

MINERAL PARTICULATE MATTER FROM THE ST. ANA LAKE SAND RELATED TO THE WATER SUSPENSIONS

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Abstract: The St. Ana Lake is the unique Crater Lake in the Eastern Europe formed by water accumulation in the Ciomadu Mare volcano cone. The shore sand contains a wide range of mineral particulate matter containing andesite with: potassic feldspar, biotite, hornblende, muscovite, hydrated potassium silicate. The dimensional measurement reveals very fine fraction of minerals having an average diameter of 1 – 10 µm mixed with some of larger microscaled particles (e.g. 100 - 300 µm average diameter) to the larger particles such gravel having an average diameter of 5 mm. The greater particles are subjected to an almost sedimentation in contact with water. The smaller particles act somehow different. We design an algorithm based on the spherical and tabular approach of mineral shape to establish a function of weight and the ascension force depending on the measured average diameter of particles. The model reveals that the tabular minerals with measured average dimension of 10 µm and beneath are subjected to the colloidal and superficial interactions remaining suspended in different water strata, meanwhile the spherical ones needs submicron dimensions to remain suspended. The measurement performed at several water strata reveals the increasing of average diameter and number of suspended explaining the variation of water properties. The suspended particle diameter and their number increase progressively with the water depth. The electrical conductivity and turbidity increase along with water depth mainly in the proximity of mud layer. The suspended biotite and hornblende presence in the water strata causes an increasing of Fe content along with water depth. Also the presence of feldspar and hydrated silicates causes a decreasing of pH value along the water depth.

Keywords: St. Ana Lake, particulate matter, suspensions, water properties

1. INTRODUCTION

St. Ana Lake is situated in Romania in the area of Călimani - Gurghiu – Harghita volcanic mountain chain. It represents the unique Crater Lake in the Eastern Europe formed by water accumulation in the Ciomadu Mare volcano cone. The earliest scientific reports on the volcanic origin of the lake were published in 1956 and 1964 (Slavoaca & Avramescu 1956, Lazar & Arghir 1964). Further the area presents a great geological interest. Some of the newest studies affect the geology of the area to Later Tertiary and Quaternary (Szakacs & Seghedi 1995, Karatson 1999). The surface strata feature andesites

with feldspars, hornblende and biotite meanwhile the deeper strata features andesite with pyroxens.

The soils resulted after volcanic rocks degradation is classified as andosols according to the current taxonomy (Florea & Munteanu 2003, Filipov et al., 2005). Some studies point out the presence of fine andosols in Harghita region nearby the volcano cone featuring large amounts of ferro and alumino silicates (Fuleky & Jakab 2007).

The rocks on the inside slope of the volcanic cone are former subjected to precipitation water interaction on the volcanic cone slopes forming a wide range of mineral particulate matter. A representative deposit of such particulate matters we

found on the St. Ana Lake shore containing large amount of fine dust and gravel. It is expected that the gravel and sand contains the minerals reported for the upper strata mainly the feldspars and clays. The clay minerals are often found as very fine particles in a large variety of soils bind with other minerals and organic compounds (Damian et al., 2010).

The lake shore sand is not yet an andosol because the lack of organic binder and humus. It is very affected by the water drainage during the rains in this condition. The mineral particles are carried out in the lake forming a suspension. It is interesting to investigate how they sediments and in which manner alter the water properties. The main goal of present research is to establish an algorithm to relate the particles in the shore sand and the variation of water properties.

2. METHODS

The gravel and sand samples were collected from 5 different points of St. Ana Lake shore. The average representative samples of gravel (ARSG) and sand (ARSS) were prepared by mixing together the collected samples from all collecting points.

The particle distribution was performed by sieving according to the standard procedures using sieves having a progressive ratio of 1.25.

The X-ray diffraction analyses were performed using a DRON 3 diffractometer equipped with data acquisition module and Matmec VI.0 software. Each mineral in the investigated samples was identified using the standard X-ray diffraction database MATCH 1.0 version 2003 from Crystal Impact Co.

The minerals distribution related to the particles morphology was observed by the mineralogical microscopy. A Carl Zeiss Jena optical mineralogical microscope equipped with a Samsung 8 Mpx digital capture was used for this purpose.

The average water samples (at least 5 different collecting points) were taken from 0, 1, 2, 3, and 4 m depth as well as average mud sample from the bottom (AMS). Samples were collected when no precipitation in area for a month before.

