Asian Journal of Chemistry; Vol. 27, No. 12 (2015), 4417-4424

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2015.19152

Distribution of Selenite and Selenate with Weathering in Various Soil Parent Materials

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Received: 3 March 2015; *Accepted*: 24 April 2015; *Published online*: 29 August 2015; AJC-17483

Selenium speciation controls the bioavailability in soils. It is hypothesized that selenium species vary with soil parent material, and genesis affect redistribution in soil. Objectives of the study were to determine selenium species in relation with soil genesis and parent material. Triplicate soil profiles were selected at three distinct stages of development in each of loess, alluvium, shale and sandstone residuum and sampled at genetic horizon level. Basic characteristics and total selenium was determined at Department of Soil Science and Soil and Water Conservation, Pir Mehr Ali Shah Arid Agriculture University, Rawalpindi, Pakistan and selenate and selenite species were determined at Department of Crop and Soil Sciences, University of Georgia USA, over the period from February 2014 to March 2015. Soil pH, redox potential, calcium carbonate, dissolved and total organic carbon and dithionite and oxalate extractable iron were determined. Selenite and selenate were extracted in 0.1 N NaOH for determination using IC-ICP/MS. Selenite distribution differed significantly with parent material. The mean selenite distribution in parent materials followed the order shale > loess > alluvium > sandstone. Selenate was highest in shale parent material, while other three parent materials were similar in distribution. Soil total selenium also differed statistically with parent material. Mean selenite was 5-7 % of total selenium in alluvial, 1-2.7 % in loess, 3-7 % in sandstone and 3-4 % in shale derived soils. Mean selenate was 6-7 % of total selenium in alluvium and 4-4.5 % in loess; 8-11% in sandstone and 5 % in shale except surface had 30 % in Murree soil profile. This study helps the better understanding of selenate and selenite distribution in soil derived from different parent materials.

Keywords: Parent materials, Selenate, Selenite, Distribution, Weathering.

INTRODUCTION

Selenium, a trace element intermediate between metals and non-metals, is an essential element for human and animals[1]. Selenium is taken up by plants although not as an essential element. Plants but serves as a major source of Se for humans and animals[2]. Concentration of selenium in plants is strongly related with selenium species, and total content in soils which eventually affects on humans and livestock through plant selenium intake[3].

Selenium content in soils is controlled by parent material and its distribution occurs under soil forming processes. Therefore, soil properties and soil forming processes determine the forms and content of selenium[4,5]. Selenium in soils has different organic and inorganic compounds. The inorganic compounds occur as selenate (SeO_4^{2-}), selenite (SeO_3^{2-}), elemental selenium (Se^0) and selenide, while organic compounds exist in the form of selenomethionine, selenocystein, methylselenides and trimethyl selenium[6]. Selenium in organic compounds is part of molecular structures or is complexed[7]. Selenate is readily available to plants and selenite is less available due to its high adsorption on soil surfaces[8,9]. Selenium is taken up by plants in the forms of selenate, selenite and organic selenium. In mineral soils selenate and selenite are of more importance.

Information on the distribution of selenate and selenite in different parent materials with respect to weathering is limited. The current study quantifies the distribution of selenate and selenite along profile depths in soils derived from various parent materials with the goal to predict the selenium availability at different weathering stages in each parent material. It is hypothesized that selenate and selenite content differ with soil parent material and its distribution changes during weathering within the parent materials.

EXPERIMENTAL

Site description and soil sampling: Soils from different parent materials at three stages of weathering were sampled (Table-1). The parent materials included were alluvium, loess, residuum from sand stone and residuum from shale and sandstone. Soil profile was exposed and sampled at genetic horizon of every profile[10-14]. The samples were air dried before passing through 2 mm sieve.

TABLE-1
SELECTED SOILS IN EACH PARENT MATERIAL AT DIFFERENT STAGES OF DEVELOPMENT WITH USDA SOIL CLASSIFICATION

Parent material	Soil series	USDA soil classification
Alluvium	Shahdra	Typic Ustifluvents
	Argan	Fluventic Haplustepts
	Gujranwala	Typic Haplustalfs
Loess	Rajar	Typic Ustorthents
	Rawalpindi	Udic Haplustepts
	Chakwal	Typic Haplustafls
Sandstone	Qazian	Lithic Ustipsamments
	Balkassar	Typic Haplustalfs
	Kahuta	Udic Haplustalfs
Shale	Ghoragalli	Typic Udorthents
	Tirnul	Typic Haplustepts
	Murree	Typic Hapludolls

The loess is deposited during Pleistocene period in subhumid to semi arid climate and occupies level, sloping, erosional surfaces. The soils from loess parent material were Rajar, Rawalpindi and Chakwal. Alluvium parent material derived from Himalayas and deposited during Pleistocene period to present. Soils included from alluvial parent material were Shahdra, Argan and Gujranwala. The sandstone and shale-sandstone admixture comprise of Siwalik formation of Pliocene period and the Murree formation of Miocene period. Both the formations host residual soils where genesis is determined by surface relief. In sandstone the Qazian, Balkassar and Kahuta soils were sampled. Shale-sandstone soils sampled were Ghoragalli, Tirnul and Murree with admixture of sandstone.

