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Charging Effect on Soil Particles in Scanning Electron Microscopy (SEM)

Abstract. *Scanning Electron Microscope (SEM), a tool for material characterization reveals information about surface, subsurface, composition, and defects in bulk materials. The objective of the article is to understand the parameters of soil particles on charging effect in SEM. The SEM images were obtained on colluvium soil particles by varying particle size (A=2-1mm, B=0.6-0.425mm, C=0.3-0.212mm, and D= <0.075mm), and number of conductive coatings (uncoated, single, and double). The article proposes a method for the preparation of soil particles for SEM imaging and is applicable for all types of soil particles. The study revealed that the soil particles of size greater than 212µm require double conductive coating and less than 75µm requires single coating to avoid charging effect. The sharpness of the image is questionable of soil particles greater than 212µm at 10000× magnification and above, after double conductive coatings.*
Key Words: *Scanning Electron Microscopy, Soil Particles, and Charging Effect*

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1 Introduction

The study of surface microstructure of soil particles gives insights of the sedimentary history of the soil particles (Vos et al., 2014). The applications of SEM in soil mechanics are to study grain outline: roundness or angularity, transportation mode, distance and time travelled, and size (Park et al., 2022), fabric orientation (Song et al., 2022), forensic analysis of soil and sediment traces (Jafery et al., 2022), trace of heavy metals in soil (Burdalski et al., 2022), and morphological investigations up to a scales of micrometer to nanometer (Islam et al., 2022). The wide applications of SEM imaging in soil mechanics have attracted attention among researchers. The sample preparation plays a major role in the SEM imaging to acquire high quality morphological images. The main problem in the SEM imaging is the charging effect on the sample while imaging. The image that is darker than background is called positive charging and brighter is called negative charging. The most common in soil particles imaging in SEM is negative charging.

The charging effect can be reduced by two methods, one is by conductive coating to a thickness of a few hundreds of angstroms. This method allows the sample surface with a thick coating cause not able to capture real fine surface morphology. The second one is by lowering the energy of the primary electrons. This method allows the low energy electrons on the surface of the sample results poor quality of images were recorded with less intensity of electrons (Ichinokawa et al., 1974). The charging effect is also used for the characterization of conductor-insulator composite materials. Insulating materials with thin films, thin films with mask, and overlay marks (Zhang et al., 2004). In soil mechanics, the application of charged micrographs is not used, and it will give only particle size but, not the morphology for microstructure analysis.

However, the article is not included the direction of specimen charging relevant to the beam energy interval (Grella et al., 2004), Shrinking effect (Flatabø et al., 2017), accelerating voltage (Chetana et al., 2022), working distance (Bayazid et al., 2020), and scanning time and inclination of the beam (Ichinokawa et al., 1974). The article presents the preparation of soil particles for SEM imaging and the effect of particle size, and number of conductive coatings on charging effect.

2 Materials and methods

2.1 Sample Preparation

SEM images were obtained on colluvium soil sample collected from Banihal, Jammu and Kashmir, India (Figure 1a, b). The specific gravity (G) of the soil is 2.74. The soil is classified as "clay with intermediate plasticity (CI)" with the gravel (1.16%), sand (7.46%), silt (65%), and clay (25%) (IS1498, 2007). The soil particles were prepared by soaking the oven dry (105°) soil sample of 0.5 kg for 24 hours (Figure 1c). The soaked soil sample was subjected to wet sieving through 75 microns sieve (Figure 1d). The soil sample retained on 75 microns was subjected to oven dry for 24 hours and subjected to dry sieving analysis as per Indian standard code (IS2720-4, 1985) (Figure 1e). The soil particles were collected at different sizes of sieve are 4.75, 2, 1, 0.6, 0.425, 0.3, 0.212, 0.150, 0.075 mm, and pan (Figure 1f).