Electrical conductivity and pH were measured with HI 255 Combined Meter & EC/TDS/NaCl Hanna Instruments. Turbidity was measured with a turbidity meter 4193414, Hanna Instruments. Water samples were preserved with 1% HNO₃ until conductivity and pH measurements. The metal content was measured by emission spectrometry / flame atomic absorption AAS Sens Spectrophotometer, GBC Australia. For the Fe

determination, water samples were prepared as following: take 50 ml of water and bring to dry, then resume with 10 ml 0.5 M HNO₃. The used wavelengths Fe was 248.30 nm. The results are the average of three readings. The quantitative determination is made based on calibration curves constructed with standards.

3. RESULTS AND DISCUSSIONS

The particle distribution according to their diameter is one of the most representative values for particulate matters. The particle distribution for ARSG sample is presented in Figure 1a. A good distribution of particle is observed having an average diameter situated around of 5 mm.

The macro size of such particles subject them to an immediate sedimentation to the bottom of the lake without influence the water strata.

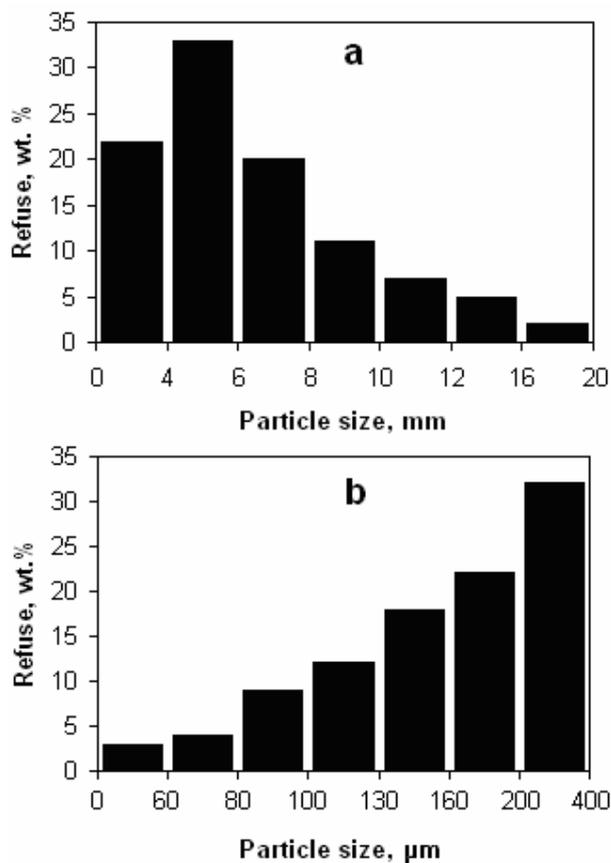


Figure 1. Particle distribution histograms for particulate matter collected from St. Ana Lake shore: a) ARSG and b) ARSS.

The particle distribution for the ARSS is presented in Figure 1b. We observe a massive grouping of the particles on the greater sizes of the plot. The ARSS average diameter is situated around of 300 μm. There are observed small fractions of

particles 4 % less than 80 μm and 3 % less than 60 μm . These amounts related to large quantities of sand on the lake shore prove to be a significant quantity of solid material able to an aqueous dispersion.

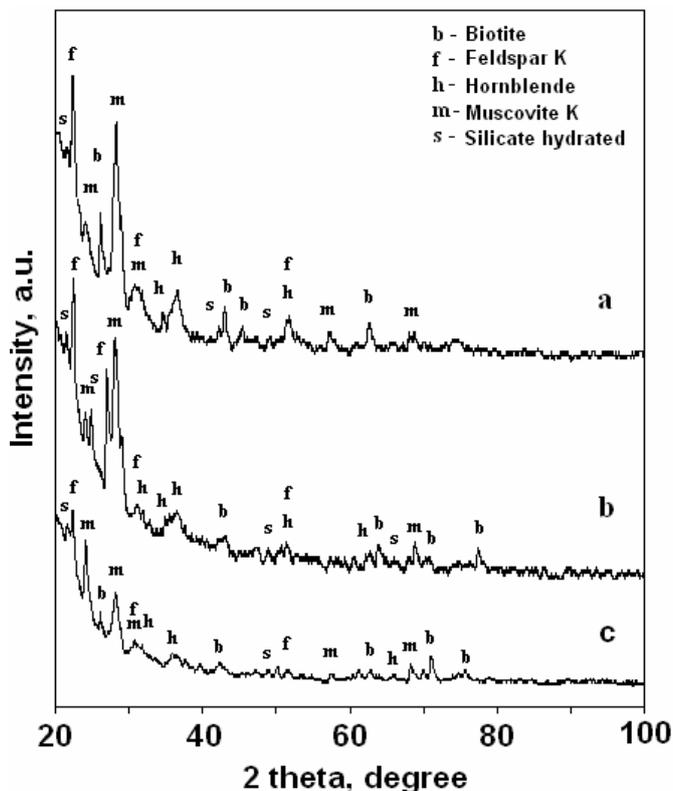
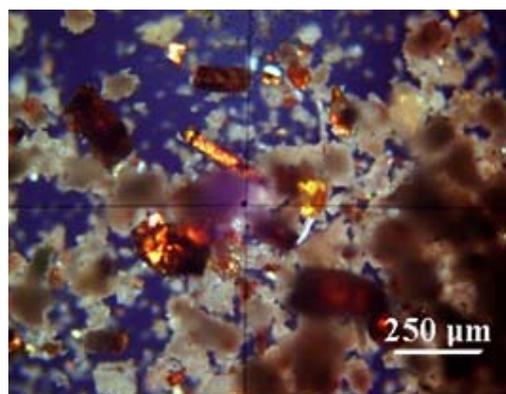


