

Soil and Water Science

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DETERMINING LEAD ACTIVITY IN AN EGYPTIAN ALLUVIAL SOIL USING COMPETITIVE CHELATION METHOD

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Received: 04/04/2017 ; Accepted: 30/05/2017

ABSTRACT: Lead has been emitted into atmosphere since the birth of metallurgy in ancient times and with increasing intensity since the establishment of medieval and modern industries. In the present study lead activity was measured in six alluvial soil samples which were collected from 6 successive layers (15-cm thick, each) from surface soil to 90 cm depth to identify the various Pb minerals which might control Pb activities in that soils (using constructed lead stability mineral diagrams). The Pb content decreased with increasing soil depth, ranging from 20.50 µgg⁻¹ in the surface layer to 11.03 μ gg⁻¹ in the deepest soil sample, which mostly reflect the impact of anthropogenic influences on lead– contamination in soil. The values of log Pb²⁺ activities varied from -4.9498 to -7.1098 and were inversely correlated with pH. The predicted relationship equation could be: Log $(Pb^{2+}) = 9.619-2.111$ (pH), indicating that lead solubility in soil decreased about 100 folds for each pH unit increase. Plotting Pb activities on the constructed stability diagram of various Pb minerals indicated the solubility of PbO being too soluble to persist under normal conditions .Pb₂SiO₄ and PbSiO₃ minerals in equilibrium with SiO₂ (soil) or SiO₂ (quartz) were too soluble to regulate Pb^{2+} activities. $Pb_5(PO_4)_3Cl$ was the most insoluble of lead phosphate minerals and controls lead solubility pH range of most soils. Values of Pb²⁺ were lower than those maintaining by Pb₅ throughout (PO₄)₃.OH mineral. Pb- mixed carbonate and hydroxide minerals indicating that PbCO₃.PbO, was too soluble to persist. Values of Pb^{+2} were super saturated with respect of $Pb(OH)_2$, $PbCO_3$ and $Pb_3(CO_3)_2(OH)_2$ at 0.0003 atm.CO₂. In presence of 10⁻³ M SO₄, PbSO₄ mineral limits Pb at 10^{-4.79} M and its solubility changes one log unit per each log unit change in SO_4^{-2} . In the high-lead surface soil sample Pb^{+2} activity values were maintained by $PbSO_4$. PbO and PbSO4 (log SO₄ at -2), confirming the importance of studying environmental chemistry of the heavy metals.

Key words: Lead, activity, chelation, stability diagram, Pb-minerals.

INTRODUCTION

Daunting challenges of Egypt are the problems, resulting from human activities, such as fast unbalanced population growth, undernourishment, unplanned urbanization, adverse land use, dangerous and harmful wastes (El-Gendi *et al.*, 1997; Badawy and El-Motium, 2002; Abd El-Aziz, 2014), unconscious energy consumption, heavy metal contamination created by industrial wastes (Esawy and Adel Mohamed, 2016) and from traffic emission (El-Gendi, 1994; Ahmed *et al.*, 2015). The majority of the heavy metals are toxic to the living organisms and even those considered as essential can be toxic if present in excess. The heavy metals can impair important biochemical processes posing a threat to human health, plant growth and animal life (Kabata-Pendias and Pendias, 1992; Chibuike and Obiora, 2014).

Lead is widespread in the environment. This toxic element has been emitted into atmosphere since the birth of metallurgy in ancient times and with increasing intensity since the

