - Wageningen: Wageningen Academic Publishers. ISBN: 978-90-8686-363-1:443 - 449.

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Book

MALDA, J. T., **DORKA-VONA, V**., RUTGERS, R. (2015): III. Tápanyag - gazdálkodás, energianövények In: Gelencsér G., Farkas D., Vona M.(szerk.): Fenntartható természeti erőforrás gazdálkodás kötetsorozat Tananyag a SEE-REUSE Projekt keretében Völgy Hangja Fejlesztési Társaság Közhasznú Egyesület Törökkoppány.:124. (ISBN 978-615-80209-0-9)

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Further papers

https://m2.mtmt.hu/api/author/10071935