Soil characteristics: Soil redox potential and pH were determined using 1:1 soil to water ratio[15]. Total extractable Fe from soil was extracted in 40 mL of 0.3 M sodium citrate bicarbonate dithionite ($C_6H_5Na_3O_4.2H_2O$) solution, 5 mL of 1 M sodium bicarbonate ($NaHCO_3$) solution heating at 80 °C and adding 1 g sodium dithionite ($Na_2S_2O_4$)[16]. Iron concentration in the extract was determined by atomic absorption spectrometer (AAS).

Calcium carbonate was determined by a procedure based on acetic acid consumption: known excess quantity of 0.4 M acetic acid is added to a given quantity of soil and pH of supernatant was measured after complete dissolution of the solid phase carbonate[17]. Organic matter was determined by wet digestion in 1 N potassium chromate solution and conc. H_2SO_4 and titrated against 0.5 N ferrous ammonium sulfate solution[18]. Dissolved organic carbon (DOC) was determined for each soil sample by extracting soluble organic carbon in potassium sulfate, digestion in potassium dichromate and concentrated sulfuric acid H_2SO_4 and measurement of excess potassium dichromate with ferrous ammonium sulfate[19].

Total selenium: Soil (1 g) was digested with 5 mL of aqua regia and 2 mL of HF and heated at 110 °C for 2 h. Boric acid (1 g) was used to complex the excessive HF and all the samples were stored at 4 °C before analysis[20].

Selenite and selenate: 5 g soil was taken and 50 mL of 0.1 M NaOH was added in centrifuge tube and shaken for 24 h and repeated to make 100 mL final volume for selenite and selenate determination on IC-ICP/MS [21].

Statistical analysis: The variance in the soil parameters was ascribed to source of parent material (PM) and soil (PM). The multivariate analysis was implemented using General Linear Model in SAS version 9[22].

RESULTS AND DISCUSSION

For understanding how distribution of selenate and selenite affected by weathering and pedogenesis, the soil characteristics (pH, Eh, $CaCO_3$, total organic carbon, dissolved organic carbon and extractable Fe) are presented (Tables 2 and 3). The distribution patterns of selenate and selenite in agricultural soils with respect to weathering stage and type of parent material (*i.e.* alluvium, loess, sandstone and shale) presented in figures to evaluate the occurrence of selenium deficiency and toxicity.

Soil characteristics: The selected soils had low organic matter. The most soils in all four parent materials had pH greater than 7 and calcareousness of soils varied between strongly calcareous to noncalcareous depending on the extent of soils development. The selenium species (selenate and selenite) related chemical properties are presented (Tables 2 and 3). The relatively weathered soils at higher stage of development (Alfisols and Mollisols) were decalcified, low pH, high oxalate and dithionite extractable iron as compared to least weathered soils at early stage of development (Entisols) were calcareous, high pH and were low in oxalate and dithionite extractable iron. The shale derived soils had higher dithionite extractable iron due to lithogenic hematite. Total and dissolved organic carbon content also varied with soil development within parent materials.

Soil $CaCO_3$, organic matter, dissolved organic carbon and extractable Fe were controlled by the relative intensity of weathering processes on each weathering stage (Tables 2 and 3). Decalcification in weathered soils in each parent material occurred up to (D5) 100+ cm profile depth. Moderately weathered soils on gently sloping areas decalcification was up to 50+ cm profile depth in Typic Haplustalfs loess parent material while in the alluvium, sandstone and shale there was 7, 12 and 6 % calcium carbonate respectively in moderately weathered soils. Soil profiles on least weather stage of development were moderately to strongly calcareous. All four parent materials had pH between 6.36-8.20, lower in weathered stages of soil development and higher in least weathered. Total organic carbon, dissolved organic carbon and extractable Fe increased toward surface in almost all soil profiles. The difference in Fe_d content among the soils with respect to weathering had prominent which suggests the release pedogenic Fe from phylosilicates (biotite, chlorite) and synthesis of iron oxides and/or eluviation/illuviation processes in relatively weathered stages of soils.