Figure 2. X – ray diffraction patterns for: a) ARSG, b) ARSS, and c)AMS.

The X-ray diffraction pattern resulted for ARSG is presented in figure 2a. It features very well developed peaks corresponding to the mineral in gravel particles. We found large amounts of biotite, hornblende, potassium feldspar, potassium muscovite, and potassium silicate hydrated. All minerals are embedded in an amorphous andesite mass of gravel particles.

The well developed shapes of diffraction peaks prove the presence of minerals in large microscale formation as shown in Figure 3a. There are some large biotite formations having around 200 μm average diameters. A single biotite sheet is presented in figure 3d. Hornblende crystals are smaller particles, around 50 μm with a greenish blue appearance, and for the feldspar, bright white.

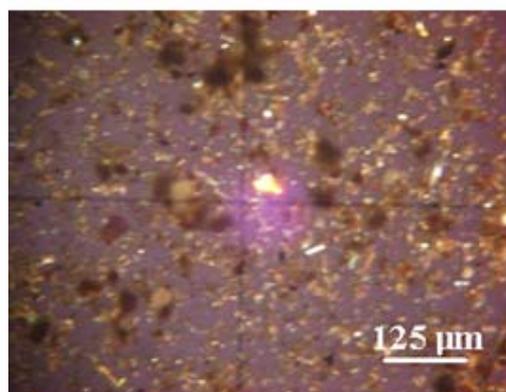
Some of our previous research figured out the mineral composition of ARSS and AMS samples (Campean et al., 2009, Campean et al., 2011). We improve the X-ray diffraction conditions for ARSS and AMS samples analysis in order to achieve more relevant diffraction peaks. The results are presented in Figure 2b and c.



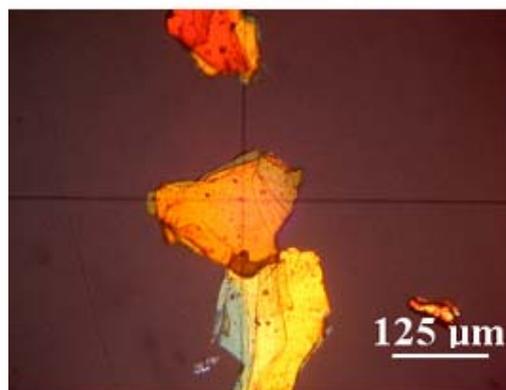
a



b



c



d

Figure 3. Optical cross polarized light microphotographs for: a) ARSG, b) ARSS, c) AMS, and d) biotite lamellas.

The X-ray diffraction pattern resulted for ARSS is presented in Figure 2b. It features well developed peaks, some of them less intense than the pattern in Figure 2a. This behavior is due to the significant refinement of the crystal particles as observed in Figure 3b. For the ARSS we found the same minerals like ARSG: of biotite, hornblende, potassium feldspar, potassium muscovite, and hydrated potassium silicate. The major difference between them is that in ARSS we found smaller particle 50 μm and less.

The more refined particle structure is observed for the AMS. The resulted X-ray pattern, Figure 2c, is very similar with the one resulted for ARSS. There are revealed the same minerals: biotite, hornblende, potassium feldspar, potassium muscovite, and hydrated potassium silicate. The amorphous andesite is also present giving a hump of X-ray pattern baseline at lower angle. Figure 3c features refined particles from with an average particle ranging from 5 to 25 μm . The orange reddish color belongs to the biotite crystals, the greenish blue belongs to the hornblende crystals and the small white spots are feldspar crystals.

The mineral structure of analyzed samples corresponds to the upper geological layer of Ciomadu Massive (Privighetorita 1970, Seghedi et al., 1995). The large amounts of hornblende and biotite derive from the degradation of the pyroxenes from the deeper geological strata. The lack of pyroxenes in the XRD analysis proves that the mineral particles from ARSG, ARSS, and AMS belong to the outer most geological layer only.