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establishment of medieval and modern industries (Settle and Patterson, 1980; Sripathy et al., 2015). It has been established that in most circumstances, the concentration as well as the activity of free metal ions is the key factor in determining metal bioavailability and toxicity (Alloway, 1995; Candelaria et al., 1995). The ion activity of trace metals can be used in thermodynamics to describe the direction and impetus of chemical reactions such as precipitation, complexation and adsorption (Santillan-Medrano and Jurinak, 1975). There are several approaches that have been widely used to determine the free ion concentration (activity). Each method has its advantages and limitations. The electrochemical method of ion selective electrode (ISE) potentiometry is one of the most powerful methods in speciation studies because the activity of free metal ion is measured directly (Mota and Correia dos Santos, The voltammetric electrochemical 1995). methods such as anodic stripping voltammetry (ASV) and adsorptive cathodic stripping voltammetry (ACSV) provide the most direct methods for the study of trace metal speciation in low concentrations. The competitive chelation method has good sensitivity and provides estimates of ion activities (Amacher, 1984). However, chelates attain equilibrium with soils very slowly (Norvell and Lindsay, 1982; Workman and Lindsay, 1990). Accuracy of the competitive chelation method depends on the reliable estimation of chelated metal contents and reference ion concentration. The method can be successful only if the metal of interest and the selected competing metal are the principal chelated metals (Workman and Lindsay, 1990). Therefore, the main objectives of the present work were to determine the active portion of Pb in alluvial soils and to identify its various solid phases in these soils along with knowledge of their solubility and kinetic of dissolution and precipitation using stability diagrams.

MATERIALS AND METHODS

Samples from Six layers from an alluvial soil from El-Kanater Horticulture Research Station (25 Km from Cairo) were taken. From each layer one composite soil sample was collected successively (at 15-cm thickness from surface layer to 90 cm depth) with a contention that deepest layer would be the least exposed to Pb pollution. By this way six soil samples varying in Pb concentration were obtained. Main properties of the tested soils were determined by (Chapman and Pratt, 1961; Black, 1965) and listed in Table 1.

Soil pH was measured in 1 : 2 soil: water ratio using 0.01 M CaCl₂ as background solution to dimension the influence of ionic strength inference among the tested samples as described by Lindsay and Norvall (1978).

 $\mathbf{P}\mathbf{h}^{+2}$ activity was measured using the competitive chelation method given in details by El-Gendi (1994). This method depends on reacting soil with a series of chelate solution having different mole fractions of Pb and a competing Cd metal. A series of chelating solutions, having different mole fractions of chelate ligand (L), that is (PbL/PbL+CdL), were prepared using reagent grade diethylene triaminepentaacetic acid (DTPA), Pb(NO₃)₂ and Cd(NO₃)₂. The initial PbL/ (PbL+CdL) mole fractions used varied from 0.0 to 0.3 and included five to ten different mole fractions. The DTPA concentration was fixed at 100 µM in all cases.

Fifteen–gram subsamples of each soil were weighed in 125 ml Erlenmeyer flasks, and 30 ml of 0.01 M of CaCl₂ solution having a given PbL/(PbL+CdL) mole fraction was added along with 10 mg of CdCO₃ to maintain a known Cd²⁺ activity in the solution. For each soil, a blank treatment without a chelating agent was prepared by shaking 15 g of soil with 30 ml of 0.01M CaCl₂ solution. The Erlenmeyer flasks were then covered with perforated parafilm to allow gas exchange with the atmosphere. The suspensions were shaken for 5 days, after which pH was measured, then centrifuged, filtered and the clear supernatant were analyzed for Pb and Cd using atomic absorption spectrophotometer.

Calculation

The final mole fraction of Pb/ (PbL +CdL) for each soil was calculated from the total soluble Pb and Cd after subtracting Pb and Cd in the CaCl₂ blank treatment. A graph relating initial and final PbL/(PbL +CdL) mole fractions was made for each soil, and the equilibrium PbL/ (PbL +CdL) mole fractions were obtained Fig.1, which was used to calculate Pb²⁺ activity in the soil.

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Table 1. Main properties of the investigated soil samples

Soil characteristics	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample6
	(0 – 15cm)	(15–30cm)	(30 – 45 cm)	(45 – 60cm)	(60 – 75cm)	(75 – 90cm)
pH	07.12	07.35	07.24	07.36	07.30	07.37
EC (μ s cm ⁻¹)	01.37	01.46	01.41	01.38	01.42	01.43
CEC (Cmol _c Kg ⁻¹ soil)	44.15	40.28	36.10	35.21	37.25	36.18
Organic matter (gKg ⁻¹)	25.40	21.50	18.20	15.60	17.10	16.50
CaCO ₃ (gKg ⁻¹)	18.70	19.10	12.60	14.30	11.20	10.90
Total Pb (µgg ⁻¹)	20.05	16.71	12.70	11.43	11.26	11.03
Available Pb (µgg ⁻¹)	01.59	01.31	01.42	01.37	0.94	01.02