TABLE-2
MEANS VALUES OF BASIC SOIL CHARACTERS OF ALLUVIUM AND LOESS PARENT MATERIALS

Parent material	Weathering stage	Soil series	Soil classification	No. of depths	Clay (g 100 g^{-1})	DOC (mg kg^{-1})	$CaCO_3$ (%)	pH	Eh (mv)	TOC (g kg^{-1})	Fe_d (g kg^{-1})
Alluvium	Least weathered	Shahdra	Typic Ustifluvents	D1	21±3.4	29±16	15±0.6	7.7±0.3	249±3	2.18±0.8	12.6±1.57
				D2	22±4.3	23±7	15±1.3	7.6±0.3	246±3	1.99±0.6	12.3±1.75
				D3	23±3.6	28±7	15±1.2	7.8±0.2	252±3	0.65±0.5	13.5±1.23
				D4	18±3.4	23±5	14±2.1	8.0±0.2	248±4	0.85±0.7	11.3±1.26
				D5	23±3.7	31±3	13±1.7	7.9±0.1	254±4	0.86±0.3	14.0±1.87
	Moderately weathered	Argan	Fluventic Haplustepts	D1	27±3.2	43±17	7±0.6	7.6±0.2	247±3	3.02±0.9	9.5±1.27
				D2	33±3.4	27±7	7±1.3	7.7±0.3	246±3	1.03±0.4	11.3±1.33
				D3	28±3.6	16±6	8±1.9	7.6±0.3	242±3	1.04±0.5	10.1±1.40
				D4	27±3.4	16±5	7±2.9	7.7±0.1	253±3	1.04±0.6	10.5±1.37
				D5	25±3.7	14±3	7±2.7	7.7±0.1	268±3	1.05±0.3	9.5±1.67
	Weathered	Gujranwala	Typic Haplustalfs	D1	14±3.3	48±13	1±0.7	7.6±0.2	232±3	2.84±0.4	13.2±1.43
				D2	15±4.3	48±7	1±0.2	7.6±0.2	228±3	2.64±0.5	13.3±1.23
				D3	31±3.3	54±6	1±0.6	7.6±0.3	250±4	3.02±0.7	16.8±1.35
				D4	32±3.5	54±5	1±0.6	7.5±0.1	247±3	2.25±0.4	19.2±1.56
				D5	28±3.6	32±3	2±.4	7.4±0.1	246±3	1.07±0.4	15.3±1.45
Loess	Least weathered	Rajar	Typic Ustorthents	D1	21±4.1	65±20	15±0.6	7.4±0.2	249±3	5.28±0.3	8.7±1.44
				D2	21±5.2	49±9	18±0.8	8.0±0.3	239±4	2.04±0.4	9.1±1.34
				D3	21±4.1	33±8	15±1.2	8.1±0.3	245±3	2.04±0.6	8.3±1.24
				D4	24±4.4	25±5	12±1.3	8.2±0.1	243±3	1.08±0.5	8.7±1.66
				D5	26±4.5	19±4	13±1.4	8.2±0.1	254±3	1.28±0.4	5.1±1.33
	Moderately weathered	Rawalpindi	Udic Haplustepts	D1	24±3.2	60±14	1±0.3	7.0±0.2	302±4	5.09±0.3	9.4±1.43
				D2	23±4.1	54±5	1±0.3	7.2±0.2	280±3	3.36±0.5	9.4±1.42
				D3	33±3.4	27±3	1±0.1	6.9±0.2	290±2	2.39±0.6	12.0±1.33
				D4	27±3.5	27±4	12±1.2	7.7±0.3	294±3	0.81±0.7	9.55±1.23
				D5	26±3.7	22±4	15±1.1	7.8±0.2	299±4	0.57±0.4	8.93±1.32
	Weathered	Chakwal	Typic Haplustafls	D1	32±3.2	47±14	1±0.3	7.3±0.1	258±4	4.03±0.3	9.37±1.41
				D2	39±3.3	47±7	1±0.3	7.6±0.2	266±3	4.03±0.4	10.3±1.55
				D3	43±3.5	52±6	2±0.4	7.4±0.2	269±2	3.76±0.6	10.5±1.53
				D4	45±3.4	25±4	3.1±0.3	7.4±0.2	268±2	3.68±0.5	9.79±1.65
				D5	45±3.5	22±3	3.0±0.3	7.8±0.1	269±4	2.42±0.6	11.2±1.76

Total selenium: Mean total selenium in all four parent materials range from 300 to 2620 μg kg^{-1}. The highest contents in the shale parent material range from 736 to 2620 μg kg^{-1} followed by each lesser content in the loess, alluvium and sandstone parent materials. The difference between the parent materials remained statistically significant $F(3, 22) = 0.81$, $P \geq 0.504$) in all four parent materials considering the variation in parent materials are genetic or inherited from the source of different rocks reported by Cuvardic[29]. The shale and loess profiles had the highest mean, the alluvium and sandstone profiles lowest. The shale soil profiles had distinctly high total selenium in Murree profile surface have the highest selenium and other profiles of shale, loess; alluvium and sandstone have on average similar trends throughout the profiles' depth. Lowest selenium concentrations were recorded in sandstones and limestones and highest in shale (Fig. 1).

The alluvial soils had similar and uniform total selenium content throughout profiles depth. The mean total selenium content in loess soils increased towards surface in relatively weathered soil while consistent with depth in Rawalpindi and Rajar soils (Fig. 2).

The total selenium content in sandstone soils were consistent with soil depth except 4th horizon in Kahuta soil which may be attributed to lithological discontinuity. The total selenium in shale derived soils remained consistent with soil depth while increased towards the surface in Murree soil profiles at relatively higher stage of development and was highest of all parent materials on two surface horizons in murree soil (Fig. 2). High organic matter at the surface of murree soils may result in high total selenium as the correlation between organic carbon and total selenium content was statistically significant also reported by Eich-Greatorex *et al.*[22].

Selenite distribution: Mean selenite content ranged from 6 to 32 μg kg^{-1}. The mean selenite significantly varied vertically in soil profiles with parent material as the hypothesis of non-significant depth × PM effect was rejected through MANOVA test criteria Wilks' λ ($P \geq 0.0001$). The mean selenite concentration differed significantly among parent material ($P \geq 0.05$) at all the horizons level (Fig. 1). The shale derived soils had highest selenite throughout soil profile followed by alluvium and sandstone while the loess soils had the lowest selenite content. High selenite content in alluvium and shale derived soils may be due to high iron oxides content as selenite significantly correlate with dithionite extractable iron in soil ($r^2 = 0.5$)[23]. Lithogenic hematite in shale derived soils was reported by Mehmood[24] and pedogenic iron oxides (goethite) were reported in alluvial soil[25]. Low selenite content in sandstone derived soils may be due to less iron oxides content in coarse texture sandstone soils which has abundance of quartz mineral[26]. Selenite distribution in soils representing the three different weathering stages in each parent material is also presented (Fig. 3). The alluvial soils had similar selenite content throughout profile depth except 2nd depth of Shahdra soil profile which may be attributed to lithological discontinuity.