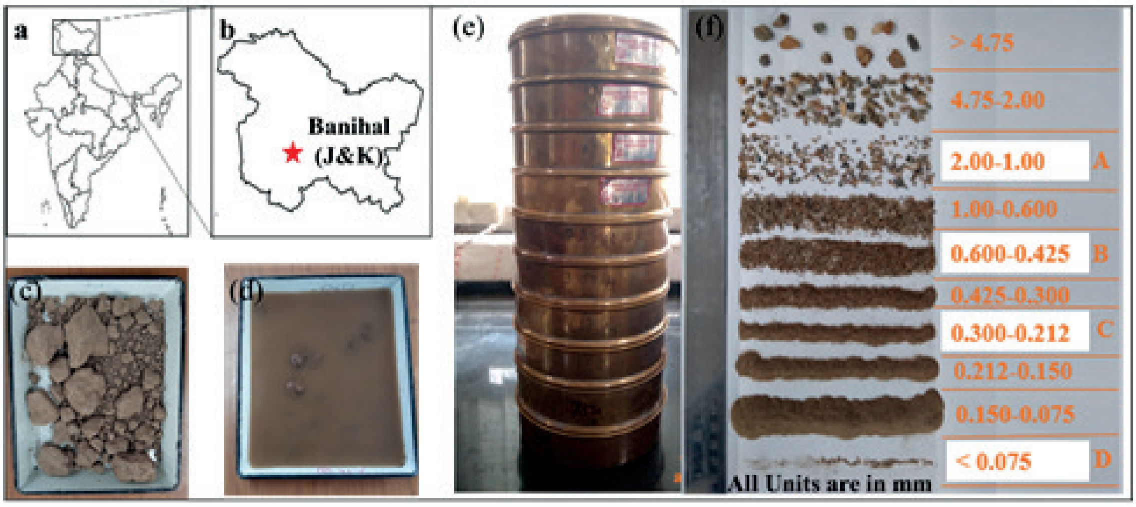


Figure 1. Colluvium soil sample, a) India, b) Location of Banihal, Jammu and Kashmir, c) Collected soil sample, d) Soaked sample for wet sieving, e) Set of sieves for dry sieving, and f) Distribution of soil particles and representation of A, B, C, and D particle size ranges.

The soil particles passing through 2 mm and retained on 1 mm are considered as particle size A and particle size B, C, and D, are of the size of 0.6-0.425 mm, 0.3-0.212 mm, and less than 0.075mm, respectively (Figure 1f). The double stick carbon tape was placed on the SEM stub and dry soil particles were placed on the carbon tape. Stiff paper was used to distribute the particles over the carbon tape and the SEM stub with soil particles was subjected to imaging.

The maximum care was taken to distribute soil particles over large scanning area and subjected to gold sputter conductive coating. After coating, the stub was placed on specimen stage and closed the chamber, subjected to vacuum. The particles of size A, B, C, and D are placed on the SEM stub, subjected to uncoated, single, and double gold sputter coating and ready for imaging.

2.2 SEM configuration

Generally, the SEM on secondary electron mode is operated to get the surface images. The ZEISS EVO50 at the Indian Institute of Technology Delhi (IITD), India was used in the present study (Figure 2). The specifications of ZEISS EVO50 are resolution: 2 nm at 30 kV, acceleration voltage: 0.2 to 30kV, magnification: 5× to 1,000,000×, field of view: 6 mm at the analytical working distance (AWD), general sample size: less than 10 mm in any direction (height or diameter), vacuum pressure: -6 millibars, and detectors: secondary electron (SE)-Everhart Thornley (ET), SE in Variable-pressure secondary electron detector (VPSE), backscattered electron detector (BSD) in all modes-quadrant semiconductor diode (Carl, 2003).

Figure 2a, the instrument consists of main unit, control system, and Personal Computer (PC). The main unit consist of vacuum chamber, specimen holder (X-Y-Z tilt rotation stage (Figure 2b), and the filament of Tungsten for the generation and acceleration of electrons. High purity Emitech/Quorum sputter coater of model K550X was used in the study (Figure 2c). The sputter emits gold (60mm diameter x 0.1mm Thick: Gold fitted as Standard) spherical particles for coating of thickness 10 ± 2 nm/minute at a vacuum gauge range of 1×10^{-4} millibars (Quorumtech, 2007). The present study taken the sputter time as 1.5 minutes and an approximate thickness of 15±2 nm surface coating.