Fig. 1. Changes in CdL/(CdL+ PbL) mole fraction in the tested soil samples

Derivation of the equilibrium equation used to calculate Pb⁺² activity is given as follows:

$$Log^{m}_{0.01}$$

$$PbL^{3-} === Pb^{2+} + L^{5-} - 19.93 \quad (1)$$

$$Cd^{2+} + L^{5-} == CdL3 - +20.27 \quad (2)$$

$$PbL^{3-} + Cd^{2+} == Pb^{2+} + CdL^{3-} + 0.34 \quad (3)$$

$$WL = 0$$

Where:

 $k_{\rm m}$ is the mixed equilibrium constant expressed in terms of concentration, except for H⁺, OH⁻, and e⁻ which, if present, are expressed as activities (Lindsay, 1979), L⁵⁻ is the concentration of free DTPA, and [] indicate molar concentrations.

Rearranging Eq. 3; gives :

$$[Pb^{2+}] = [PbL^{3-}]/[CdL^{3-}] \times 10^{0.34} \times [Cd^{2+}] \dots (4)$$

Since the activity coefficients of Pb^{2+} , Cd^{2+} PbL²⁺and CdL³⁻ are equal; Equation 4 can be written in terms of activities, as follows:

$$(Pb^{2+}) = (PbL^{3-})/(CdL^{3-}) \times 10^{0.34} \times (Cd^{2+}) - ---(5)$$

Inputs to eq. (5) include $(PbL^{3-})/(CdL^{3-})$ and (Cd^{2+}) . Values for $(PbL^{3-})/(CdL^{3-})$ were obtained from the measured $[Pb^{2+}]=[PbL^{3-}]/[CdL^{3-}]$ obtained for each soil.

Since $CdCO_3$ was added to the soil suspensions to control Cd^{2+} activity throughout the experiment and the solutions were open to atmospheric CO_2 , Cd^{2+} activity was obtained according to Lindsay (1979) as follows:

$$\begin{split} CdCO_3(C) &+ 2H+ === Cd^{2+} + CO2(g) + H_2O\\ LogK^o & 6.16 \dots (7)\\ (Cd^{2+}) &= 10^{6.16} * (H^+)^2/CO_2 \ , \ at \ 0.003 \ atm \ .CO_2 \end{split}$$

Hence, $(Cd^{2+})=10^{9.68} * (H^{+})^2 \dots (8)$

Substituting Eq. 8 into Eq. 5 gives:

 $(Pb^{2+})=(PbL)/(CdL)*10^{(10.02-2pH)}....(9)$

The pH value used in Eq.9 was the pH of the suspension closest to the equilibrium point in the initial and final mole fraction plot.

Stability Diagrams

Lead minerals that may control the level of Pb^{2+} in soils were plotted on an equilibrium solubility diagram in terms of Pb^{2+} activity and pH (Fig. 3) using the thermodynamic data in

Table 2 taken from Lindsay (1979) to calculate the equilibrium relationships in the following manner;

 $PbCO_{3(c)}{+}2H^{+}{=}{=}{=}Pb^{2+}{+}CO_{2(g)}{+}H_{2}O \quad 4.65 \ (Log \ K^{o})$

$$Log Pb^{2+} = 4.65 - Log CO_2 - 2pH$$

At $CO_2 = 0.0003$ atm , this equation becomes;

$$Log Pb^{2+} = 8.17 - 2pH$$

At $CO_2 = 0.003$ atm, this equation becomes;

Log Pb $^{2+}$ = 7.17 -2pH

The remaining minerals were plotted in same manner, meanwhile lead silicate minerals were depicted in equilibrium with SiO_2 (soil) and SiO_2 (quartz). Lead phosphate minerals were depicted in equilibrium various phosphate minerals which in turn depend on level of $CaCO_3$ which in turn controlled by CO_2 partial pressure.

RESULTS AND DISCUSSION

A plot of the final PbL/(PbL+CdL) mole fraction of the filtrates against the initial mole fractions are presented from Figs. (1a to 1f). The intersected between them pointed out to the equilibrium point, where the soil sample neither gain nor loss of Pb. This equilibrium value was used in the following equation to calculate Pb activities by using the following formula; and the results are listed in Table 2.

$$(Pb^{2+}) = (PbL) / (CdL) * 10^{(10.02 - 2pH)}$$

It is clear from the Figs that at low concentrations of initial mole fraction; Pb released from the soil sample to the equilibrium solution, whereas at high concentrations; the chelate lost Pb.

