CALCIUM AND MAGNESIUM CONTENT IN BRIER (*Rosa canina* L.) FRUITS AT THE "CAMPUL LUI NEAG" STERILE COAL DUMP (HUNEDOARA COUNTY, ROMANIA)

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Abstract. The present study is focused on the close relation between Ca and Mg content in the brier fruits with the soil of the Campul lui Neag sterile coal dump, which is in early formation processes from industrial entiantrosoil to a fertile soil. The minerals found in soil sample are: quartz, calcite, biotite, and potassium feldspar. Some of the soil particles are chemical inert (quartz) but calcite provide Ca^{2+} ions and biotite provides Mg^{2+} ions. The brier roots assimilate those ions despite of lack of humus of entiantrosoil. This interaction between brier roots and entiantrosoil facilitate on the long term the formation of a fertile soil. Chemical analysis of brier fruits feature Ca and Mg content in the reference range (Ca = 0.297 g Ca/100 g solid sample ± 5 %, and Mg = 0.212 g Mg/100 g solid sample ± 5 %). The calculated Ca extraction coefficient is 20% and Mg extraction coefficient is 16 % for the brier fruits growth on the dump soil. The available Ca amount is directly proportional to the calcite content, which is significantly increased comparative with biotite. The less amount of biotite conducts to a less extraction coefficient for Mg. This proves that dump soil is able to assure an optimal amount of Ca and Mg for the brier fruits growing. The lack of heavy metals in the dump site is a favorable assumption for human use of brier fruits. The brier wild crop growth on the sterile dump soil is suitable for home application (brier marmalade, depurative tea, etc…). The sterile dump soil rehabilitation prove to be a slowly process counting only on the brier interaction with mineral components. The actual state feature a good progress of dump rehabilitation. The process could be fastened by improving some natural fertilizing procedures. Finally a professional crop for industrial applications could be growth on the sterile dump site only with a proper soil fertilization.

Keywords: *Rosa canina*, calcium, magnesium, coal dump rehabilitation.

INTRODUCTION

Brier (*Rosa canina*) is a native rose species spread in all Europe, northwestern Africa and western Asia. It grows on various land types nearby roads and forests. It is a shrub having 1-3 m high, bearing thorns with tip curved sickle-shaped bottom and strong roots developed underground. It is well known that brier grows up even in less fertile soil, fact used for entiantrosoils stabilization against earth sliding [6, 25]. The “Campul lui Neag” sterile dump is a typical entiantrosoil deposit formed up due to the coal mining operation in the area [6, 4]. The coal sterile usually contains silicate particles such as quartz sand, feldspars, marls and clays. Such formations are reported for “Campul lui Neag” sterile dump belonging to the Eggerian lower age [4]. This type of soils presents a relative low ammount of nutrients but exceeds the mineral oligoelements like calcium and magnesium. In this case, a significant variation it is expected in the calcium and magnesium content of briar fruits.

The brier blooms in June having pink flowers grouped 2-3 on the top branches. At maturity the receptacle forms a false fruit containing the seeds which become red later in August and September [26, 29]. The briar fruit are harvested in various stages of maturity. The harvesting is recommended when false fruit becomes red which means that a highest level of C vitamin is achieved (around of 5 % [29]). It is recommended for medicinal use due to the rich amount of C vitamin and antioxidants. The therapeutically benefits are: a general detoxification of tissues, reduces anemia effects, and reduces the intestinal worms proliferation [8, 21, 22, 25].

Brier fruits also contains pectin, tannins, carotenoids, flavones derivatives [24, 28], fatty acids (palmitic, stearic, oleic, linoleic and linolenic) which were used in the cosmetic industry [7, 31]. They are a rich source of proteins, starch, vitamin E, sterols, and minerals and therefore are used in pharmaceutical and food industry for several specific purposes: juice, marmalade, tea, syrups, and alcoholic beverages after fermentation [30, 34].

There is a very close relation between fruits content and the soil where the plant grows. The brier bushes on the “Campul lui Neag” sterile dump have an environmental role to protect against particle spreading and earth sliding. Few questions appear in this situation: is the sterile dump a proper soil for development of proper brier fruits, respectively did the brier fruits have enough calcium and magnesium for specific purposes. The adequate answer at risen questions could be obtained only by an enhanced soil analysis related to the determination of calcium and magnesium in collected brier fruits from the “Campul lui Neag” sterile dump.

MATERIALS AND METHODS

The brier fruits were collected in September from the Câmpul lui Neag coal sterile dump (Petroşani Basin, Hunedoara County, Romania), they were preserved according to the standard procedures. The fruits were collected manually without leafs and branches and were mixed together for a homogeneous distribution. The crop was stored and naturally dried in a thin layer on wood shelves in a dark and cool place (average temperature 15 °C and atmospheric pressure).

There were followed three series of average representative fruit samples. An average fruit sample contains fruit harvested from all area of steril dump, at least 50 different brier shrubs (bushes). The averaging of the sample ranges covers at least 150 individual samples to gain an average characteristic of brier fruits harvested on Camplul Lui Neag sterile dump.

The calcium content was measured using a murexid indicator and magnesium content was determined with black eriocrom T by titration with complexon according to the standard titrimetric methods [9]. For a more precise result the calcium and magnesium content was also determined by atomic absorption spectrometry using an AA 6300 Shimadzu AAS spectrometer.

The mineralization of each average representative brier fruit samples were performed in a Berghof WMS 2 furnace using 3 power steps: 10 minutes at 145° C; 10 minutes at 160° C and 20 minutes at 190°C. The white ash resulted was treated with HNO_3 1 % and further bring to the quota of 10 ml with deionized water.

AAS measurements for Ca determination was performed using a Ca HCL lamp having the absorbtion maximum at 422.7 nm, the current intensity was 10 mA. The baseline compensation was performed with deuterium lamp and compensation gas used was air/acetylene mixture.

AAS measurement for Mg determination was performed using a Mg HCL lamp having the absorption maximum at 485.2 nm, the current intensity was 8 mA. The baseline compensation was performed with deuterium lamp and compensation gas used was air/acetylene mixture. All AAS procedures was performed according to EN 1134:1994 standard prescriptions. There AAS measurements for Ca and Mg content were performed on 3 average representative samples, final results are the average of this determinations.

Soil samples were collected from several points of the Câmpul lui Neag coal sterile dump which were mixed together into a representative soil sample. The mineralogical composition of soil sample could be revealed only by X-ray diffraction due to the similitude between X-ray wavelength and the specific interplanar distances of different minerals [1, 2].

The soil sample was deposed on a thin amorphous layer on the X-ray diffraction specimen holder. The mineralogical composition was determined by X-ray diffraction (XRD) using a DRON 3 diffractometer equipped with data acquisition module and Matmec VI.0 soft. The X-ray patterns were obtained with a Co K_α monochrome radiation [2, 27]. The resulted X-ray diffraction peaks were identified by comparing with standard database Match 1.0 from Crystal Impact Corporation.

Soil particle morphology was investigated with an optical mineralogical microscope Laboval 2 produced by Carl Zeiss Jena equipped with a Samsung 8 MPx digital capture. The optical microscopy inspection was performed in transmitted light in order to reveal the particle shape and size and in cross polarized light as a complementary mineralogical observation.

The elemental soil analysis was performed according to the standard sampling and operating procedures using a Rigaku ZSX100 X-ray fluorescence spectrometer (XRF) in order to measure the main elements corresponding to the minerals identified by XRD. There was used a WDXRF wavelength detector for a wide range of atomic species from beryllium to uranium having a resolution of ppb range for various sample type such: solid samples or solutions. The determinations are processed automatically by the device soft using an integrated international Dyna Match database for XRF studies respecting the quality standards prescribed by EN ISO 9001:2000 standard.

RESULTS

In Figure 1 is presented the calcium content resulted for brier fruit samples series 1, 2, and 3, related to the reference values. The reference values varies from different research Ercsilli [11] point out an amount of 0.196 g Ca/100g solid sample and Kazaz [18] presents a standard value of 0.388 g Ca/100g.

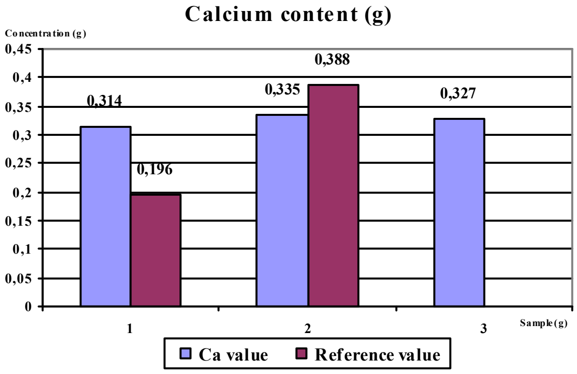


Figure 1. Calcium content in brier fruits.

According to the articles studied we consider a reference range for the calcium content from 0.196 to 0.388 g Ca/100g solid sample. We observe in Figure 1 that all results are inside the reference range, proving that the briar fruits are suitable for topic applications. The result beneath the reference range means that the respective briar fruits are not suitable for topic application and should be rejected. The results beneath the standard range conducts to certain dosage modification in the technological flux of topic applications.

The average calcium content resulted by titrimetric method for all 3 average representative samples is situated around the value of 0.325 g Ca/100g solid sample ± 11 %. Assuming the gross errors which could occur by titrimetric method we observe that the obtained value fits the reference range.

The atomic absorption analysis shows Ca content of 0.297 g Ca/100g solid sample ± 5 %. The result obtained by AAS fits the considered reference range.

The value is in good agreement with titrimetric method proving a high calcium content.

Similar issues are observed for the magnesium content, Figure2. The lower limit of the reference range of magnesium content is set by Ercsilli [11] at 0.114 g Mg/100g solid sample and the upper limit is situated at 0.217 g Mg/100g solid sample Kazaz [18].

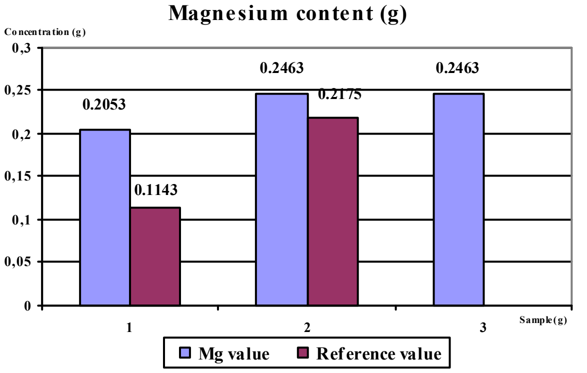


Figure 2. Magnesium content in brier fruits.

In Figure 2 we observe that the magnesium content for sample series 1 is in the reference range featuring 0.205 g Mg/100g solid sample and the series 2 and 3 are situated slightly above the reference range having 0.246 g Mg/100g solid sample, resulted average is 0.232 g Mg/100g ± 6 %. The atomic absorption analysis shows that the Mg content is situated in the reference range having the value of 0.212 g Mg/100g solid sample ± 5 %. The average value obtained by AAS spectrometry is very close to the one reported by Ercsilli [11].

The results obtained by AAS spectrometry are in good agreement with those obtained by titrimetric method for both determination of Ca and Mg, but they are more precise. For this former purpose, we will refer further at discussion only to the values obtained by AAS.

Calcium and magnesium from brier fruits is assimilated from the coal sterile dump. In this situation it is important to perform an enhanced soil investigation. The representative soil sample was investigated by X-ray diffraction, the resulted pattern is presented in Figure 3.

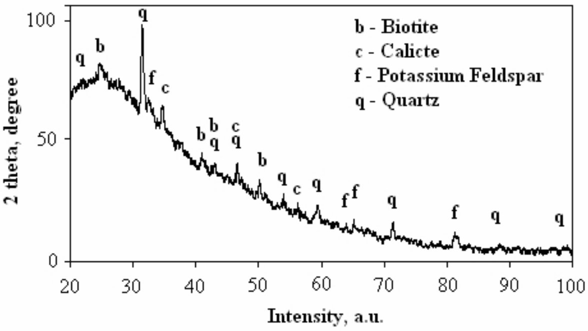
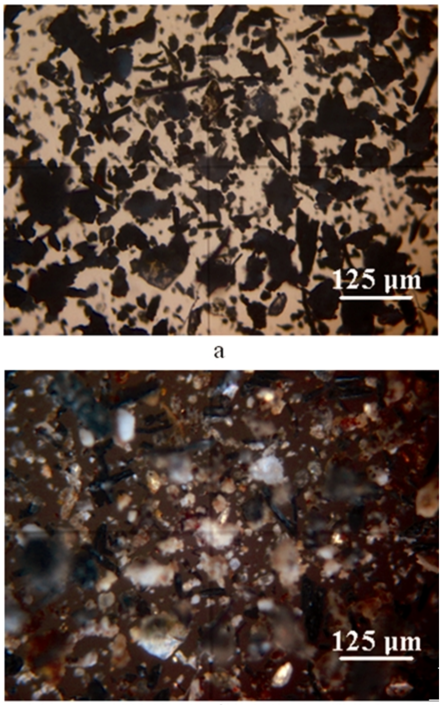


Figure. 3. X-ray diffraction pattern for soil sample, Co k_α radiation.

Following minerals: quartz – SiO_2: hexagonal crystallization [13]; calcite – $CaCO_3$: rhombohedral crystallization [15]; potassium feldspar – $K(AlSi_3O_8)$: crystallization in monoclinic system [35]; biotite - $H_4K_2Mg_6Al_2Si_6O_{24}$, crystallization in monoclinic system [20] were found in the soil sample.

Quartz and calcite are the most representative minerals in the dump soil sample due to their well developed diffraction peaks which corresponds to larger micro scaled particles. The soil particle shape could be observed in Fig. 4a. There are some rounded grains featuring an average diameter of 50 µm which corresponds to quartz observed in diffraction spectrum. It appears in cross polarized light in a light green – gray, Fig. 4b. Calcite particles presents an equiaxial shape having an average diameter of 30 µm as observed in Fig. 4a, which corresponds to the yellow particles in cross polarized light.

Biotite and potassium feldspar feature broader and less intense diffraction peaks corresponding to a more refined particle structure. In figure 4a are also observed some tabular particles with a planar average size of 20 µm with a reddish brown appearance in cross polarized light, which corresponds to the biotite. Feldspar is also well represented by some bright white particles featured in cross polarized light, having an average size around 25 µm.



a

b

Figure. 4. Optical microphotographs for the soil sample: a) transmitted light; b) cross polarized light.

The mineralogical investigation prove that the dump soil is granulated having only natural and non toxic mineral compounds. There also is observed the lack of humus binder characteristic for a common fertile soil. The chemical elements absorbed by brier roots are ions resulted from the interaction of identified minerals (mainly calcite and biotite which are sensitive to relative humidity).

In order to find out the available ions for brier growth we performed an elemental XRF analysis, results are presented in Table 1.

Table 1. XRF elemental analysis results.

Element	Si^{4+}	Al^{3+}	K^{+}	Ca^{2+}	Mg^{2+}
wt. %	57.3	16,3	2.37	1.50	1.36

The Si^{4+} ions are over the half of sample content because of their proliferation in quartz, biotite and feldspar as basal structure unit. The most of Si^{4+} are immobilized in stronger bonds of crystal structure of identified minerals as well as Al^{3+}. The mobile ions in relative humidity are K^{+}, and Mg^{2+} due to their position as interplanar bonds of phylosilicate such biotite and potassium feldspar [2]. The Ca^{2+} amount results by partial decomposition of calcite in presence of the humidity at the proximal contact with brier roots.

DISCUSSIONS

The minerals found in the dump soil sample are subjected to an intimate contact with brier roots, which provides the main oligoelements necessary for shrub growth. Some of them could be chemical inactive or could be active in presence of water. Quartz is the main mineral found in the soil sample. As observed before the hexagonal crystallization of quartz assure a very compact and resistant structure without cleavage. Featured properties of quartz particles prove that it is chemical inert, in this case it acts as a neutral component.

Calcite is a typical mineral for sedimentary soils [17, 19]. Calcite particles, even bigger formations, are affected by water presence which could release Ca^{2+} ions in aqueous solution similar to the process involved at cave formations. Brier roots could easily assimilate Ca^{2+} ions from dump soil in presence of relative humidity due to larger amount of calcite particles found.

Biotite is a magnesium clay mineral which belongs to the phyllosilicate crystal class, having a monoclinic crystallization. The biotite structure features hexagonal crystal planes of SiO_2 tetrahedrons (which have a very high mechanical strength) bonded in multi – layers by Mg^{2+}, Al^{3+}, and K^{+} ions which trapped free valences of SiO_2 tetrahedrons. The mechanical strength is weaker between the layers than inside of them. The inter – layer distance allows water to penetrate inside of biotite crystal and consequently to release some of the bonding ions. The ions releasing in aqueous solution was observed in several research concerning clay minerals [23, 33, 12]. The released ions amount is favored by presence of small micro – scaled particles [3, 5]. The biotite observed in the investigated dump soil feature a good refinement of the particles, average diameter of 20 μm, enough to release an significant amount of Mg^{2+} ions in the water presence. Similar, the amount of potassium feldspar represents an important source of K^{+} ions.

The usual fertile soil have large amounts of humus which stabilize oligoelements such Ca and Mg as humic acid salts insoluble in water. Furthermore this colloidal suspension is absorbed by plants roots due to the cellular osmotic pressure [11, 10, 32]. The dump soil is characterized by an acute lack of organic material and humus due to the industrial pedogenesis. Furthermore the available minerals are released from parent minerals directly on the water present in dump soil. The brier roots absorb this mineralized water in the feeding circuit. A strong interconnection appear between the Ca and Mg content in the soil samples and the amount in the brier fruits.

The results of XRF elemental analysis are in good agreement with XRD observation. All identified ions belong to the minerals identified by XRD. In this particular case we could calculate the Ca and Mg amount per solid soil sample as follows:

$$\begin{cases} Ca_{sol} = 1.50 \;\; gr/100gr\ sol \\ Mg_{sol} = 1.36 \;\; gr/100gr\ sol \end{cases} \qquad (1)$$

This allows further to calculate the brier fruit extraction coefficient, R, of Ca and Mg according to the relations (2) and (3):

$$R_{Ca} = 100 - \frac{Ca_{soil} - Ca_{brier}}{Ca_{soil}} \cdot 100 \ [\%] \qquad (2)$$

$$R_{Mg} = 100 - \frac{Mg_{soil} - Mg_{brier}}{Mg_{soil}} \cdot 100 \ [\%] \qquad (3)$$

Finally results that the Ca extraction coefficient is 20 % and Mg extraction coefficient is 16 % for the brier fruits collected from the shrubs growth on the dump soil. The resulted extraction coefficients agree the mineralogical observations. Available Ca amount is directly proportional to the calcite content, which is significantly increased comparative with biotite. The less amount of biotite conducts to a less extraction coefficient for Mg. This proves that dump soil have enough mineral resources to assure an optimal amount of Ca and Mg for the brier fruits to be situated inside of the reference range. The lack of heavy metals in the dump site is a favorable assumption for human use of the brier fruits. This is a sufficient condition for a random and wild crop, suitable only for home application (brier marmalade, depurative and refreshing tea, etc.).

The evidenced interrelation of brier and dump entiantrosoil in plant feeding chain assure the success of the dump rehabilitation. The stabilization of the soil by brier shrubs roots it is an completely achieved goal, but the conversion of dump entiantrosoil to a fertile one is a slowly process in the actual condition. The process could be fastened by improving some environmental fertilization methods. Nowadays the brier fruits are suitable only for home or small and limited manufacturing production. The fertilization implementation will increase the conversion rate to a fertile soil and could facilitate the obtaining of a professional brier crop. Such crop could grow up on the dump soil only in the condition of fertilization in order to form an organic humus binder for identified mineral particles. The fertility of dump soil could be increased by several natural methods such: spreading of animal manure (pig, cow, hen) combined with a proper wetting [3, 14, 16]. This method will preserve the Ca^{2+} and Mg^{2+} in soil by the reaction with humic acid. Improving the soil fertility will assure a good brier fruits crop along with the dump rehabilitation in a proper environmental conditions.

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