TABLE-3
MEANS VALUES OF BASIC SOIL CHARACTERS OF SANDSTONE AND SHALE PARENT MATERIALS

Parent material	Weathering stage	Soil series	Soil classification	No. of depths	Clay (g 100 g^{-1})	DOC (mg kg^{-1})	$CaCO_3$ (%)	pH	Eh (mv)	TOC (g kg^{-1})	Fe_d (g kg^{-1})
Sandstone	Least weathered	Qazian	Lithic Ustipsamments	D1	16±3.1	28±8	9±0.7	7.57±0.2	240±3	3.27±1.4	6.23±1.71
				D2	14±3.2	28±7	10±1.1	7.7±0.3	236±4	2.41±0.67	6.05±1.29
				D3	19±3.5	34±5	22±1.2	7.58±0.3	247±3	2.01±0.66	6.00±1.23
				D4	13±3.6	17±3	13±1.5	7.7±0.2	246±4	1.25±0.49	4.50±1.87
				D5	8±3.1	17±4	11±1.5	7.72±0.3	236±3	0.80±0.37	2.65±1.43
	Moderately weathered	Balkassar	Typic Haplustalfs	D1	23±3.4	57±7	2±0.3	7.6±0.2	232±3	4.25±0.8	6.10±1.43
				D2	35±4.3	57±8	2±0.6	7.42±0.2	228±3	4.53±0.6	6.09±1.11
				D3	40±3.6	51±6	6±0.4	7.38±0.3	239±3	4.30±0.6	9.18±1.24
				D4	38±3.4	28±4	5±0.4	7.52±0.2	240±3	3.21±0.4	9.44±1.32
				D5	32±3.7	28±3	5±0.6	7.53±0.1	236±4	2.40±0.5	9.90±1.22
	Weathered	Kahuta	Udic Haplustalfs	D1	16±3.3	49±11	0.59±0.01	6.37±0.2	270±3	3.62±0.5	9.63±1.22
				D2	15±4.3	30±7	0.59±0.05	6.36±0.3	266±3	3.62±0.3	9.12±1.43
				D3	28±3.4	30±6	0.59±0.03	6.58±0.2	274±4	3.66±0.4	10.90±1.43
				D4	31±3.6	27±6	0.61±0.05	6.70±0.3	265±3	3.46±0.5	12.05±1.51
				D5	25±3.2	14±3	0.84±0.04	6.72±0.2	261±3	1.98±0.6	11.01±1.52
Shale	Least weathered	Ghoragalli	Typic Udorthents	D1	38±3.2	98±17	17±1.2	7.48±0.1	243±4	9.60±0.5	21.17±1.66
				D2	35±3.2	28±7	18±1.1	7.4±0.2	267±3	4.60±0.5	19.77±1.61
				D3	46±3.5	31±6	15±2.1	7.42±0.3	262±2	4.39±0.6	19.17±1.62
				D4	44±3.3	11±5	12±0.9	7.47±0.2	251±4	4.42±0.7	18.97±1.75
				D5	39±3.6	22±3	12±0.8	7.47±0.1	251±4	1.98±0.5	21.15±1.55
	Moderately weathered	Tirnul	Typic Haplustepts	D1	33±3.4	30±12	13±1.3	7.54±0.2	241±3	2.25±0.6	18.06±1.65
				D2	34±4.5	24±6	13±1.6	7.53±0.2	239±4	1.74±0.5	17.72±1.66
				D3	34±3.4	22±7	9±1.3	7.54±0.3	230±3	1.28±0.6	18.61±1.69
				D4	30±4.2	22±4	12±0.6	7.69±0.1	228±3	1.03±0.5	16.90±1.56
				D5	30±3.2	19±3	6±0.7	7.72±0.1	223±3	0.70±0.4	18.93±1.71
	Weathered	Murree	Typic Hapludolls	D1	18±3.4	56±11	0.66±0.03	6.59±0.2	351±3	6.01±0.4	13.63±1.28
				D2	23±4.6	6±5	0.65±0.01	6.87±0.3	395±4	3.81±0.5	16.67±1.29
				D3	24±3.2	3±6	0.74±0.07	7.00±0.2	390±3	2.75±0.6	25.33±1.43
				D4	49±3.5	3±1	0.75±0.04	7.52±0.1	349±3	1.75±0.6	30.00±1.54
				D5	56±3.6	3±1	6.2±0.04	7.5±0.10	343±4	0.96±0.4	35.42±1.65