As shown in Table 3 the values of log Pb^{2+} activity varies widely among the tested soil samples; being from -4.9498 to -7.1098. Log Pb activity increased with increasing available Pb (r of 0.970, highly significant correlation).

On the other hand, as shown in Fig 2 the correlation between logarithmic Pb^{2+} activity and soil pH being very highly significant correlated (r=-0.997) and the predicted equation is;

 $Log \ Pb^{2+} = 9.619 - 2.111(pH)$

Equilibrium reaction	Log K ^o
$Pb(OH)_2 + 2H^+ ==== Pb^{2+} + 2H_2O$	8.16
$PbCO_3 + 2H^+ === Pb^{2+} + CO_2(g) + H_2O$	4.65
$Pb_3(CO_3)_2(OH)_2 + 6H + = = 3Pb^{2+} + 2CO_2 + 4H_2O$	17.51
$PbCO_3.PbO_{\odot} + 4H + ==== 2Pb^{2+} + CO_2 + 2 H_2O$	17.39
$PbSiO_3 + 2H^+ + H_2O = = Pb^{2+} + H_4SiO_4$	5.94
$Pb_2SiO_4 + 4H^+ === = 2Pb^{2+} + H_4SiO_4$	18.45
$PbHPO_4 + H^+ = Pb^{2+} + H_2PO_4$	-4.52
$Pb_3(PO_4)_2 + 4H^+ = = = = = = = 3Pb^{2+} + 2H_2PO_4$ -	-5.26
$Pb_4O(PO_4)_2 + 6H^+ = = = = = = 4Pb^{2+} + 2H_2PO_4 - H_2O$	2.24
$Pb_5(PO4)_3OH + 7H^+ ==== 5Pb^{2+} + 3H_2PO_4 + H_2O$	-4.14
$SiO_2(soil) + 2 H_2O === H_4SiO_4$	-3.10
$SiO_2(quartz) + 2 H_2O === H_4SiO_4o$	-4.00
$CaCO_3 + 2H^+ = = Ca^{2+} + CO_2 + H_2O$	9.74
$CaHPO_{4}.2H_{2}O + H^{+} = = = Ca^{2+} + H_{2}PO_{4^{-}} + 2H_{2}O$	0.63
$(Ca_3PO_4)_2 + 4H^+ = = = 3Ca^{2+} + 2H_2PO_4$ -	10.18
$Ca_5(PO_4)_3OH + 7H^+ = = 5Ca^{2+} + 3H_2PO_4 - +H_2O$	14.46

Table 2. The equilibrium activity constants $({\bf k}^{\rm o})$ of some lead minerals

 Table 3. Calculated Pb activity in the tested soil samples:

Sample No	Soil depth (cm)	Final mole Fraction	pH eq.	Log Pb ²⁺
1	0 – 15	0.040	6.830	-4.9498
2	15 -30	0.100	7.200	-5.26606
3	30 - 45	0.080	7.080	-5.26494
4	45 -60	0.040	7.170	-5.63876
5	60 -75	0.040	7.750	-6.7898
6	75 -90	0.040	7.910	-7.1098



Fig. 2. Lead activities as a function of pH for the tested soil samples

This relationship indicates that Pb solubility in the soils decreased about 100 folds for each pH unit increase.

Lead as shown in Fig. 3 forms numerous minerals including Silicate (*i,e.*, $PbSiO_3$ -alamosite); oxides (*i.e.*, PbO-massicot, PbO_2 -plattnerrite); carbonate (*i.e.*, $PbCO_3$ -Cerussite, $Pb_3(CO_3)_2$ (OH)₂ - hydroceussite, $Pb_2(CO_3)Cl$ -phosgenite); sulfate (*i.e.*, $PbSO_4$ -anglesite); phosphate (*i.e.*, $Pb_5(PO_4)_3CL$ -Pyromorphite); and halide (*i.e.*, $Pb_2(CO_3)Cl_2$ – phosgenite) minerals.