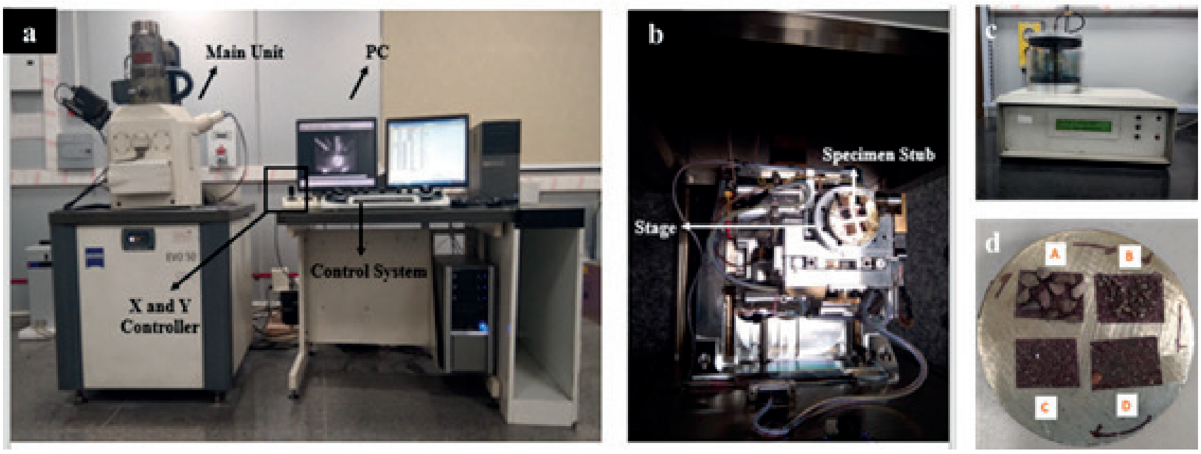


Figure 2. Scanning Electron Microscope Central Facility at IIT Delhi, a) External view of ZEISS EVO 50 SEM, b) Specimen stub assembly chamber (internal), c) Emitech/Quorum sputter gold coater of model K550X, and d) Prepared SEM stub with soil particles after conductive coating.

The prepared SEM stub (Figure 2d) placed on the stage for scanning and closed the chamber. The chamber is filled with vacuum at a pressure of -6 millibars. After successful completion of vacuum, the incident beam is on to the sample to capture the images through personal computer (PC). The images were collected at different locations of the sample by adjusting the X and Y stage knob. The captured images were exported to save the images as tag image file format (tiff).

2.3 Testing Methodology

The SEM images were obtained on soil particles of parameters considered are varying size (A, B, C, and D), and number of conductive coatings (uncoated, single, and double). All the images were taken at an accelerating voltage of 20 KV. The soil particles of size A, B, C, and D are placed on the SEM stub having four slots (Figure 2d). The stub without conductive coating was taken for imaging, after that, single and double conductive coatings were applied on the same soil particles. Throughout the imaging the soil particles are the same of A, B, C, and D but the number of conductive coatings were changed.

3 Results and Discussions

3.1 Effect of Particle Size

The soil sample consists of distribution of different sizes of particles due to the weathering. The distribution of particles tells us about the type of weathering conditions and the type of forces acted on it through morphological studies. The environment of the soil sample greatly affects the particle size distribution. The alluvium, aeoline, lacustrine, marine, and colluvium soil types exhibit different types of microstructures of particles due to their environment is different. Within one type of soil sample the distribution of soil particles sizes and their microstructure are very important for morphological studies to investigate micromechanical behavior of soil particles. The uncoated soil particles of size A, B, C, and D were subjected to SEM at a constant magnification of 65× times. From Figure 3, with the increase in particle size, charging effect is increased. The surface potential (Vs) of soil particles increases and causes charging effect (Ichinokawa et al., 1974). The charged surface areas and particles were marked on images (Figure 3).