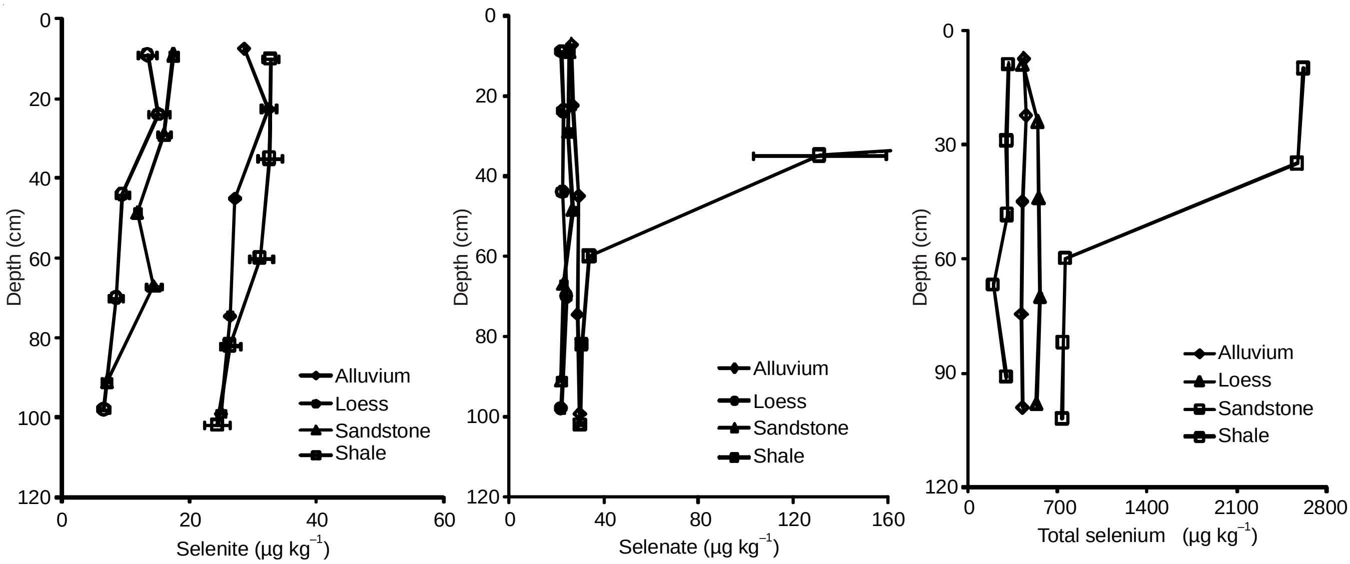

Fig. 1. Distribution of means selenite, selenate and total selenium in different parent materials

The mean selenite content in loess soils increased towards surface in relatively weathered soil while consistent with depth in young Rajar soil which might be due to less calcium carbonate on surface horizons increasing the selenite adsorption on dithionite extracted iron. The selenite content in sandstone soils were consistent with soil depth except 3rd depth in Balkassar soil which may be attributed to shale/ sandstone interbeding. The selenite distribution in shale derived soils remained consistent with soil depth in young Ghoragalli and intermediate stage of development (Tirnul soil) while increased towards the surface in soil relatively at higher stage of development (Murree soil). High organic carbon at the