Fig. 3 shows that the solubility of $Pb(OH)_2$, PbCO₃ and Pb₃(CO₃)₂(OH)₂ are almost identical at 0.0003 atm. CO₂. The hydroxide Pb(OH)₂ is considerably more stable at pH 8 maintaining approximately 10^{-8} M Pb.

Fig. 3 also shows that the solubility of PbO are too soluble to persist under normal conditions. For example, even at pH 8, the PbO requires nearly 10^{-3} M Pb for equilibrium. Plotting the measured Pb²⁺ activities of the tested soils on the stability diagram as shown from Fig. 2 indicate that Pb₂SiO₄ mineral in equilibrium with SiO₂ (soil) or SiO₂(quartz) are too soluble to persist in the tested soils . Similar

findings also observed with $PbSiO_3$ mineral in equilibrium with either SiO_2 (quartz) or with SiO_2 (soil).

Furthermore, Pb-phosphate minerals, as shown from the figure, indicated that Pb⁺² values were lower than those maintaining by $Pb_5(PO_4)_3$.OH. As stated earlier by Lindsay (1979) solubility of Pb- phosphate depends on the level of p in the soil. In developing the figure, phosphate activity was fixed either by hydroxyl apatite (HA) or by tricalcium phosphate (TCP) and calcite at 0.003atm.CO₂. The solubilities of the various Pb phosphate minerals decrease as phosphate shifts equilibrium from HA to TCP. On the other hand, it is obvious from Fig 3 that Pb_5 (PO₄)₃Cl is the most insoluble of lead phosphate minerals and has the capability of controlling Pb solubility throughout the pH range of most soils. In soils below pH 6, Pb solubility is increased as phosphate is depressed by trivalent cations such as Fe and Al. These data tend to support the condition of Nriagu (1972) that Pb phosphate formation can serve as a sink of Pb in the ecosystem.

The Figure also shows that Pb-mixed carbonate minerals, PbCO₃.PbO, is too soluble to persist in the soil, it requires only 10^{-6} M Pb for equilibrium at 0.003 atm.CO₂. On the other

hand, Pb^{+2} values were supersaturated with respect of $Pb(OH)_2$, $PbCO_3$ and $Pb_3(CO_3)_2(OH)_2$



Fig. 3. Stability diagrams of various Pb- minerals

at 0.0003 atm.CO₂ minerals. Hem (1973) concluded that in many natural surface waters the Pb²⁺ activity was regulated by either PbCO₃ or, Pb₃(CO₃)₂ (OH)₂. Fig. 3 also shows that with the increasing in CO₂, carbonate minerals become more stable and *vice versa* with the decrease in reducing CO₂. Under these conditions the solubility line of the carbonate minerals will shift downwards, reflecting lower equilibrium level of Pb²⁺ activity (*i.e.*, Pb₃ (CO₃)₂ (OH)₂ at CO₂= 0.003 atm).

Lead–sulphate minerals as shown from Fig. 3 indicate that In presence of 10^{-3} M SO₄, mineral PbSO₄ limits Pb at $10^{-4.79}$ M and its solubility changes one log unit for each log unit change in

 SO_4^{-2} . The Figure also shows that in the enriched Pb- top soil samples , Pb^{2+} activity values were maintained by $PbSO_4$.PbO and $PbSO_4$ (log SO₄ at -2).

Santillan-Medrano and Jurinak (1975) reported that Pb solubility decreased in soils as pH increased. They added that in noncalcareous soils, solubility of Pb appeared to be regulated by Pb (OH)₂, Pb₃(PO₄)₂, Pb₄O(PO₄)₂, Pb₅(PO₄)₃ OH, depending on the pH. In calcareous soils, PbCO₃ also assumed importance in auto-exhausted soils. El-Gendi (1994) reported that the Pb²⁺ activities were within the range maintained by the formation and /or mixture of PbCO₃, Pb₄O(PO₄)₂,

 $Pb_5(PO_4)_3OH$, $Pb_3(PO_4)_2$ at equilibrium with (hydroxy apatite) and $CaCO_3$ at 0.003 atm. CO_2 , and PbHPO₄ at equilibrium with (tricalcium phosphate) and $CaCO_3$ at 0.003 atm. CO_2 . This disparity in the results emphasizes studying environmental chemistry of heavy metals as well as the importance of SO_4 ligand in soil system as a tool for Pb remediation.