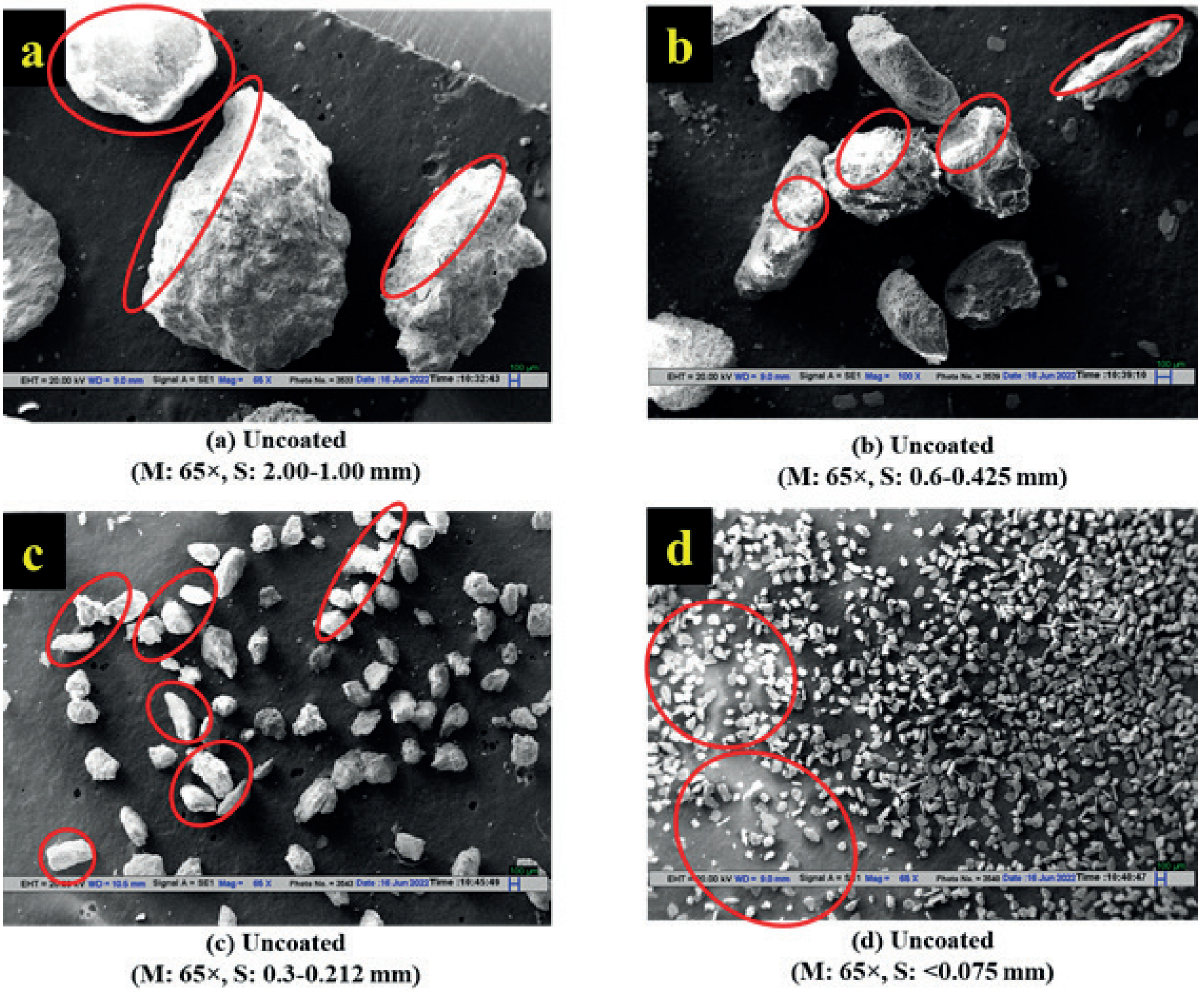
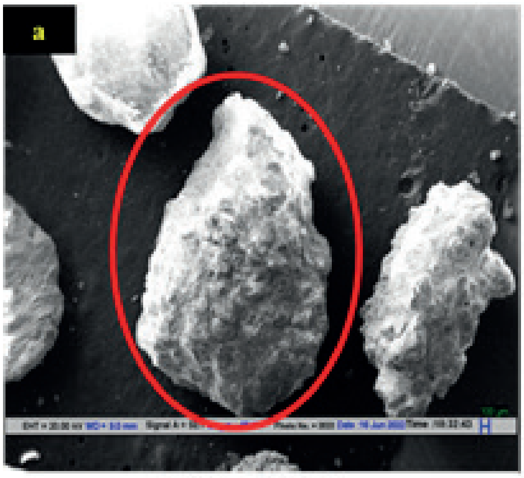


Figure 3. Uncoated soil particles at 65× times magnification, (a) Particle size 2-1 mm, (b) 0.6-0.425 mm, (c) 0.3-0.212 mm, (d) <0.075 mm.

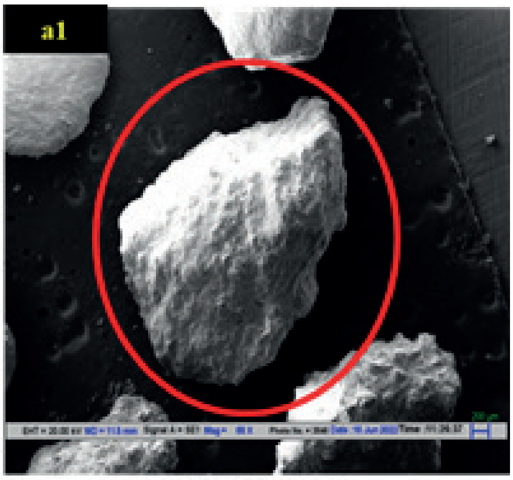
The letters M and S represent magnification and size, respectively. The particle of size 2-1mm, and 0.6-0.425mm shows the partial charging of particles (Figure 3a, b). The images are useful to investigate the boundary outline of the particles but not the microstructural features. As shown in Figure 3(c, d) the particles were charged are not used in the microstructure analysis and causes the misrepresentation of surface features. Ichinokawa et al. (1974), obtained SEM images on flat surface of Teflon sheet of uncoated and coated with gold evaporated films excepts for a strip of 200, 100, and 50μm. The conclusions are that the charging effect is more in uncoated strips (Ichinokawa et al., 1974).

3.2 Effect of Number of Conductive Coatings

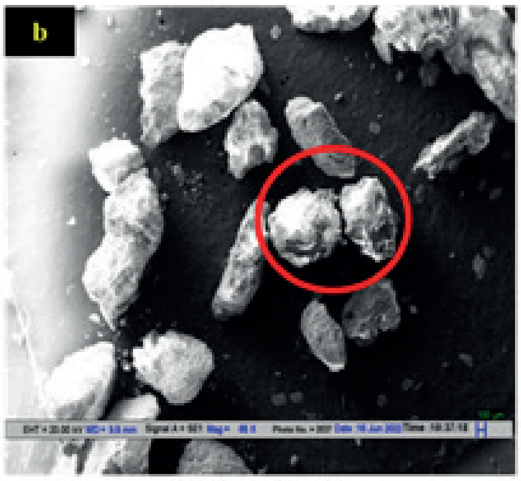
The surface potential is reduced by providing conductive material coating (gold/copper) so that the conductive coating acts as bridge between the specimen and stub (earthing to the concentrated electrons) to avoid charging. Soil particles are a mixture of mineral grains and amorphous material. Mineral grains act as conducting medium to the electrons, but the amorphous material acts as nonconductive medium causes the accumulation of electrons, resulting negative charging of soil particles. The charging effect reduced with the number of conductive coatings (Figure 3, 4).



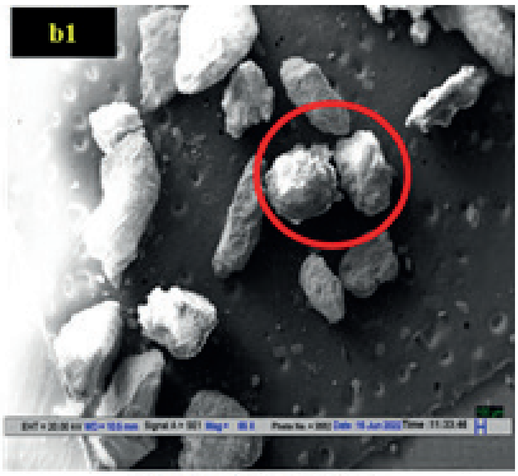
(a) Single Coating
(M: 65×, S:2.00-1.00mm)



(a1) Double Coating
(M: 65×, S:2.00 -1.00mm)



(b) Single Coating
(M: 65×, S: 0.6-0.425 mm)



(b1) Double Coating
(M: 65×, S: 0.6-0.425 mm)

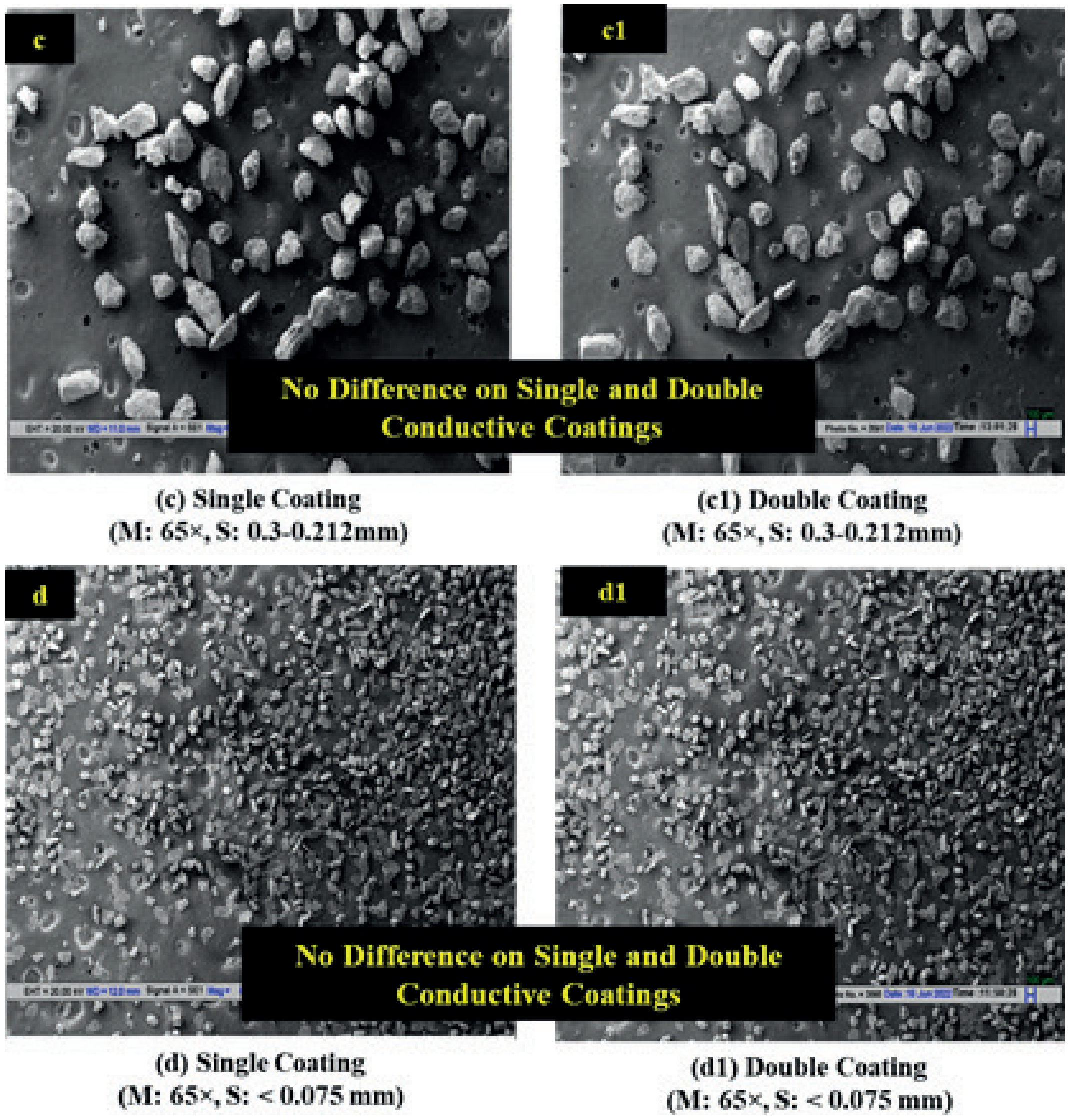


Figure 4. Effect of number of copper coatings on charging, a) Particle Size 2-1 mm, b) Particle Size 0.6-0.425 mm.

From Figure 4, the particles of size grater 0.425mm (varying from 2 to 0.425mm) exhibit charging after single conductive coating. For analysis of microstructure of soil particles greater than 0.425mm were subjected to a second conductive coating (Figure 4a, a1, b, and b1). The particles of size less than 0.425mm were not affected by single and double conductive coatings (Figure 4c, c1, d, and d1).

The image has blunt edges after single conductive coating at the magnification of 10000× times of particle size 2-1mm (Figure 5a). After the second conductive coating, the sharpness of image was achieved, and the microstructure is clearly observed at 10000× Magnification (Figure 5b). It is evident that, for shape microstructure images for a particle of size greater than 0.425mm require double conductive coating on higher magnification.

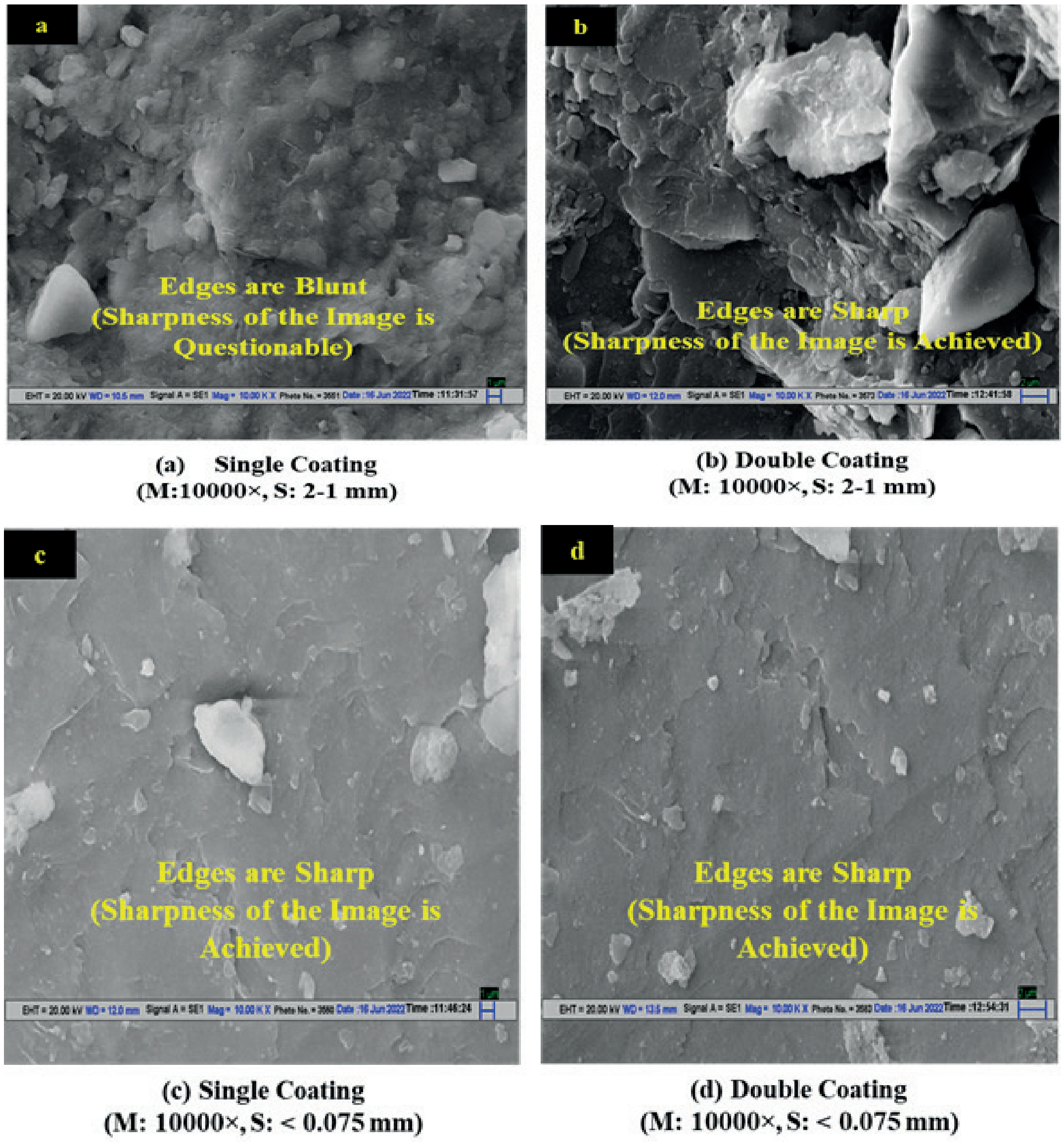


Figure 5. Microstructure of soil particles at single and double conductive coatings at 10000× Magnification, (a and b) Particle Size 2-1 mm, (c and d) Particle Size <0.075 mm.

There is not much difference between the single coating and double coating of particle size less than 0.075mm at 10000× times magnification (Figure 5c, d). The above discussion enables that, for microstructure analysis of soil particles of size greater than 0.425mm require double conductive coating and particles of size less than 0.425mm require only single conductive coating for higher magnification. For the measurement of particle size and shape does not demand the requirement of conductive coating, but for microstructure analysis conductive coating is compulsory. The conductive coating reduces the charging effect by driving the electrons to the ground (Borchert et al., 1991)

The maximum care was taken to avoid moisture and samples were kept at oven dry of 105 °C before subjecting to SEM imaging. Another reason for charging porous particles is due to the accumulation of nonconductive clay minerals in the boundaries of the micropores.

4 Conclusions

The soil particle size greater than 212 microns, go for the double coating of sample for better conducting of material. The soil particles size less than 75 microns requires single coating, if the sample has more porous particles requires double coating to avoid blunting of mineral edges. The time of incident beam on a small area of sample should be minimized and can be adopted as imaging strategy.

The charge balance is not possible on the surface of the sample, change the incident beam energy to less than 5 kV, but the sharpness of the image is questionable. The study made conclusions on analysis of colluvium soil particles SEM images and the methodology can be used for all types of soil.

The practical solution to avoid the charging effect from the instrument is to work on lowest usable or suitable magnification, to avoid small scan-square modes of operation, and to go for high resolution microscopes for more detailed study of morphology on nanoscale like field emission scanning electron microscope (FE-SEM), transmission electron microscope (TEM), and high-resolution transmission electron microscope (HR-TEM).

References

1. Vos, K., N. Vandenberghe, and J. Elsen. "Surface textural analysis of quartz grains by scanning electron microscopy (SEM): From sample preparation to environmental interpretation." Earth-Science Reviews, 2014. 128: p. 93-104.

2. Park, J.S., et al. "Investigating physical and mechanical properties of nest soils used by mud dauber wasps from a geotechnical engineering perspective." Scientific reports, 2022. 12(1): p. 2192.

3. Song, X., et al. "Elimination of the charge effect on zirconia coatings using a focused ion beam for characterization of the three-dimensional microstructure." Materials Characterization, 2022. 185: p. 111769.

4. Jafery, K.M., et al. "SEM-EDX and AFM analysis for the surface corrosion morphology structure and roughness on embedded X70 external pipeline in acidic soil (peat) environment." Materials Today: Proceedings, 2022. 48: p. 1929-1935.

5. Burdalski, R.J., et al. "Mineralogy, morphology, and reaction kinetics of ureolytic bio-cementation in the presence of seawater ions and varying soil materials." Scientific Reports, 2022. 12(1): p. 17100.

6. Islam, A., et al. "Scanning Electron Microscopic Analysis of Clays in The Soils of Lower Atrai Basin of Bangladesh." Dhaka University Journal of Biological Sciences, 2022. 31(1): p. 105-115.

7. Zhang, H.-B., R.-J. Feng, and K. Ura. "Utilizing the charging effect in scanning electron microscopy." Science progress, 2004. 87(4): p. 249-268.

8. Grella, L., et al. "Simulations of SEM imaging and charging." Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, 2004. 519(1-2): p. 242-250.

9. Flatabø, R., A. Coste, and M. Greve. "A systematic investigation of the charging effect in scanning electron microscopy for metal nanostructures on insulating substrates." Journal of Microscopy, 2017. 265(3): p. 287-297.

10. Chetana, S., et al. "Study on the DC supply and charging effect on the growth of carbon nanotubes and their electrochemical properties." Journal of Materials Science: Materials in Electronics, 2022. 33(25): p. 19937-19946.

11. Bayazid, S.M., et al. "Investigation of the Effect of Magnification, Accelerating Voltage, and Working Distance on the 3D Digital Reconstruction Techniques." Scanning, 2020. 2020: p. 1-9.

12. Ichinokawa, T., et al. "Charging effect of specimen in scanning electron microscopy." Japanese Journal of Applied Physics, 1974. 13(8): p. 1272.

13. IS1498, Classification and identification of soils for general engineering purposes. Indian Standards, Bureau of Indian Standards, 1–28. 2007.

14. IS2720-4. Indian Standard, Methods of Test for Soils, Part 4: Grain Size Analysis [CED 43: Soil and Foundation Engineering]. 1985.

15. Carl, Z., Carl Zeiss AG - EVO50 Series. EVISA. 2003.

16. Borchert, A., K. Vecchio, and R. Stein. "The use of charging effects in Al/Al2O3 metal-matrix composites as a contrast mechanism in the SEM." Scanning, 1991. 13(5): p. 344-349.

Сканерлеуші электронды микроскопиядағы (СЭМ) топырақ бөлшектеріне зарядтың әсері

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Аңдатпа. Сканерлеуші электронды микроскоп (СЭМ), материалдардың сипаттамаларын анықтауға арналған құрал, борпылдақ материалдардың беті мен жер асты қабаттары, құрамы мен ақаулары туралы ақпарат алуға мүмкіндік береді. Мақаланың мақсаты-сканерлеуші электронды микроскопия (СЭМ) кезінде зарядтауға топырақ бөлшектерінің параметрлерінің әсерін түсіну. СЭМ суреттері коллювиалды топырақ бөлшектерінде бөлшектердің мөлшерін (A=2-1 мм, B=0,6-0,425 мм, C=0,3-0,212 мм және D= <0,075 мм) және өткізгіш жабындардың санын (жабынсыз, бір және қос) өзгерту арқылы алынды. Мақалада топырақ бөлшектерінің барлық түрлеріне қолданылатын СЭМ әдісімен кескіндерді алу үшін топырақ бөлшектерін дайындау әдісі ұсынылған. Зерттеу көрсеткендей, 212 мкм - ден асатын топырақ бөлшектері зарядтау әсерін болдырмау үшін қос өткізгіш жабынды, ал 75 мкм-ден аз бір жабынды қажет етеді. Кескіннің айқындылығы 212 мкм ден асатын топырақ бөлшектері үшін күмәнді Қос өткізгіш жабындарды қолданғаннан кейін 10000 есе және одан жоғары үлкейту кезінде.

Түйін сөздер: сканерлеуші электронды микроскопия, топырақ бөлшектері, зарядтау әсері.

Влияние заряда на частицы почвы при сканирующей электронной микроскопии (СЭМ)

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Аннотация. Сканирующий электронный микроскоп (СЭМ), инструмент для определения характеристик материалов, позволяет получить информацию о поверхности и подповерхностных слоях, составе и дефектах сыпучих материалов. Цель статьи - понять влияние параметров частиц грунта на зарядку при сканирующей электронной микроскопии (СЭМ). СЭМ-изображения были получены на частицах коллювиального грунта путем варьирования размера частиц (A=2-1 мм, B=0,6-0,425 мм, C=0,3-0,212 мм и D= <0,075 мм) и количества проводящих покрытий (без покрытия, одинарное и двойное). В статье предлагается способ подготовки частиц почвы для получения изображений методом СЭМ, который применим для всех типов частиц почвы. Исследование показало, что частицы грунта размером более 212 мкм требуют двойного токопроводящего покрытия, а менее 75 мкм - одинарного покрытия, чтобы избежать эффекта зарядки. Резкость изображения сомнительна для частиц грунта размером более 212 мкм при увеличении в 10000 раз и выше после нанесения двойных проводящих покрытий.

Ключевые слова: сканирующая электронная микроскопия, частицы почвы, зарядный эффект.