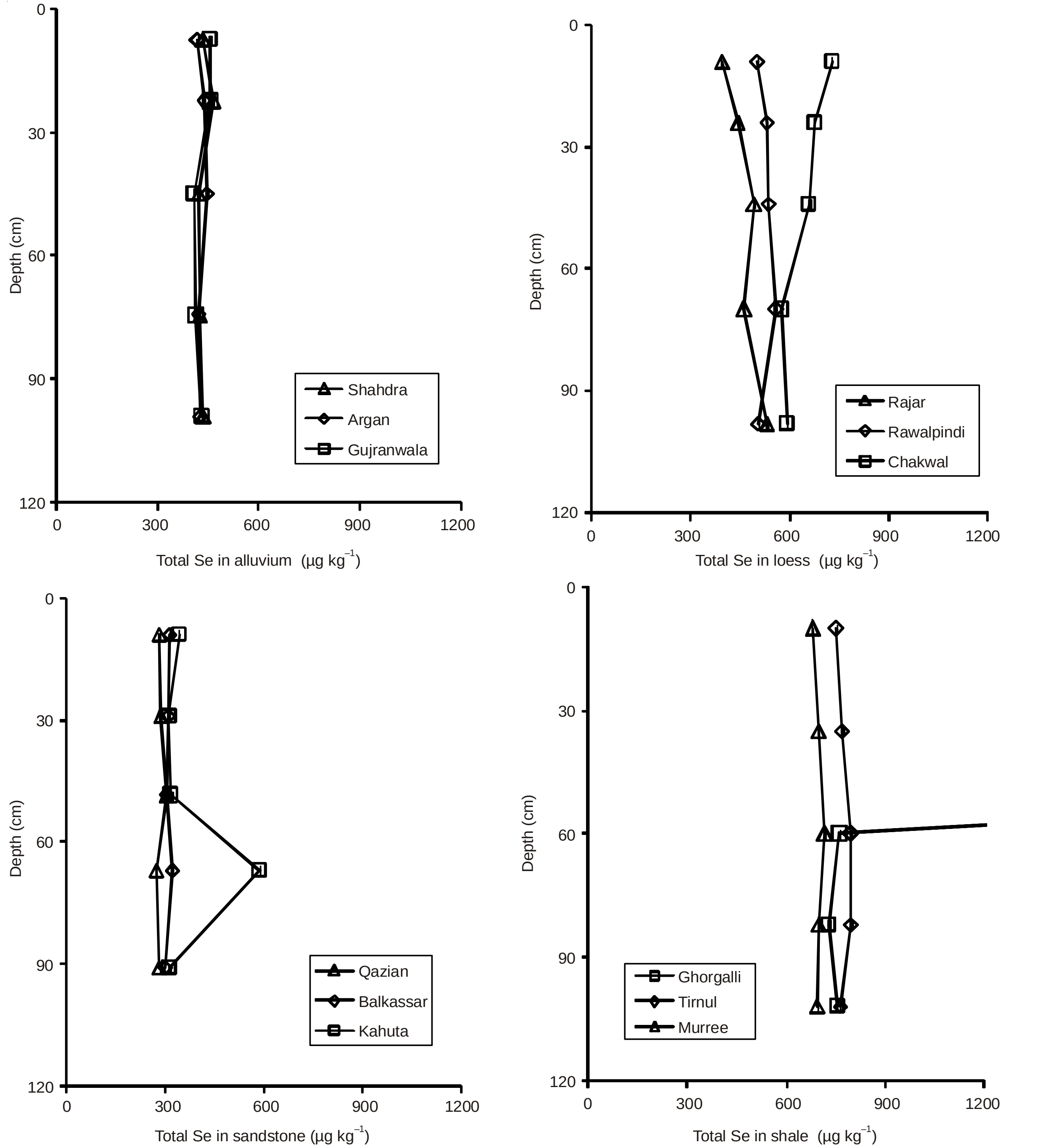

Fig. 2. Total selenium in soil of each parent materials at three different weathering stages

surface may result in high selenite content in Murree soil as the correlation between organic carbon and selenite content was statistically significant. As reported by Cary *et al.*[27] that the behaviour of selenium in calcareous system when soils are low in sesquioxides, the selenite becomes easily water soluble.

Selenite was 5-7 % of total selenium in alluvium, 1-2.7 % in loess, sandstone with 3-7 % and shale with 3-4 % on average (Fig. 5). Selenite had positive correlation for Fe_d, Fe_O, Eh, Clay, DOC, TOC (r^2 = 0.46, 0.13, 0.53, 0.11, 0.24, 0.34 respectively) and negative correlation with the pH r^2 = -0.23). Selenium distribution along soil profile resembles those of Fe and clay[21,22].

Selenate distribution: Selenate content in the data set ranged from 26 to 30 µg kg^{-1} while the surface horizon of Murree soil profiles had exceptionally high selenate contents. The mean selenate concentration significantly varied with depth in soil profiles with parent material as the hypothesis of non-significant depth × PM effect was rejected through MANOVA test criteria Wilks' λ ($p \geq 0.0001$). The mean selenate concentration differed significantly among parent material ($p \geq 0.05$) at most of the horizons level (Fig. 1). The shale derived

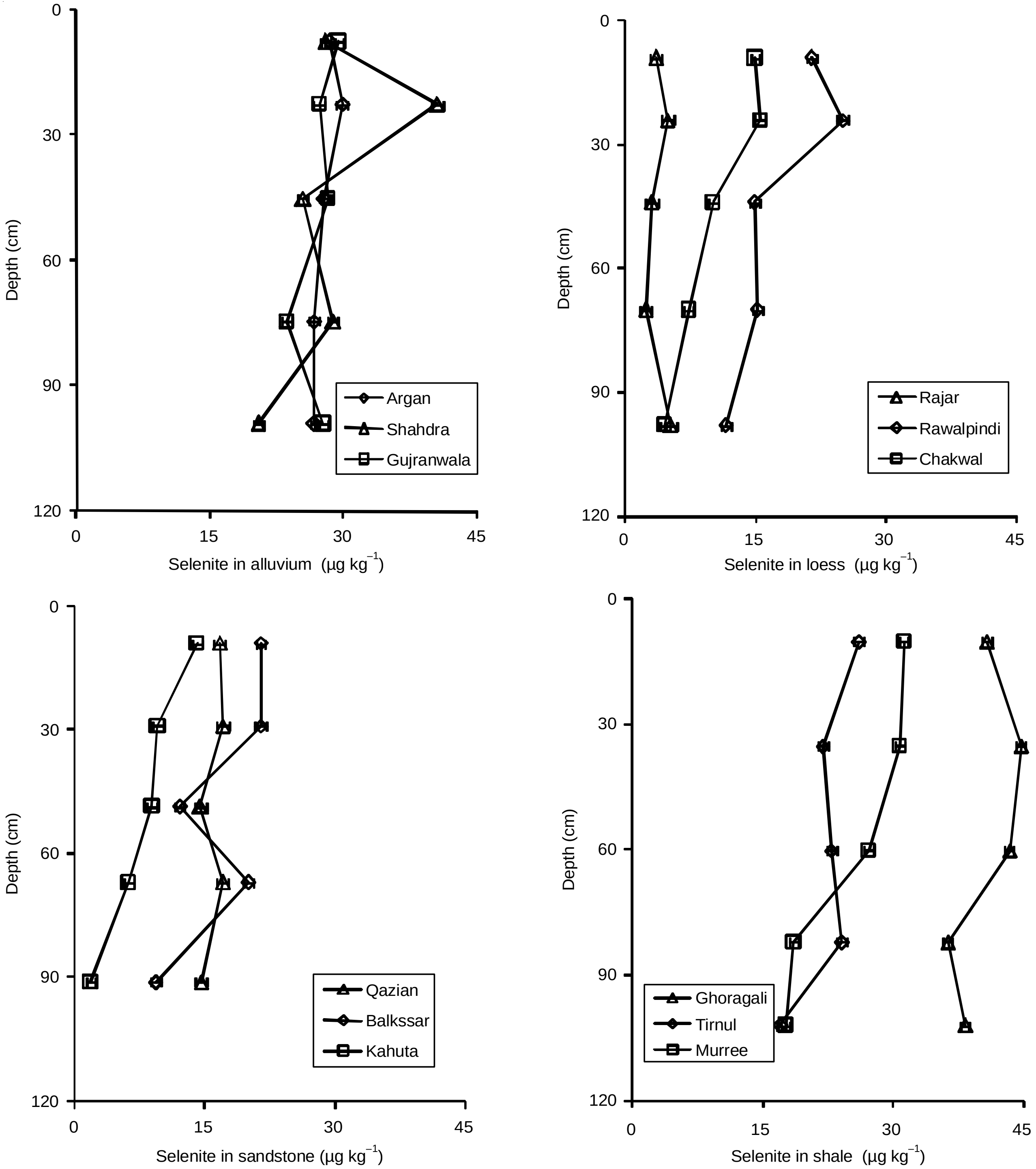

Fig. 3. Selenite distribution in each weathering stage of different parent materials

soils had significantly greater selenate content than alluvium, loess and sandstone derived soils. Mean selenate content increased towards the surface in the parent materials. High selenate content in the shale derived soils may be attributed to redox condition deposition of fresh rocks material material as well as organic matter content on surface. Selenate content in soils representing different stages of development is presented in Fig. 4. Selenate content increased towards surface in weathered Gujranwala soil but decreased towards the surface in young Shahdra and Argan soil. Selenate content in Rawalpindi and Rajar increased with depth while increased towards surface in weathered Chakwal soil. Selenate content in sandstone derived soils differed with soil development and depth, increased towards surface in Kahuta and Qazian while decreased towards surface in Balkassar soil. Selenate content increased towards surface in all the soils. The distribution of different selenium species of the shale parent material reflects the degree of surface exposure and weathering. Weathering of parent materials causes selenium oxidation and mobilization and selenium is bound to the organic and clay fractions[28,29].

Selenate of total selenium was 6-7 %, in alluvium, 4-4.6 % in loess, sandstone with 8-11% and shale with 5 % overall

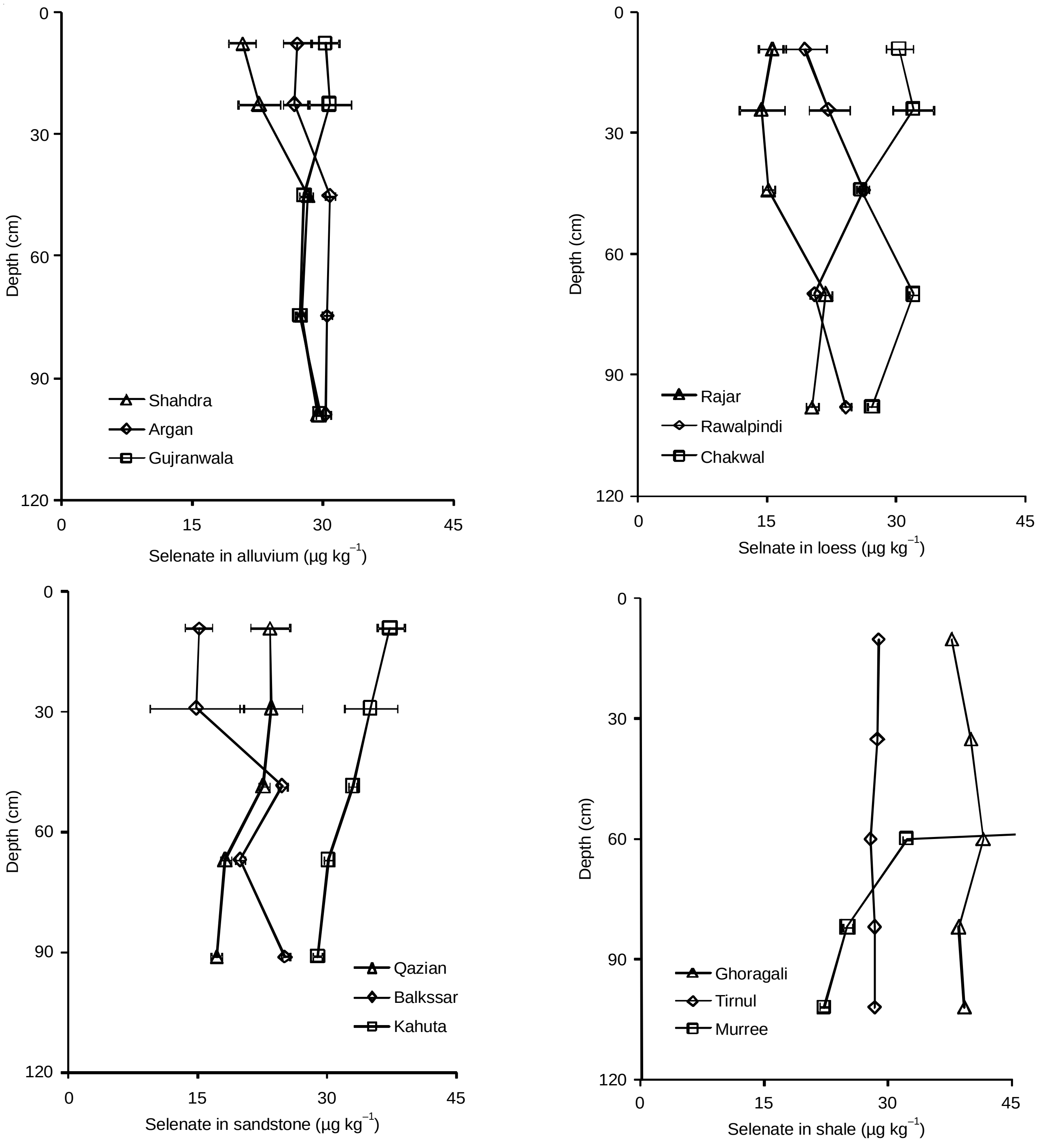

Fig. 4. Selenate distribution in each weathering stage of different parent materials

except on surface with 30 % (Fig. 5). Selenate had positive correlation for TOC, Fe_d, Fe_O, DOC, (r^2 = 0.69, 0.21, 0.50, 0.54 respectively) and negative correlation with the clay, $CaCO_3$ and pH r^2 = -0.019, -0.16 and -0.39). The extent of relationship of selenate with above soil properties is represented by the r^2 value for each property. Selenate in soil solution is dominant under well aerated conditions in semiarid region, which is not adsorbed and does not form insoluble salts[30].

Conclusion

Selenite and selenate distribution varied in each parent material and within parent material alluvium and shale parent materials have significantly higher selenite contents as compared to loess and sandstone. While selenate in parent materials was nearly similar in alluvium, loess, sandstone except the shale with highest selenate on surface. Distribution of selenite and selenate differed with type of parent material even within each parent materials there was different distribution pattern of selenite and selenate which is controlled by the soil properties beside the genetic parameters of the parent materials. For further understanding of selenium uptake selenium fractions as well as selenium species need to be investigated for each parent materials with goal to predict the rate of availability of selenium in each parent material.

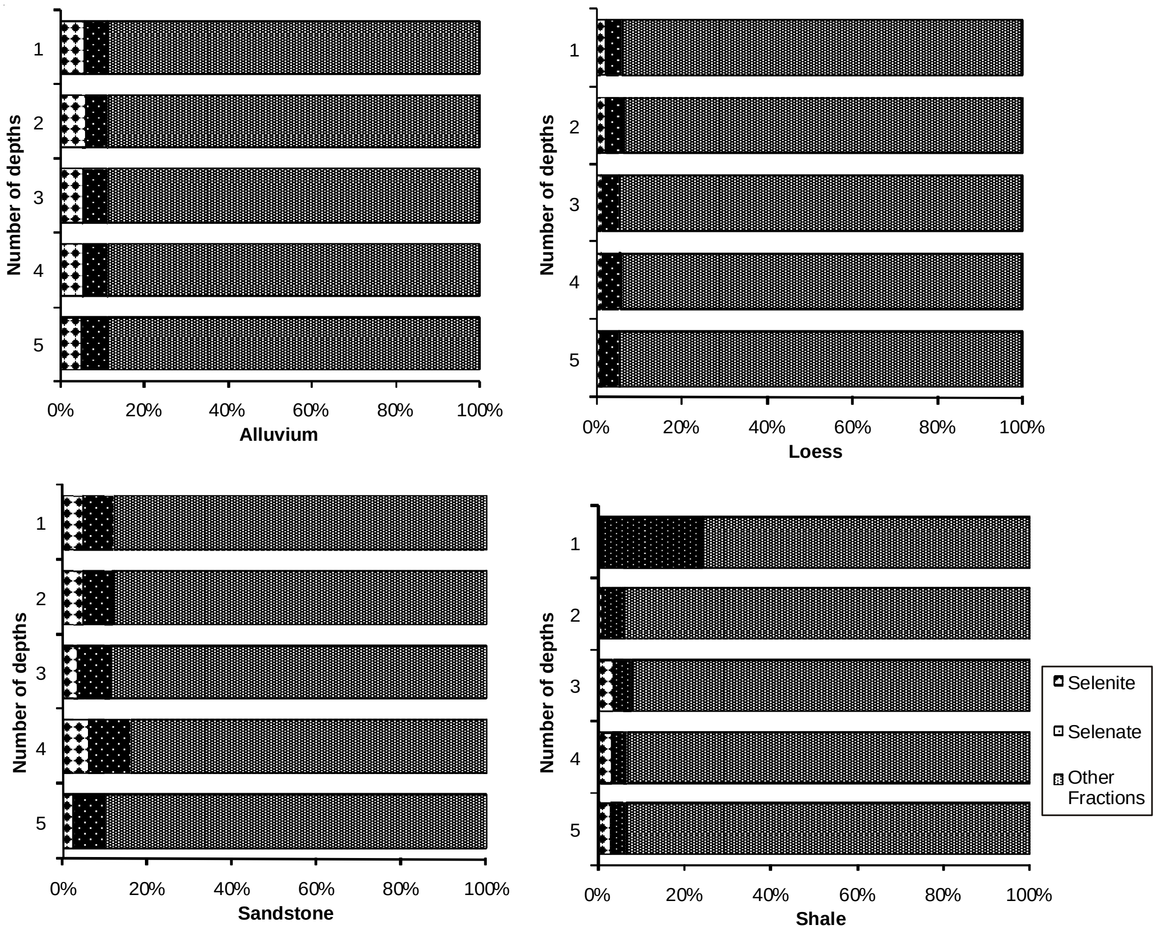

Fig. 5. Selenate and selenite percentage of total selenium in different parent materials

ACKNOWLEDGEMENTS

The authors thank to Higher Education Commission of Pakistan for financial assistance through Indigenous Scholarship Program as well as International Research Support Initiative program.

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