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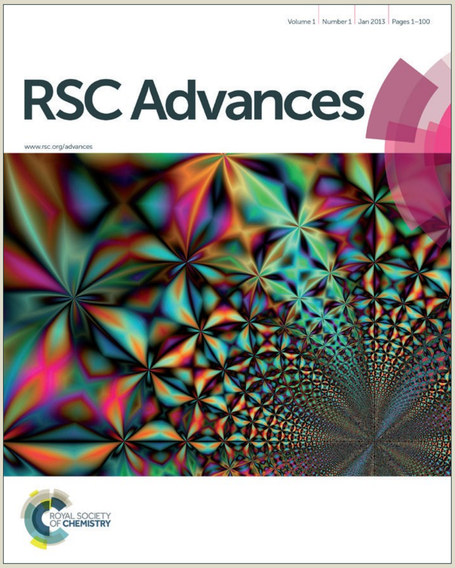

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Granulous KMS-1/PAN composite for Cs^+ removal

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A novel KMS-1/PAN composite was successfully fabricated simply by combining KMS-1 with PAN. The KMS-1/PAN combines the rapid adsorption of Cs^+ efficiently by KMS-1 with the granulation for easy separation after adsorption. The maximum Cs^+ adsorption capacity of KMS-1/PAN was estimated to be 72.00 mg/g according to Langmuir model.

Nuclear power has been regarded as a sustainable energy source that reduces carbon emissions. However, it poses potential threats to living organisms and environment for extremely long existence of radioactive pollutant due to nuclear leakage and unsuitable disposal of nuclear waste.[1-4] Presently, radioactive pollution is getting more and more attention for the bloom of nuclear plant. Among the radioactive pollutants, ^{137}Cs is one of the most harmful radioactive nuclides and it was one of the largest amounts of radionuclides released from the Fukushima Daiichi Nuclear Power Plant accident in 2011 due to magnitude 9.0 earthquake and tsunami. Furthermore, ^{137}Cs is more concerned because it is a major contributor to heat and radiation generation in the high active liquid waste disposal.[5, 6] In the past decades, various methods have been attempted to remove Cs^+ ions, such as ion-exchange, evaporation and electrodeionization.[7-9] Undoubtedly, ion exchange is an environmentally friendly technology with ease manipulation.[4] So far, many kinds of ion exchangers including inorganic and organic ones had been applied for the removal of Cs^+.[7, 10] Inorganic ion exchangers had better chemical and thermal stability than organic ion-exchangers and could be as promising advanced materials for the removal of Cs^+. Among them, oxide-based ion exchangers, such as silicotitanate and graphene oxide-based materials, have been widely used to remove Cs^+.[3, 11]

Compared to oxide-based ion exchangers, which is only effective in narrow pH range, the metal chalcogenides might exhibit superior adsorption of Cs^+ ions over the wide pH range[12]. Furthermore, the selectivity of Cs^+ over other alkali ions was also confirmed due to the more favourable Cs···S soft Lewis acid/Lewis base interactions. However, the removal of Cs^+ by chalcogenides from water had seldom been investigated probably due to the difficult preparation and easily hydrolyzation of chalcogenides. The available examples include $K_{2x}Mn_xSn_{3-x}S_6$ (x = 0.5-0.95) (KMS-1), $K_{2x}Mg_xSn_{3-x}S_6$(x = 0.5–1) (KMS-2) and $[(CH_3)_2NH_2]_2Ga_2Sb_2S_7{\cdot}H_2O$.[12-14] KMS-1 has been proved to be an excellent adsorbent for the removal of Sr^{2+}, Cs^+, Hg^{2+}, Pb^{2+} and Cd^{2+}, particularly under extremely alkaline conditions and with large excess of disturbed ions such as Na^+.[12, 15] However, the reported chalcogenides including KMS-1, used as adsorbents are powder morphologies and might encounter the difficulties of being separated from aqueous solution after treatment in wastewater, which limited its practical applications.

Magnetic functionalization and macro-scale granulation could be possible solution to the engineering application of these promising materials. As far as oxides are concerned, they can be easily functionalized with magnetic properties. The examples include magnetic (Cr, Mn, Fe, Co, Ni) oxide nanocrystals, core-shell structured Fe_3O_4@alpha-MnO_2 microspheres, graphene oxide-Fe_3O_4 nanoparticles hybrid and magnetic chitosan/graphene oxide composites.[16-20] But for chalcogenides, it might be extremely difficult to obtain high-quality monocrystalline hetero structure magnetic chalcogenides due to the great lattice mismatch difference between Fe_3O_4 or zero valent iron and chalcogenides.[21] It is apparent that granulation of chalcogenides with polymer would be a more suitable way for engineering application of chalcogenides.

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Electronic Supplementary Information (ESI) available: The TEM images of the sliced KMS-1/PAN, the EDS of KMS-1, percentage of Cs^+ adsorption by KMS-1/PAN with various amount of KMS-1, recipes for KMS-1/PAN composite adsorbent, synthesis of KMS-1 and KMS-1/PAN, competitive adsorption of KMS-1/PAN and adsorption kinetics. See DOI: 10.1039/x0xx00000x

Polyacrylonitrile (PAN) is an ideal polymeric matrix with a series of merits, such as solvent resistance, thermal and mechanical stability, abrasion resistance, and high tensile strength.[22] It has been proposed as a universal binding polymer for inorganic ion-exchanger by Šebesta.[23] So far, PAN has been applied as a binding polymer to support various inorganic adsorbents, such as KCFC-PAN, PAN-TiO_2, cerium molybdate–PAN and PAN-2-amino-1,3,4-thiadiazole.[24-27] It is worthwhile to mention that Riley et al. reported the PAN-chalcogel hybrid sorbents, which made the chalcogels more mechanically stable for $I_2(g)$ by imbedding them in PAN.[28] Inspired by this, herein, we unprecedentedly use PAN to support layered metal sulphide, KMS-1, to form granulation of the composite (KMS-1/PAN), and investigate its property to remove Cs^+. The composite was firstly characterized by powder X-ray diffraction, transition electron microscopy and scanning electron microscopy equipped with an energy dispersive spectroscopy detector. Secondly, the effects of solution pH and contact time on the removal were investigated. Thirdly, various kinetics and isotherms have also been discussed for the removal of Cs^+ from aqueous solution. These results might significantly contribute to the practical application of chalcogenides as adsorbents in the treatment of waste water.

The photographs of PAN and KMS-1/PAN beads are shown in the Figure 1. It is apparent that both PAN (Figure 1a) and KMS-1/PAN beads (Figure 1b) are spherical, while the pure PAN beads are white in contrast to grey colour of the KMS-1/PAN, and the diameters of the pure PAN beads and KMS-1/PAN beads are about 2 mm and 3 mm, respectively. The microstructure of PAN and KMS-1/PAN are shown in the Figure 2. As shown, SEM images of cross-sectional samples indicate the porous structure existed inside the PAN and KMS-1/PAN beads. The addition of KMS-1 to PAN seems to increase the surface area of the beads. It can be indicated from the specific surface area of the pure PAN and PAN/KMS-1 beads, which are 3.30 m²/g and 7.89 m²/g, respectively. In addition, the porosities of PAN beads and KMS-1/PAN beads were evaluated according to Eqn (1) and (2).[29] It can be calculated that the porosity of pure PAN beads is 82.16%, while after granulation, the porosity of KMS-1/PAN increases to 91.76%. The reason might be due to the including large size of KMS-1 powder. The result indicates the well dispersion of KMS-1 in the supporter of PAN, and KMS-1/PAN can be used as a promising adsorbent in the environmental remediation.

$$\text{apparent density } (\mathrm{g\cdot cm^{-3}}) = \frac{\text{pellets mass (g)}}{\text{apparent volume (cm}^3)} \qquad [1]$$

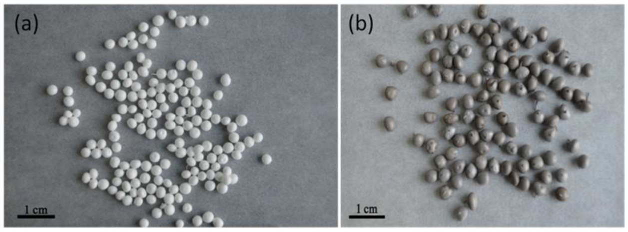

Fig. 1 Photographs of pure PAN beads (a) and KMS-1/PAN beads (b).

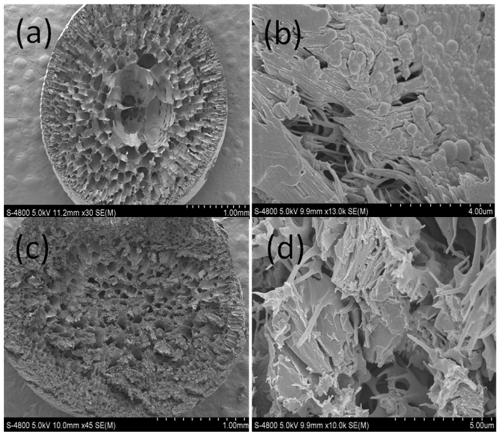

Fig. 2 The SEM images of pure PAN ((a) and (b)) and KMS-1/PAN ((c) and (d)).

$$\text{porosity} = \left(1 - \frac{\text{apparent density } (\mathrm{g\cdot cm^{-3}})}{\text{bulk density of mixture } (\mathrm{g\cdot cm^{-3}})}\right) \times 100\% \qquad [2]$$

To further study the component and structure of the composite, the samples were examined by TEM, EDS and PXRD. Figure S1 show typical TEM images of one slice of KMS-1/PAN. The grey color composition is PAN and the dark color composition can be ascribed to be KMS-1. It indicates that KMS-1 was mixed well with PAN to form KMS-1/PAN. The SAED was used to further confirm the existence of KMS-1 in the beads. As shown in the Figure 3, K, Mn, Sn and S of KMS-1 composition can be clearly detected in the designated area of KMS-1/PAN. The chemical composition of KMS-1 in this experiment is about $K_{1.74}Mn_{0.87}Sn_{2.13}S_6$ according to the result of the EDS analysis (Figure S2). The XRD patterns of KMS-1 powder, pure PAN and KMS-1/PAN beads are shown in the Figure 4 (a). The (003) and (006) Bragg peaks are easily observed on the pattern of KMS-1, which is consistent with previous reports,[12] and indicates the successful preparation of KMS-1. The pattern of KMS-1/PAN matches that of pure KMS-1 well, and the additional peak at 2θ ~ 17° can be ascribed to that of pure PAN pattern. There are no peaks shifted and no additional peaks emerged in the pattern of KMS-1/PAN. Thus, it can be concluded that the KMS-1 was combined successfully with PAN and the new desired spherical adsorbent which didn't change the phase of KMS-1 and PAN was formed. The contrast of the XRD patterns of KMS-1/PAN before and after Cs^+-exchanged is shown in Figure 4 (b). A slight shift of the

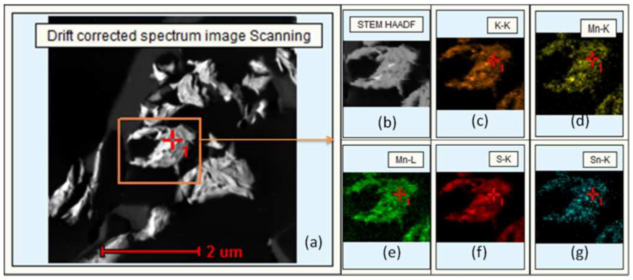

Fig. 3 (a) the drift corrected spectrum image, (b) the STEM HAADF, (c) (d) (e) (f) (g) are the pulse spectra of K-K, Mn-K, Mn-L, S-K and Sn-K respectively.

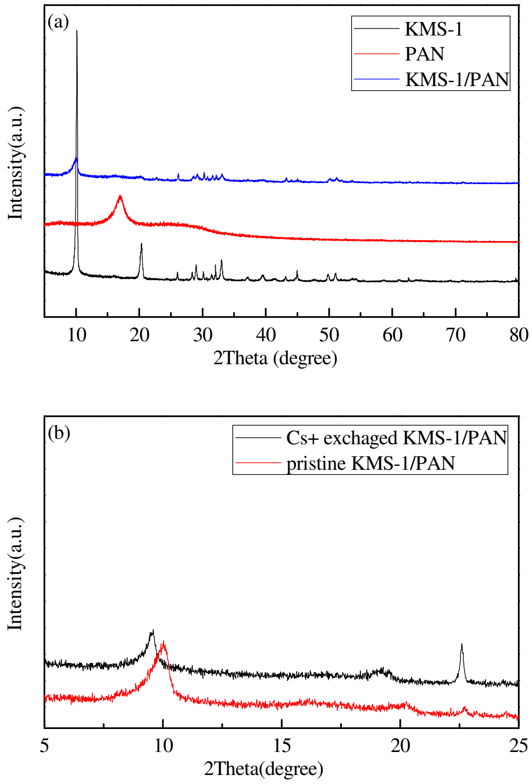

Fig. 4 (a) The X-ray powder diffraction patterns for the pristine KMS-1 (dark), PAN (red) and KMS-1/PAN (blue), (b) The X-ray powder diffraction patterns for the pristine KMS-1 (red) and the Cs^+ exchanged KMS-1.

(003) and (006) Bragg peaks to lower 2θ values, from 10.11° to 9.55° and from 20.25° to 19.07°, appeared in the Cs^+-exchanged KMS-1/PAN material, which suggest ion exchange reaction of the KMS-1 occured in the KMS-1/PAN beads. The interlayer distance of KMS-1 in the composite was estimated to be 8.68 Å, and, after exchange, the interlayer distance of KMS-1 was changed to 9.19 Å. This similar phenomenon was also observed in the previous Cs^+-exchanged KMS-1.[12]

To optimize the KMS-1/PAN composite for the remediation of Cs^+ in the wastewater, KMS-1/PAN beads with different rations of KMS-1 and PAN was prepared (Table S1) and tested for the adsorption of Cs^+ from the 100 mg/L caesium nitrate solution, and the results are showed in Figure S3. It shows that the adsorption efficiency of KMS-1/PAN increased with the increase of KMS-1 component and tended to be constant in the end. When the KMS-1 content was 33.3% and 50%, 54.3% and 57.1% of the initial Cs^+ amount was removed. When the KMS-1 content further increased to 60%, 66.7% and 71.4%, the Cs^+ removal reached to constant of about 66%. Considering minimizing the dose of PAN in the experiment, KMS-$1_{71.4}$ was selected as the optimal adsorbent in this research.

The effect of solution pH on the removal efficiencies of Cs^+ was also investigated. The pH effect on the adsorption of Cs^+ was examined at the pH range of 0.9-11.95 with 5 ppm Cs^+ solution. As shown in the Figure 5, at pH of 0.9 and 2, the adsorption of Cs^+ by KMS-1/PAN was relatively low. When pH was 3.16, the adsorption increased, and at pH value of 3.95 the adsorption reached to 2.26 mg/g, which was 83.7% of the maximum value. From pH of 4.87-11, the adsorption capacity had no obvious change and reached the maximum value of 2.7 mg/g. A drop of Cs^+ adsorption was observed at pH~12. The similar trend was also indicated from the result of KMS-1.[12] The possible explanation can be suggested as that at low pH value of 1 - 3 in the solution, H^+ would compete with Cs^+ to exchange with K^+ in the KMS-1. The extensive capture of Cs^+ by KMS-1/PAN under acidic conditions was attributed to the remarkable adsorption capacity of KMS-1 under strongly acidic conditions and the wider interlayer space in the KMS-1.

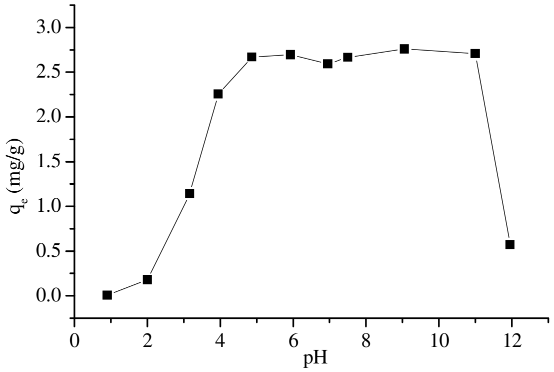

Fig. 5 Effect of pH on the adsorption of Cs^+ by KMS-1/PAN (V/m=1000 mL/g, initial concentrations of Cs^+ was about 5 ppm).

The adsorption efficiencies of Cs^+ at different contact time (1-25 h) with the initial Cs^+ concentration of 8 ppm were given in the Figure 6. The concentration of Cs^+ decreased rapidly within the first 1 h and then gradually reached an equilibrium value in 3 h. A further increase in contact time showed no significant effect on the adsorption capacity. The fast adsorption equilibrium may be attributed to the porous structure of the composite and the strong affinity of Cs^+ by KMS-1. It has been reported that high mobility of the interlayer K^+ ions and the higher affinity of the framework for the relatively “soft” Cs^+ ions over the harder K^+ ions of KMS-1 according to Lewis acid/Lewis base interactions.[12] The results indicate that the KMS-1/PAN could be a promising adsorbent

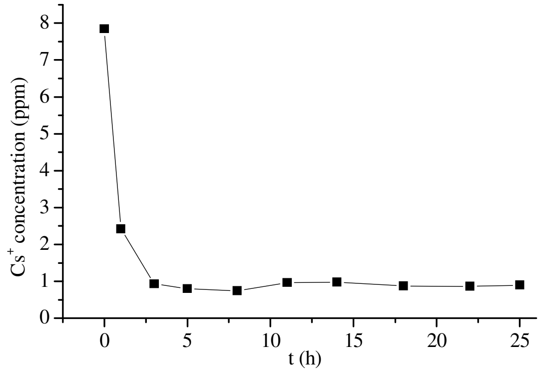

Fig. 6 The effect of contact time on the uptake of Cs^+ by KMS-1/PAN (V/m = 1000 mL/g, pH ~ 7, initial concentrations of Cs^+ was about 8 ppm).

Table 1.The value of the maximum adsorption quantities and Cs^+ uptake constants of KMS-1 and KMS-1/PAN got from the Langmuir, Freundlich and Langmuir-Freundlich adsorption isotherm models.

Adsorbent	Langmuir			Freundlich			Langmuir-Freundlich			
	q_e(mg/g)	b(L/mg)	R^2	K_F	n	R^2	q_e(mg/g)	b(L/mg)	n	R^2
KMS-1	100.878	0.151	0.936	44.085	6.760	0.857	107.352	0.136	1.369	0.974
KMS-1/PAN	72.003	0.097	0.924	31.173	6.931	0.806	75.232	0.095	1.276	0.930

for the removal of Cs^+ from aqueous environment. The KMS-1/PAN allows rapid ion exchange kinetics of the intercalated K^+ ions with Cs^+, in order to further study the adsorption process of Cs^+ onto KMS-1/PAN, the adsorption kinetics was analyzed using pseudo-first-order and pseudo-second-order models. The calculated kinetic parameters for pseudo-first-order and pseudo-second-order models were listed in Table S2 and related discussion was presented in the electronic supplementary information.

The equilibrium data of the Cs^+ ion exchange experiments of the KMS-1/PAN, KMS-1 powders and pure PAN beads under various concentrations (40-500 mg/L, V/m = 1000, pH ~ 7,temperature 25 °C) were shown in the Figure 7. The data can be fitted with the various models expressed as

a) Langmuir
$$q = q_m \frac{bC_e}{1+bC_e} \quad [3]$$

b) Freundlich
$$q = K_F (C_e)^{\frac{1}{n}} \quad [4]$$

c) Langmuir-Freundlich
$$q = q_m \frac{(bC_e)^{\frac{1}{n}}}{1+(bC_e)^{\frac{1}{n}}} \quad [5]$$

Where q (mg/g) is the amount of the ions adsorbed at the equilibrium concentration C_e (ppm), q_m is the maximum adsorption of the adsorbent, and b (L/mg) is the Langmuir constant associated with the free energy of adsorption, K_F and 1/n are the Freundlich constants.

Results of the fitting of various models are shown in Table 1. It suggests from the correlation coefficient (R^2) that the data was best fit by Langmuir and Langmuir-Freundlich models and the correlation coefficients (R^2) of the fitting of the Cs^+ exchange equilibrium data of KMS-1/PAN with Langmuir and Langmuir-Freundlich were 0.924 and 0.930. It can be found that the maximum Cs^+ adsorption capacity of KMS-1 and KMS-1/PAN were about 100.88 mg/g and 72.00 mg/g, respectively, according to Langmuir model. In contrast, the adsorption capacity of Cs^+ by pure PAN was approximate zero. It indicated that the Cs^+ adsorption by PAN pellets without KMS-1 could be neglected. The adsorption capacity of as-prepared KMS-1 was less than that in the previous research.[12, 30] It may be due to the less x value (0.87) in the $K_{2x}Mn_xSn_{3-x}S_6$ (x = 0.5-0.95) (KMS-1) as-prepared, which means that the amount of exchangeable K^+ ions was less than that reported (0.95).[12] The maximum adsorption value of Cs^+ by KMS-1/PAN was less than that of KMS-1, however, separation of the spherical beads from solution was easily achieved by simple and fast filtering, and the practical use of the composite exchangers may not be influenced by the slower adsorption of caesium.

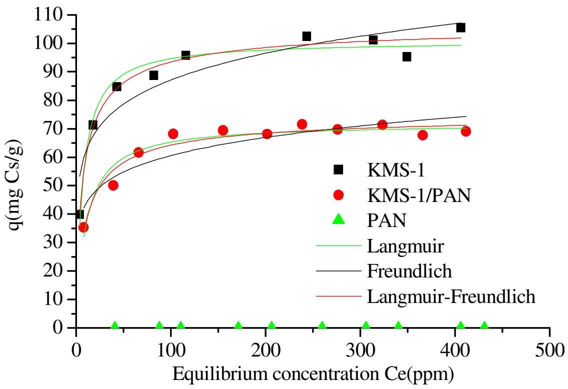

Fig. 7 Effect of concentration on the Cs^+ adsorption capacities of KMS, KMS-PAN and PAN beads (V/m=1000 mL/g, pH~7, 24h, initial concentrations of Cs^+ varies from 50-500 ppm).The lines was the fitting of the data with various isotherm models.

In order to examine the selectively of KMS-1/PAN for Cs^+ and other heavy metal ions, the Sr^{2+}, Cu^{2+}, Pb^{2+}, and Ni^{2+} was added into the Cs^+ solution with the same molar concentration (0.7524 mmol/L), respectively. The distribution coefficients of testes metal were shown in Fig S4. The competitive K_d values for Cs^+, Sr^{2+}, Cu^{2+}, Pb^{2+}, and Ni^{2+} are 35, 40, 754, 567 and 139 mL/g, respectively. The result showed that the adsorption of Cs^+ was affected significantly when Sr^{2+}, Cu^{2+}, Pb^{2+}, and Ni^{2+} existed. According to the hard and soft acids and bases theory, Pb^{2+}, Ni^{2+} and Cu^{2+} are border acid while Cs^+ and Sr^{2+} are hard acid. The sulfur atom is prone to bond with Pb^{2+}, Ni^{2+} and Cu^{2+} that made the removal efficiency of Cs^+ decreased greatly. As for the Sr^{2+}, it is bivalent and has the stronger competitiveness than univalent Cs^+. These results are consistent with the reports in the literatures.[30, 31]

The maximum adsorption capacity of the KMS-1/PAN for removal of Cs^+ has been compared with those of some adsorbents reported in the literatures (Table S3). As shown, the maximum adsorption capacity of Cs^+ by KMS-1/PAN is higher than those of some natural materials, but lower than those of some synthetic adsorbents.[12, 32-39] Nevertheless, KMS-1/PAN is still a promising adsorbent for radioactive ions due to its several advantages such as potentially safe as permanent materials, low cost and application respect under alkaline conditions.

In summary, we unprecedentedly use PAN to support layered metal sulphide, KMS-1, to form granulation of the composite KMS-1/PAN. The specific surface area and porosity of the composite was 7.89 m²/g and 91.76%, respectively. The batch adsorption experiments showed that the composite exhibited favourable Cs^+ adsorption capacity under a wide range of pH (3-11) and the kinetic studies of Cs^+ adsorption reflected the relative rapid adsorption rate with about 90% removal of Cs^+ within 3 h. The maximum Cs^+ adsorption capacity was 72.00 mg/g according to Langmuir model. These results might significantly contribute to the practical application of KMS-1/PAN as an adsorbent in the treatment of waste water with radioactive pollution.

Acknowledgments

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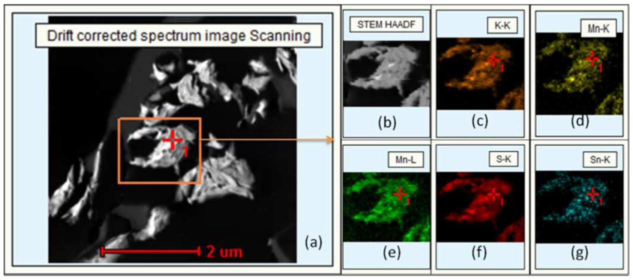

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