However some of the recent studies found interesting associations of feldspar and clay minerals (e.g. muscovite, biotite, montmorillonite) in various soil type (Damian et al., 2010, Brasovan et al., 2011). The usually soils presents organic binder such humus which gives consistency to the soil (Damian et al., 2010). The soil presents an enhanced resistance at water erosion than without organic binder case found for entantrosols containing also feldspar and biotite (Brasovan et al., 2011). The particulate matter found on the St. Ana Lake shore is very similar to the second one in consistency. These particles lack of organic binder conducts to a high probability to be moved along with water streams from the rainfall directly to the lake. This probability increases with decreasing of particle size. The ARSG is less affected from this possibility than small fractions in ARSS.

The river water streams erode the shore and create deposits depending on various parameters including particle size (Radoane et al., 2006). In our case the water streams during the rainfall carry the

particle into the lake where the greater ones are subjected to an immediately sedimentation meanwhile the smaller ones remains in suspension at different water strata as observed in our previous study (Campean et al., 2009; Campean et al., 2011).

The St. Ana Lake particular supply conditions, exclusive rainfall, conducts to a particular sedimentation process, previously mentioned. The question is that all particles go down to the sediment layer or there is a possibility to remain suspended in the water strata. This is the former quest of the present research.

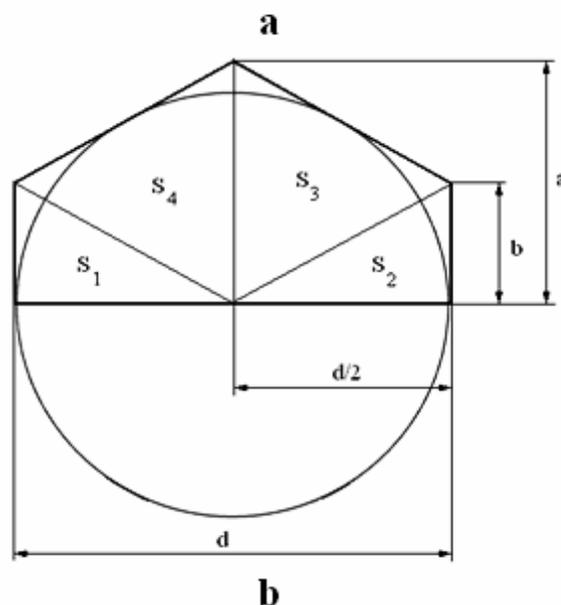
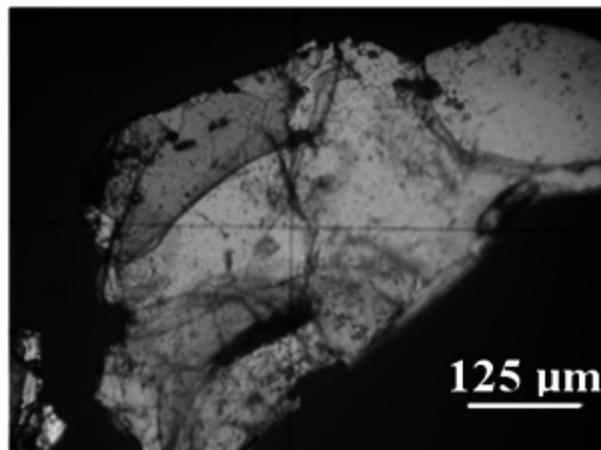


Figure 4. Design of the tabular geometrical model: a) shape of the single biotite lamella and b) decomposing of lamella into elementary geometrical shapes, figure (a).

Considering the mineral identified in ARSG, ARSS and AMS, we could approximate their shape in two categories according to their crystal habitus, spherical approximation: feldspar and hydrated silicate; and tabular approximation for biotite, muscovite, and hornblende.

Based on the biotite particle featured in figure 4a we draw the particle contour in order to achieve the proper geometrical model. We observe in figure 4b the resulted contours for the most representative biotite particle. All resulted contours features a half of regular hexagon, aspect related to the pseudo-hexagonal – tabular habit.

Considering the geometrical shape revealed in figure 4a such as half of regular hexagon results the model presented in figure 4b. There are described the tabular pseudo-hexagonal geometrical assumption of particle and the circle inscribed to the hexagon meaning the model for spherical assumption. For both cases involves the mentioned relations valuable for the given condition.

The notation in the algorithm, figure 5, are the following physical values: d – average particle size obtained by microphotography analysis, S – tabular surface, a – hexagon side; G – gravitational force – weight, m – particle mass, g – gravitational acceleration, ρ – particle density, and V – particle volume; F_a – ascension force, G_{water} – disclosed water weight, ρ_{water} – water density.

In static water regime such the closed aquatic environment of St. Ana Lake, the sedimentation is a matter of time for the mineral particles in general and biotite particularly due to the water viscosity related to the particles weight. This situation is evident for macroscopic particles. On the microscopic particles could appear some other forces such colloidal interaction forces which could be greater than particle weight and combined with ascension force lead to particle stacking in some intermediary water strata.

It is interesting to observe the particle weight and ascension force variation with the particles dimension. To fulfill this purpose we elaborate an algorithm for the previous discussed gravimetric model, presented in figure 5. This algorithm needs as input data the maximum value of particle diameter, d , as could result from a microphotography, and the minimum value for particle diameter d_0 . The preset decrement for the considered diameter, x , is set to be an input variable. It will decrease the initial d with value x given in μm for each iteration until the stop condition ($d = d_0$) is obtained. The resulted values are direction to a registering device which could be a digital registering system.

The formulation of the algorithm is suitable for several program media such turbo Pascal, C++ and many others. The results will be plotted in force versus diameter plots, which are very useful for the establishing of sedimentation regime.

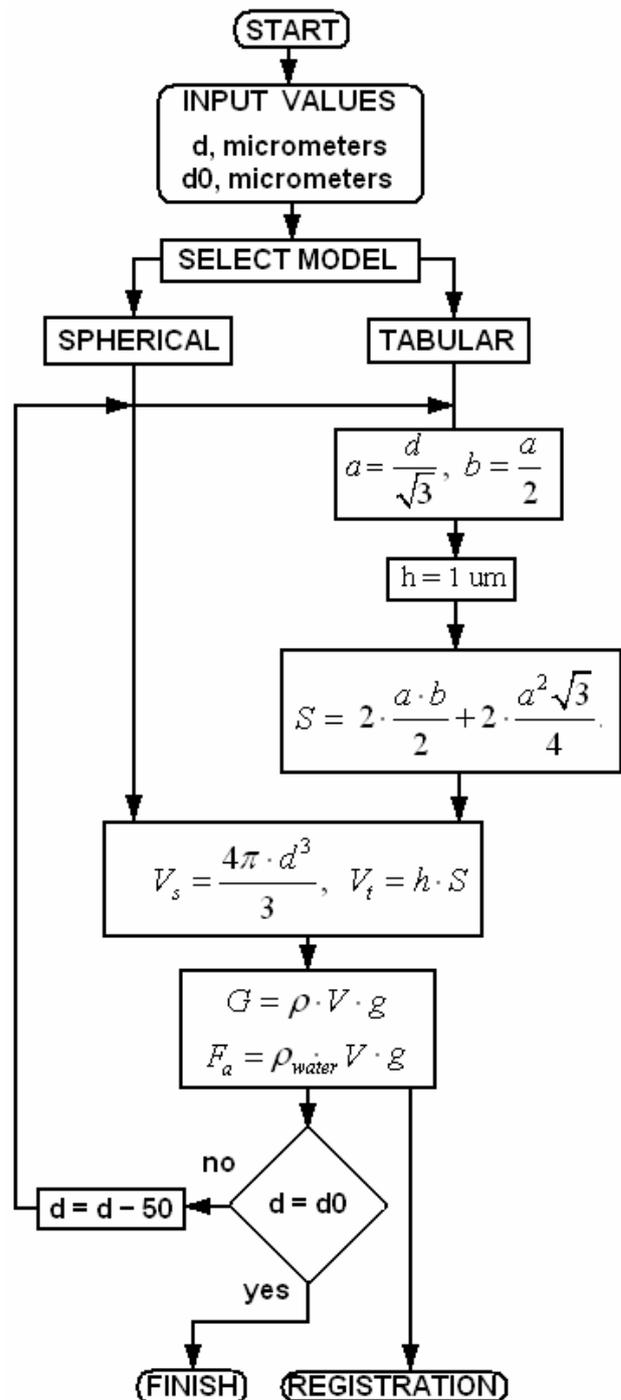


Figure 5. Algorithm for spherical and tabular sedimentation model.

At the first iteration, for $d = 500 \mu\text{m}$ we observe that the particle weight obtained by spherical model results in the range of a few Newton meanwhile for the tabular model results in the range of few mN. It means that the spherical model is not suitable for biotite lamella, the tabular model prove to be suitable for the followed purpose. We refer further only to the tabular model iterations which describe in real terms the force distribution for biotite particles.

The same iteration with the input values: $d = 500 \mu\text{m}$ and final value $d_0 = 5 \mu\text{m}$, respectively the decrement $x = 5 \mu\text{m}$ results the plot in figure 6a.

Considering the particle weight variation line as reference the resultant position indicate the direction of particle movement, if resultant is situated over the weight variation line the particle will float, and if the resultant is under the considered reference the particle will sink. The first observation on the plot in figure 6a is the great difference between particles weight and ascension force and the resultant force is situated under the weight variation. This is due to the great difference between biotite and water density. Results show the particles are exposed to a faster sinking, in the range of $150 - 500 \mu\text{m}$, the sedimentation time depending only by the distance from the top to the bottom of water, due to the exhaustive action resultant force situated under the considered reference.

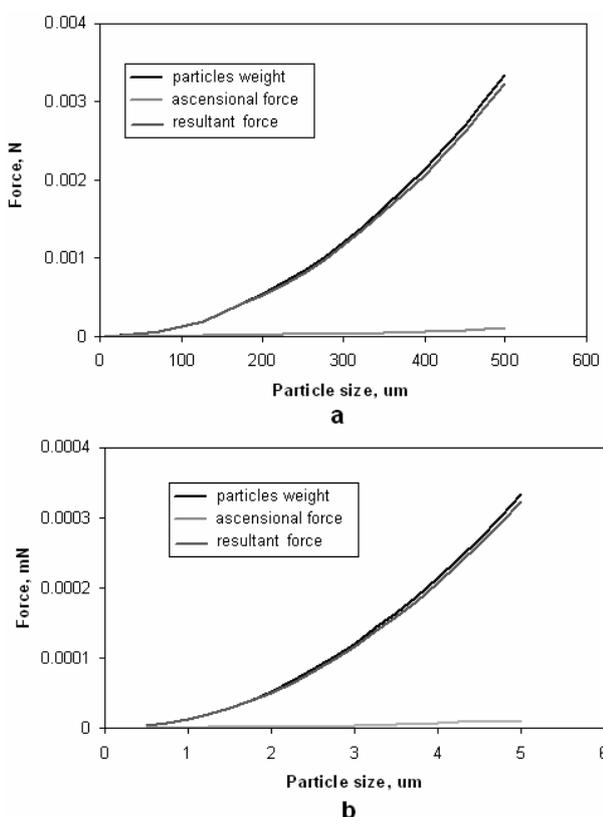


Fig.6. Particles weight, ascension force and the resultant force versus particles diameter for:
a) large micro-scale and b) small micro – scale.

For the range between $5 - 150 \mu\text{m}$ we observe, Figure 6a, very weak ascension force which almost do not influence the sedimentation resultant force, the resultant overlaying the considered reference. This situation need a new iteration with input parameters: $d = 5 \mu\text{m}$ and final value $d_0 = 0.5$

μm , respectively the decrement $x = 0.5 \mu\text{m}$, resulting a more detailed plot in figure 6b.

The forces variation in figure 6b is very similar to the previous one in figure 6a, but according to the tabular model the effective force values are situated in the range of few μN which are considerably weak to speak about eventually sedimentation due to their action, even that the resultant is situated beneath the reference. The resulted values for the gravity forces proves that the involved surface forces, respectively surface tension, are the driving forces which controls the particles movement at this size.

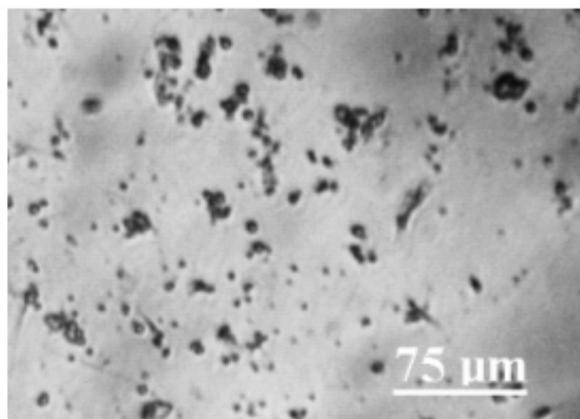
The surface activity in liquids increases for intrusion bodies exhausting a large specific surface related to small dimensions forming several types of stable suspensions such are colloids (Voegelin & Kretzschmar 2002, Jantschi & Bolboaca 2003). The involved forces involved due to the large contact surface between particles and immersion liquid are greater than gravitational forces established by our model. This evidence leads to the only one possibility which satisfy all observed conditions, the lower micro-scaled biotite particles ($\leq 5 \mu\text{m}$) remains suspended in water due to the surface activity related to their flake shape.

Although it was reported an average diameter of 60 nm for a stabile opallinus clay colloid featuring quartz mixed with illite fine particles (Voegelin & Kretzschmar 2002) results that the average dimension must to be related to the particle shape. So the pseudo-hexagonal tabular shape of biotite particles featuring relatively large planar pedion faces which in relation with their small thickness presents a favorable disposing for the particle sustentation due to the water contact surface at higher values of d such the range of $1 - 5 \mu\text{m}$.

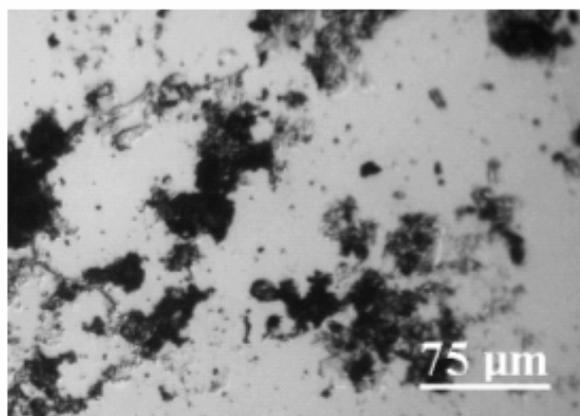
The results obtained by iteration on the tabular model are in great agreement with the suspensions observed in middle water strata of St. Ana Lake. In Figure 7 are presented the microphotographs of an average water sample collected from 2, 3, and 4 meter depth. The water drops were deposited on optical microscopic glass slides and dried at room temperature.

The water samples collected from surface of the lake and 1 meter depth presents no micro scaled particles at optical microscopic inspection performed in transmitted light. Some of our study concerning nano-dispersion in the water strata mentioned before evidence nanoparticles under 100 nm (Campean et al., 2009). Furthermore, the micro-dispersion appears at 2 meter depth as observed in figure 7a. The average particle size is situated around of $6 \mu\text{m}$

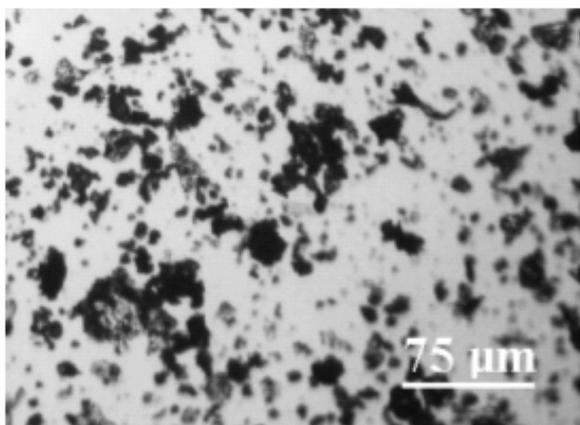
corresponding to a particle number in observed area of 18 %.



a



b



c

Figure 7. Optical transmitted light microphotographs of the glass slides with water drops collected from: a) 2m, b) 3 m, and c) 4m depth.

The microscopically suspension are more enhanced at 3 meter depth as appear in Figure 7b. There appear some small particles grouped in some larger associations. The average particle size is situated around 12 μm . The particles number in observed area increases at 30 %.

The micro particles are denser and features greater diameters at 4 meter depth due to the proximity of the mud layer from the bottom of the lake. The microstructure in Figure 7c reveal an average diameter of 17 μm related to a particle number in observed area of 55 %. We notice also the presence of some of those finest particles associated together in the nest formations having around of 50 μm .

The obtained values for particle in suspension at different water level agree the results from the tabular model. The dispersed particles observed for 2 and 3 m depth are included in small weight force according to the situation in Figure 6b. The effective weight of those particles are lower than superficial, colloidal forces involved at solid water interface according to similar observation in literature (Voegelin & Kretzschmar, 2002; Jantschi & Bolboaca, 2003). The particle micro-dispersion from 4 meter depth features some of greater particles mainly because of the mud layer proximity rather than to colloidal forces.

All the observed distribution of mineral particles suspended in water strata of St. Ana Lake are in great concordance with the data obtained by designed tabular model and affects directly the physical and chemical properties of different water strata. The results obtained by the performed measurements of water properties are presented in Table 1. All water samples were collected and processed in a similar manner as it was used for analysis in our previous researches (Campean et al., 2010, Campean et al., 2011). We observe a very good concordance with previous achieved values.

Table 1. The measured water properties

Water depth, m	0	1	2	3	4
properties of solid particles suspended in water					
Average diameter, μm	0	0	6	12	17
Part. number in area, %	0	0	18	30	55
water properties					
Turbidity, NTU	2.14	2.49	3.00	3.52	26.2
Electrical cond., $\mu\text{Si}/\text{cm}^2$	15.4	16.9	20.1	22.7	29.2
pH	6.79	6.50	6.49	6.05	5.78
Fe content, ppb	0.25	0.57	0.77	2.12	7.01

The particulate matter found in water suspension is the main cause which affects the turbidity (Sorocovschi, 2002; Dumitran, 2002; and Serban & Batinas, 2005). Considering this assumption as a mandatory parameter for the model validation we measure the turbidity at several water levels.

The given values in table 1 are the average resulted from at least 5 readings performed on the collected water sample. We observe for 0 and 1 meter depth a very low turbidity which increases significantly from 2 and 3 meter depth. The turbidity increasing with water depth confirms the previously observations. This is due to the observed mineral particles in suspension due to the light beam scattering at microscopic level in the turbidimeter investigation recipient. However the obtained values correspond to a very low turbidity according to the international standards (EN ISO 7027: 2000). Values situated over 40 NTU are considered as the limit from clear and dirty water.

The turbidity resulted at 4 m depth is significantly increased that at 3 meters. The value (26.2 NTU) is situated close to the dirty water. This is a result of the mud layer influence according to the observation for microstructure in Figure 7c.

Usually, the electrical conductivity of water is affected by the total dissolved salts (TDS) (Serban & Batinas, 2005; Campean et al., 2010). Theoretically, the water in St. Ana Lake is rain water and the electrical conductivity of water strata must to be similar. The measurements performed in laboratory reveals values close to the rain water for the surface water and 1 meter depth. The electrical conductivity feature significant increasing beginning with 2 meter depth.

It is a strange situation because the minerals in suspension at mentioned water level are not water soluble. Some of the recent research reveals that the clay minerals are able to release ions in the water presence for prolonged times (Tateo et al., 2006; Tateo & Summa 2007; Odillon Kikouama et al., 2009). Considering this assumption as eligible, the presence of muscovite and biotite in suspension from 2 m and below in the St. Ana Lake water explains the progressive increasing of electrical conductivity with the depth, respectively with the number of particles in suspension.

The pH features a slightly acidic range. The acid tendency increases progressively with water depth. The micro particles being suspended in different water strata are volcanic ones as complex silicates which presents an acidic tendency (e.g. potassium feldspar, clays, hydrated potassium silicate, hornblende, etc.). The pH variation supports the observed particles distributions which are in a good agreement with the data provided by the designed tabular model.

The iron content of the different water strata it is another important parameter related to the particles being in suspension. The biotite chemical formula is $\text{KFeMg}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$, respectively the

hornblende $\text{Ca}_2(\text{Fe}_2,\text{Mg})_4\text{Al}(\text{Si}_7\text{Al})\text{O}_{22}(\text{OH},\text{F})_2$. These two minerals have significant amount of iron placed as intermediary connector between pseudo-hexagonal layers of silicon tetrahedrons. The particles suspended in water for long times are able to release ions due to the water penetration between the pseudo-hexagonal layers. Our performed measurements evidenced the increasing of Fe amount with water depth, and with the amount of particle in suspension respectively. The greater value of Fe content, 7.01 ppb, was found for 4 meter depth in the close proximity of mud layer. The resulted iron content values correlated with pH of each water strata are in good agreement with the data in literature (Tateo & Summa 2007, Odillon Kikouama et al., 2009).

Finally, it results that the ARSS smallest mineral particles are responsible for the variation of water properties because of their presence as resident suspension at different water levels due to the superficial interactions at water particle interface. The evidenced distribution of particles fits the data achieved by tabular model instead of spherical model. Both aspects correspond to an almost static state of the lake specific to the nice weather. The strong rainfalls causes rush floods which carry large amounts of ARSG and ARSS into the water in completely disordered manner. It takes some time periods to re establish the evidenced distribution of particles.

4. CONCLUSIONS

The shore sand contains a wide range of mineral particulate matter containing andesite with: potassic feldspar, biotite, hornblende, muscovite, hydrated potassium silicate. The dimensional measurement reveals very fine fraction of minerals having an average diameter of 1 – 10 μm mixed with some of larger micro scaled particles (e.g. 100 - 300 μm average diameter) to the larger particles such gravel having an average diameter of 5 mm. The greater particles are subjected to an almost sedimentation in contact with water. The smaller particles act somehow different. We design an algorithm based on the spherical and tabular approach of mineral shape to establish a function of weight and the ascension force depending on the measured average diameter of particles. The model reveals that the tabular minerals with measured average dimension of 10 μm and beneath are subjected to the colloidal and superficial interactions remaining suspended in different water strata, meanwhile the spherical ones needs submicron dimensions to remain suspended. The measurement

performed at several water strata reveals the increasing of average diameter and number of suspended explaining the variation of water properties. The suspended particle diameter and their number increase progressively with the water depth. The electrical conductivity, turbidity increases along with water depth mainly in the proximity of mud layer. The suspended biotite and hornblende presence in the water strata causes an increasing of Fe content along with water depth. Also the presence of feldspar and hydrated silicates causes a decreasing of pH value along the water depth. The performed measurements fit the designed tabular model and correspond to an almost static water regime of the lake specific to the nice weather.

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