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تقدير نشاط الرصاص في بعض الأراضى الرسوبية باستخدام طريقة الخلب التنافسى سمير عبد الظاهر الجندى (_ محمد عبدالتواب حسن ' _ السيد أحمد الناقه ' ١ - معهد بحوث الأراضى والمياه والبيئة – مركز البحوث الزراعية – مصر ٢ - قسم الأراضى - كلية الزراعة – جامعة الزقاريق – مصر

يعتبر الرصاص من أكثر المعادن انبعاثا في البيئة منذ عرف الإنسان أهمية المعادن حتى انتشار الثورة الصناعية في العصور الوسطى والحديثة، تهدف هذه الدراسة إلى تقدير نشاط الرصاص وكذلك التعرف على أهم المعادن المتحكمة في ذوبانه في ستة عينات من الأراضي الرسوبية تم جمعها من سطح التربة حتى عمق ٩٠ سم بالتتابع (كل ١٥ سم) وذلك باستخدام طريقة الخلب التنافسي Competitive chelation method، وكذلك استخدام منحنيات الذوبان لمعادن الرصاص المختلفة للتعرف على أكثر معادن الرصاص المتحكمة في نشاطه، أوضحت النتائج أن المحتوى الكلي من الرصاص في العينات المدروسة يقل مع العمق حيث تراوحت بين ٢٠,٥ (ميكروجرام/جم تربة) في العينة السطحية إلى ١١.٠٣ (ميكروجرام/جم تربة) في العينة السفلي (٩٠ سم من سطّح الأرض)- مما يؤكد أن السّبب الأساسي لُتراكم الرصاص في الأراضي محل الدراسة هو نتيجة الفعل البشري، كذلك أوضحت الدراسة أن قيم لوغاريتم نشاط الرصاص في العينات المدروسة تراوح مابين (-٤.٩٤٩٨) إلى (-٧.١٠٩٨) وكانت العلاقة بين تلك النتائج وقيم الـ pH علاقة عكسية ومتمشية مع المعادلة الآتية: Log (Pb)²⁺ = 9.619 – 2.111(pH) كلما زاد الـ pH وحدة واحدة يقل نشاط الرصاص ١٠٠ ضعف، وبتوقيع قيم نشاط الرصاص على منحنيات الذوبان لمعادن الرصاص المختلفة يتضح أن معدن PbO يكون أكثر ذوبانا بحيث لا يمكن تواجده في الطروف العادية، كذلك تدل النتائج أن معدن Pb2SiO4 ومعدن PbSiO3 عند الاتزان مع SiO₂ سواء كان مصدرة soil or quartz يكون أكثر ذوبانا تحت ظروف الدراسة، أما بالنسبة لمعادن الرصاص المحتوية عنصر الفوسفور تدل النتائج أن معدن Pb₅(PO₄)3Cl كان أكثر معادن الفوسفات ثباتا على مدى واسع من pH بينما قيم نتائج +2b²⁺ كانت أقل من أن يتحكم فيها معدن Pb₅(PbO₄)₃OH، أما بالنسبة لمعادن كربونات وأكاسيد الرصاص المختلفة يتضح من النتائج PbCO3.PbO لا يمكن أن يتحكم في نشاط الرصاص في العينات المدروسة، في حين قيم نشاط الرصاص المتحصل عليها كانت فوق مشبعة بالنسبة لمعادن pb(OH) وكذا PbCO3 و كذا Pb₃(CO₃)₂(OH)₂ (عند ضغط .CO₂= 0.0003 atm)، أما بالنسبة لمعادن الرصاص المحتوية على كبريت تدل النتائج على أن معدن PbSO4.PbO وكذلك معدن PbSO4 (عند تركيز SO₄ قدرة v. ۰۱ مولر) يتحكمان في ذوبان الرصاص في طبقات التربة السطحية، كذلك تؤكد النتائج على أهمية در اسة البيئة الكيماوية للمعادن الثقيله في الأر اضي.

المحكمون: