Suspended sediment concentration and particle size distribution, and their relationship with heavy metal content

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This paper aims at assessing the feasibility of suspended sediment concentration (SSC) estimation by using predictor variables of heavy metal concentration (HMC, viz., iron, chromium, zinc and nickel) transported in solution and solid. The study was conducted in the Research and Educational Forest Watershed of the Tarbiat Modares University (Kojour) which comprises an area of ca. 50000 ha. For this study, suspended sediment samples were collected from the left bank of the Kojour River twice a week, as well as during runoff events from November 2007 to June 2008. The samples were then prepared through direct digestion and finally analyzed by atomic absorption spectrophotometry (AAS). The relationship between SSC and particle size distribution (PSD) were correlated with HMC by using bivariate and multivariate regression models. Proposed models were then selected based on statistical criteria. The results showed high correlation between dissolved and particulate chromium content with efficiency coefficients beyond 77% ($P < 0.001$). However, a lower relationship was found between SSC and nickel content. From these results, it is clearly shown that the HMC can practically be estimated by SSC in watersheds with different accuracy and *vice versa*. It is also understood that heavy metal pollution can be easily managed by controlling SSC.

1. Introduction

The sediment transported in streams originates either from the stream channel or from the soil surface in the watershed (Klove 1998; Carter *et al* 2003). Sediment production and delivery are affected by both physical and human factors (DeBoer 1997; Walling 1997; Neal *et al* 1998; Siakeu *et al* 2004; Zhang *et al* 2006; Sadeghi *et al* 2008, 2009). Over the past millennia, humans have had a plethora of impacts on fluvial systems, directly (e.g., by the construction of dams and embankments) or indirectly (e.g., by changes in land cover) (Goudie 2005). Since suspended sediment is a nonpoint source pollutant, whose concentration in natural streams varies rapidly and unpredictably, changes in transport associated with human causes are usually difficult to demonstrate (Lewis 1996). Therefore, without detailed measurements of sediment

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transport in catchments, the various erosion mechanisms responsible for sediment mobilization cannot be identified (Krause *et al* 2003; Chao *et al* 2009).

Knowledge of the processes involved in the generation, transport and deposition of sediment, as well as the associated changes in the particle size characteristics of sediment during erosion, is clearly of fundamental importance to an understanding of the fate of chemicals (Walling and Moorehead 2004; Pacifico *et al* 2007). The particle size distribution (PSD) of the sediment sample may have a significant effect on the concentration of such elements. The presence of trace metals in sediments is influenced by the particle size and composition of the sediments (Horowitz and Elrick 1987; Singh *et al* 2005), since each class of particle size has its own characteristics (e.g., specific surface area, shape, minerals and source) and consequent interaction with heavy metal concentrations (HMCs). Recent studies have shown a growing awareness of the wider environmental significance of the suspended sediment loads transported by rivers and streams. This includes the importance of fine grain sediment in the transport of nutrients and contaminants, such as phosphorus, pesticides, heavy metals and pathogens through fluvial systems (Carter *et al* 2003; Warren *et al* 2003; Walling and Moorehead 2004; Singh *et al* 2005; Walling 2005; Rovira and Batalla 2006; Iadanza and Napolitano 2006; Butler *et al* 2008; Chao *et al* 2009).

Numerous studies have demonstrated that the HMCs in suspended and bed sediments can be sensitive indicators of contaminants in hydrological systems (Jain and Sharma 2001; Chao *et al* 2009). In addition, some studies (e.g., Salomons and Forstner 1980; Collins *et al* 1997, 1998; Dessouki *et al* 2005; Walling 2005; Koroluk and De Boer 2007; Kamala-Kannan *et al* 2008; Chao *et al* 2009) have also attempted to quantify the sources of suspended sediment transported in river systems using heavy metals.

To the author's knowledge, there was no comprehensive research on the estimation of HMC using SSC particularly in developing countries. The aim of the present study conducted in Research and Educational Forest Watershed of Tarbiat Modares University (Kojour) was to assess the feasibility of HMCs estimation using suspended sediment concentration (SSC). Iron (Fe), zinc (Zn), chromium (Cr) and nickel (Ni) were selected for the study based on their natural presence in soils due to the weathering of their parent materials (Hardy and Cornu 2006). It was then hypothesized that the HMCs can be estimated by knowing SSC.

2. Experimental/materials and methods

The Research and Educational Forest Watershed of the Tarbiat Modares University is a part of central Alborz forest study zone, which is located in the southeastern part of the Nowshahr drains an area of ca. 50000 ha (figure 1) to the Caspian Sea. The watershed is mainly covered by forest stands with an average density cover of more than 75%. Elevation ranges from some 200 to 2650 m above mean sea level. More than 90% of the geologic formations belong to the second geological era which mainly contains limestone. Maximum and minimum temperatures, and mean annual precipitation from the data recorded between 1977 and 2007 are 19.9°C, 13°C and 1287.8 mm, respectively. The maximal and minimal monthly rainfall is also 280.4 and 37.4 mm. The stated meteorological variables decrease as elevation increases so that the mean annual precipitation at Kojour station located at the topmost point of the study watershed declines by about 250 mm. The region including the study site has humid and semi-humid climates in the northern and southern parts of the watershed, respectively. The value of SSC in the study watershed revolved from 0.025 to 110.07 g/l, whereas discharge variability for the same period was in range of 0.06 to 2.82 m^3/s with an average value of 0.44 m^3/s (Sadeghi and Saeidi 2010; Sadeghi *et al* 2010).

2.1 *Field methods and sample collection*

In order to conduct the study, 65 suspended sediment samples were collected manually from left bank of the Kojour River semi-weekly from November 2007 to July 2008 and during storm events with 1-hr interval (Achite and Ouillon 2007). SSCs were typically in the range of 0.08–42.75 g/l for the entire study period. Major factors affecting the mobility of heavy metals in rivers include pH (Wen and Allen 1999) and water temperatures, which were simultaneously measured. All samples were analyzed according to the Standard Methods for the Examination of Water and Wastewater (American Public Health Association 1998). SSCs were obtained through water sampling in a 2000-ml plastic vessel using depth-integrating procedures (Edwards and Glysson 1999; Rovira and Batalla 2006). All plastic vessels in contact with samples were first cleaned with a diluted nitrate detergent (Singh *et al* 2005; Fernández *et al* 2008), rinsed with distilled water several times, and finally dried in an oven for 24 h at 70°C. The samples with the least volume of 1000 ml were then analysed at the research laboratory of Tarbiat Modares

University located at about 30 km from the study watershed, in Noor City (figure 1). The SSCs were determined by filtration of the samples through Whatman filters (Klove 1998).

The filtrate (Whatman 42, Germany) were dried for 24 h to a constant weight (Sadeghi *et al* 2006). The SSC in the known volume of filtered water was ultimately determined using the weight of the dried filtrate and volume of the sample. Supernatant was acidified with several drops of HNO_3 solution to pH < 2 (Jain and Sharma 2001; Singh *et al* 2005) for sample filtration.

The filters were then digested at 90°C for a minimum of 4 h with 15 ml of concentrated 2:1 HNO_3/HCl solution. Hydrochloric acid (33% w/v) and nitric acid (67% w/v) were used as suprapure reagents (Scharlou). Samples for analysis of dissolved metals were consequently filtered through Whatman 42 filters. The samples were kept in 25-ml polypropylene bottles for analysis of heavy metals using AAS Model Philips, PU9400, USA (Amini *et al* 2008), calibrated with standard metal solution. Standard solutions were prepared by diluting a 1000 mg/l element solution with the same acid mixture used for sample dissolution. Glassware were cleaned by soaking in a 10% (w/v) nitric acid solution for a while and then rinsed with deionized water (Bettinelli *et al* 2000).

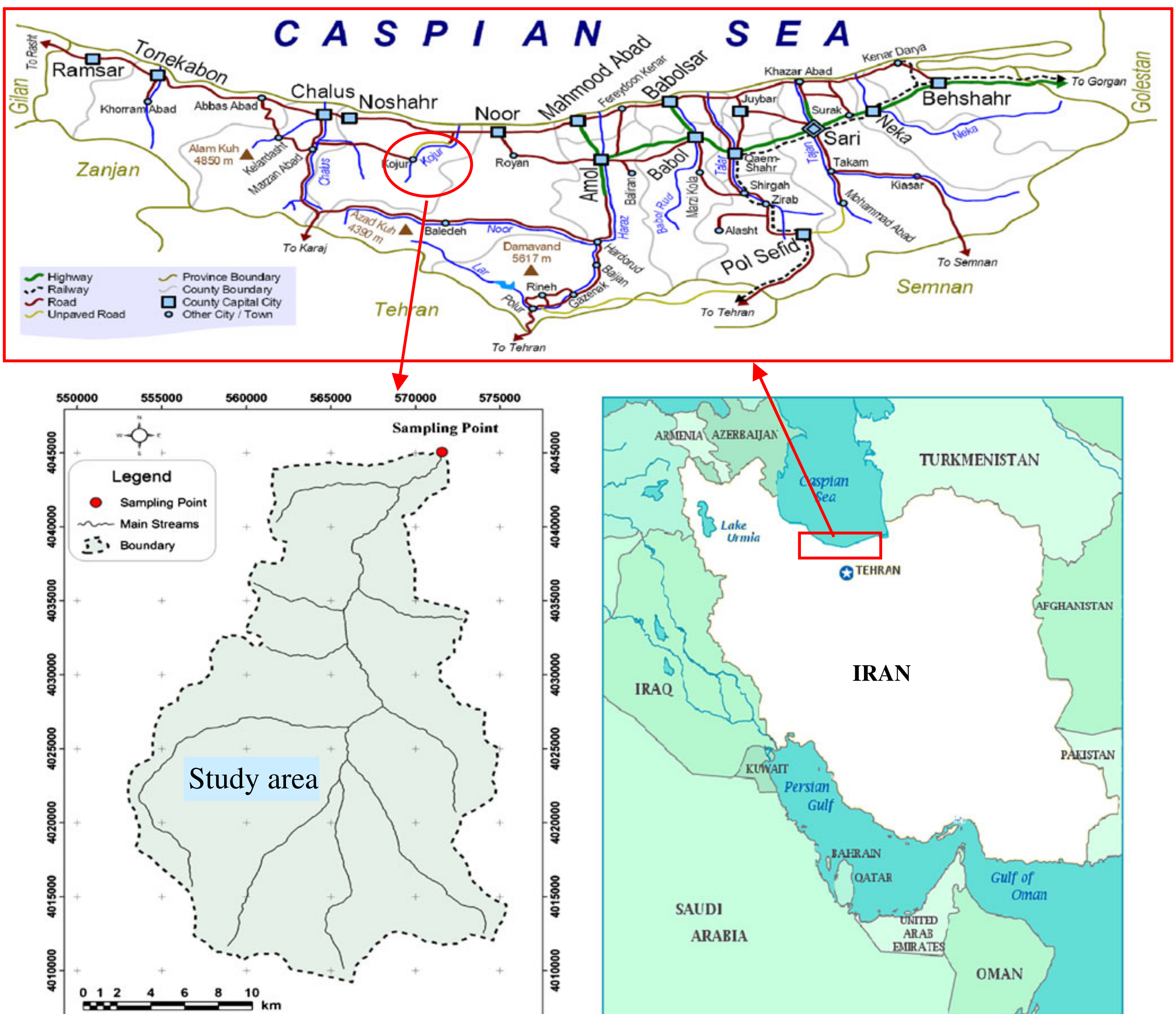

Figure 1. General feature and location of the Research and Educational Forest Watershed of Tarbiat Modares University and sediment sampling site.

2.2 *Statistical analysis*

The data bank development for discharge, SSC and HMCs and statistical calculations were supported by EXCEL 2003 and SPSS 13.5 software packages. The corresponding relationship between SSCs (g/l) as criterion variables and PSD (%) as predictor variable were thus correlated to HMCs (mg/g and/or μg/g) as dual role variables by applying bivariate and multivariate regression models.

In order to determine the best-performed SSC–HMC and HMC–PSD rating curve, different regression fitting procedures were applied to the entire datasets collected at the study station. Both the linear and the non-linear models using ordinary and transformed data, viz., square root, inverse square root, third to fifth root, as well as their combination were also applied.

Different statistical criteria, viz., correlation coefficient (r), significant level of p-value, standard error of estimate were then respectively applied to the experimental data to evaluate the fitness, soundness and reasonability of the regression models. The reliability of the developed models was then checked using relative error (RE) and root mean square of error (RMSE) with the help of following equations:

$$\mathrm{RE} = \left|\frac{Y_o - Y_E}{Y_o}\right| \times 100 \tag{1}$$

$$\mathrm{RMSE} = \sqrt{\frac{\sum_{i=1}^{N} (Y_o - Y_E)^2}{N}}, \tag{2}$$

where Y_o is the quantity of observed SSC or HMC, Y_E is the quantity of estimated SSC or HMC and N is the number of data. A 1% level of significance for coefficient of determination, RE less than 40%, and RMSE tends to zero (Wang and Liu 2006; Achite and Ouillon 2007; Winter 2007; Sadeghi *et al* 2008; Ulses *et al* 2008); were used as evaluation criteria.

3. Results and discussions

3.1 *Descriptive statistics*

During the study period, 65 samples including base flow and flood conditions, were collected from the main river reach of the Research and Educational Forest Watershed of Tarbiat Modares University and then analysed. The analysis of SSCs of the collected samples in the study watershed provides insight into the characteristics of the suspended sediment load variability in the watershed. The descriptive statistics of SSCs, flow discharge, temperature and pH, and HMCs for the study station (figure 1) have been summarized in table 1. The variation of mean daily flow discharges have also been depicted in figure 2. The mean values of SSC for the collected data are about 0.29 g/l and 10.61 g/l for the entire watershed in the base flow and flood datasets, respectively (table 2). As can be seen from table 1, there are some variations in the heavy metal contents of the particulate Fe, Zn and Cr, which decrease during flood conditions compared to those recorded for base flow conditions. It may be due to the low pH as particulate reported by Wen and Allen (1999), which causes metals to be more soluble. It can also be inferred that a fraction of the particulate Fe, Zn and Cr released from the suspended sediment dissolves in water, but no systematic variation is detected which agrees with Gikas (2008), who reported similar finding in connection with Ni. The hydroxylated Ni can be formed in appreciable amounts only at pH above

Table 1. *Descriptive statistics of some study variables in the Research and Educational Forest Watershed of the Tarbiat Modares University, Iran.*

Variables	Periods of study								
	Whole			Base flow condition			Flood events		
	Max.	Mean±SD	Min.	Max.	Mean±SD	Min.	Max.	Mean±SD	Min.
SSC (g/l)	42.75	2.67 ± 7.49	0.08	1.14	0.29 ± 0.25	0.08	42.75	10.61 ± 12.99	0.10
Discharge (m^3/s)	1.60	0.59 ± 0.41	0.04	1.60	0.57 ± 0.42	0.04	1.27	0.62 ± 0.42	0.04
Temperature (°C)	28.00	14.04 ± 6.25	2.00	28.00	14.38 ± 6.56	2.00	20.00	12.98 ± 5.25	4.00
pH	9.14	8.41 ± 0.27	7.80	9.14	8.41 ± 0.29	7.80	8.90	8.42 ± 0.20	8.17
Fe content in water (mg/l)	2.69	0.55 ± 0.47	0.09	1.44	0.42 ± 0.27	0.09	2.69	0.98 ± 0.71	0.14
Fe content in SSC (mg/g)	38.83	14.23 ± 9.21	1.26	38.83	13.43 ± 9.50	1.44	27.09	16.89 ± 7.56	1.26
Zn content in SSC (µg/g)	39.94	12.43 ± 0.01	4.05	39.94	14.17 ± 0.01	5.03	19.01	9.08 ± 0.04	4.05
Cr content in SSC (µg/g)	541.18	83.88 ± 0.07	10.99	541.18	94.31 ± 0.08	10.99	69.35	49.08 ± 0.01	34.52
Ni content in SSC (µg/g)	91.46	37.74 ± 0.01	4.31	91.46	38.13 ± 0.01	4.31	69.74	36.43 ± 0.01	24.27
No. of samples	65			50			15		

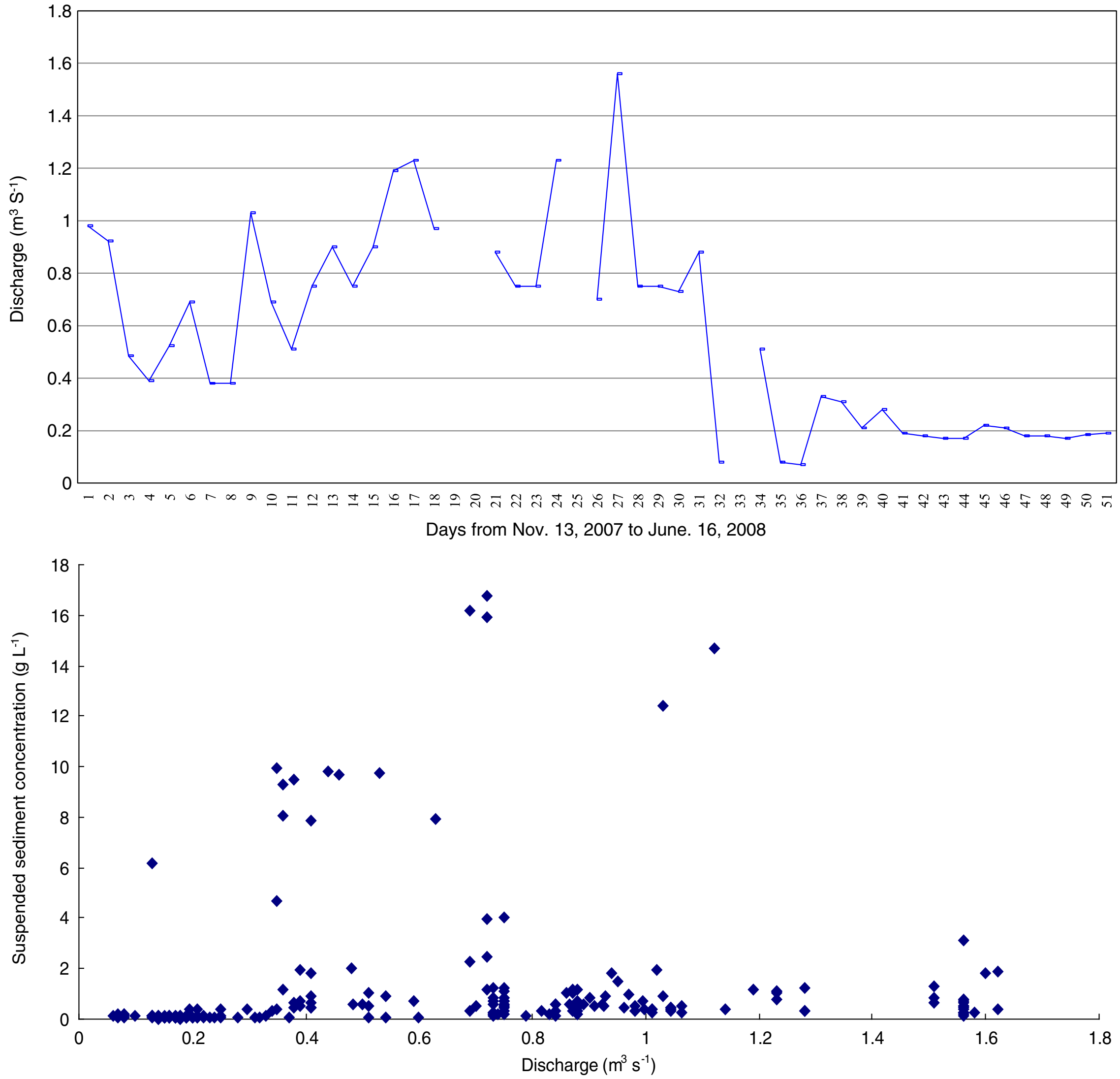

Figure 2. Variation of mean daily flow discharges during the study period (above) and scatter plot of flow discharge and suspended sediment concentration relationship (Sadeghi and Saeidi 2010) (bottom) in Educational and Research Forest Watershed of Tarbiat Modares University.

9.5. It is also understood that due to the neutral to alkaline nature of the river water, most of the heavy metals can be precipitated and settled as carbonates, oxides, and hydroxide-bearing sediments as reported by Singh *et al* (2005). The high values of pH and corresponding variations under different hydrological conditions can be attributed to the alkaline geologic formations belonging to the second geological era and mostly covers the watershed area which contributes to runoff generation, differently.

3.2 *SSC–HMC relationships in base and flood runoffs*

The initial results of the application of different types of SSC–HMC relationships in the Research and Educational Forest Watershed of the Tarbiat Modares University are demonstrated in table 2. It is obvious from the table that almost all different types of models established good SSC–HMC relationships with respect to correlation coefficient criterion. However, other criteria were also considered in order to determine the best-performed model. The model with higher correlation coefficient, lower level of significance (p-value), standard error of estimation, estimation and absolute errors and RMSE was supposed to be a better-performed model (Wang and Liu 2006; Achite and Ouillon 2007; Winter 2007; Sadeghi *et al* 2008; Ulses *et al* 2008; Sadeghi and Saeidi 2010). The linear, logarithmic, quadratic, cubic, s-shape and growth type equations were eliminated at very early stages of the analysis due to large values of estimation errors, even though some of them had resulted in

Table 2. *Optimal relationships established between SSC and HMC under different conditions in the Research and Educational Forest Watershed of the Tarbiat Modares University, Iran.*

	Data	n	Equation	r	p-value	Std. error of estimation	Error (%) Estimation	Error (%) Absolute	RMSE
SSC (g/l)–Fe content	All base flow	6	$SSC = e^{-2.15+(2.26HMC)}$	0.73	0.00	1.12	78.56	82.95	0.13
in water (mg/l)		7	$SSC = e^{-2.21+(1.59HMC)}$	0.58	0.00	0.65	50.16	32.69	4.59
	Flood runoff	–	–	–	–	–	–	–	–
SSC (g/l)–Fe content	All base flow	8	$SSC = 0.07HMC^{0.72}$	0.43	0.00	1.48	127.32	85.80	0.13
in SSC (mg/g)		9	$SSC = 0.09HMC^{0.37}$	0.45	0.01	0.72	57.92	44.93	3.84
	Flood runoff	10	$SSC = 0.18(1.19^{HNMC})$	0.75	0.01	1.27	147.28	103.20	0.06
SSC (g/l)–Zn content	All base flow	11	$SSC = 0.04HMC^{-0.88}$	0.35	0.02	1.54	164.15	133.20	0.13
in SSC (mg/g)		–	–	–	–	–	–	–	–
	Flood runoff	12	$SSC = e^{-3.59+(0.25/HMC)}$	0.77	0.01	1.22	195.94	708.65	0.22
SSC (g/l)–Cr content	All base flow	13	$SSC = e^{1.19+(-101.81HMC)}$	0.53	0.01	1.49	136.42	188.92	0.10
in SSC (mg/g)		14	$SSC = e^{-0.18+(-69.33HMC)}$	0.77	0.00	0.55	41.61	50.24	3.94
	Flood runoff	–	–	–	–	–	–	–	–
SSC (g/l)–Ni content	All base flow	–	–	–	–	–	–	–	–
in SSC (mg/g)		15	$SSC = 0.02HMC^{-0.72}$	0.48	0.00	0.70	58.66	71.29	3.78
	Flood runoff	–	–	–	–	–	–	–	–

Table 3. *Optimal relationships established between heavy metals concentration (HMC) and particle size distribution (PSD) of suspended sediment in base and flood runoffs in the Research and Educational Forest Watershed of the Tarbiat Modares University.*

	Data	n	Equation	r	p-value	Std. error of estimation	Error (%) Estimation	Error (%) Absolute	RMSE
PSD (silt%)–Fe content	All base flow	16	$HMC = 0.24PSD^{0.49}$	0.78	0.00	0.53	44.96	53.82	2.91
in water (mg/l)		17	$HMC = 0.24PSD^{0.44}$	0.69	0.00	0.53	46.23	37.97	4.28
	Flood runoff	18	$HMC = 0.22PSD^{0.51}$	0.84	0.01	0.63	49.63	57.63	1.67
PSD (silt%)–Fe content	All base flow	19	$HMC = 6.63PSD^{0.46}$	0.66	0.00	0.71	66.86	67.35	0.12
in SSC (mg/g)		20	$HMC = 5.81PSD^{0.53}$	0.65	0.00	0.72	68.71	47.41	0.12
	Flood runoff	21	$HMC = 5.11PSD^{0.47}$	0.79	0.01	0.70	61.18	35.32	0.06
PSD (clay%)–Zn content	All base flow	22	$HMC = 0.31PSD^{-0.57}$	0.39	0.02	0.51	38.32	36.77	21.29
in SSC (mg/g)		–	–	–	–	–	–	–	–
	Flood runoff	–	–	–	–	–	–	–	–
PSD (silt%)–Cr content	All base flow	–	–	–	–	–	–	–	–
in SSC (mg/g)		23	$HMC = 1.01 + 0.01/e^{PSD}$	0.51	0.04	0.008	0.51	0.42	121.24
	Flood runoff	–	–	–	–	–	–	–	–
PSD–Ni content	All base flow	–	–	–	–	–	–	–	–
in SSC (mg/g)		–	–	–	–	–	–	–	–
	Flood runoff	–	–	–	–	–	–	–	–

maximum correlation coefficients in the earlier step (Sadeghi *et al* 2008). Additionally, high correlations ($r > 0.73$, $P < 0.001$) were also established between dissolved iron and SSC for the whole study period. High correlations were also established between particulate chromium and dissolved iron with SSC; the respective correlation coefficients were greater than 0.77 and 0.58 ($P < 0.001$) during base flow conditions. Ultimately, the highest correlation was also established between particulate iron and zinc content with SSC, the correlation coefficients were greater than 0.75 and 0.77 ($P < 0.01$) during flood conditions with large values of estimation and verification errors. It clearly verified the existing uncertainties in the sytem. The weakest relationship was found between SSC and nickel content. Despite many researches verifying the relationship between bed load and HMC (Chen *et al* 2007; Koroluk and de Boer 2007; Zhang *et al* 2007), no distinct literature addressed the SSC–HMC relationship and therefore no comparison could be made. The variations of HMCs at different depths of the soils and in connection with a range of effects of different types of soil erosion (i.e., sheet, rill and gully) may be considered as influencing factors on SSC–HMCs relationship. This also clearly emphasizes the necessity for considering PSD of the SSC in controlling the transportation of HMCs. These findings are partially or completely supported by earlier literature (Carter *et al* 2003; Krause *et al* 2003; Warren *et al* 2003; Walling and Moorehead 2004; Singh *et al* 2005; Walling 2005; Rovira and Batalla 2006; Iadanza and Napolitano 2006; Butler *et al* 2008; Chao *et al* 2009).

3.3 *Suspended sediment PSD–HMC relationship in base and flood runoffs*

Since the knowledge of particle size and the size distribution of suspended sediment is a pre-requisite for understanding transport, sedimentation and control processes (Jillavenkatesa *et al* 2001), the relationship between HMC and PSD of SSC in base flow and flood conditions have also been studied in this study and the corresponding results have been presented in table 3. From table 3, higher correlations were also established between dissolved iron content and silt percentage with correlation coefficients more than 0.69, 0.78 and 0.84 ($P < 0.01$) in base flow, entire and flood runoff datasets, respectively. High correlations were also established between particulate iron and silt percentage with correlation coefficient greater than 0.79 ($P < 0.01$), 0.65 ($P < 0.05$) and 0.66 ($P < 0.01$) in flood runoff, base flow and whole datasets, respectively. High correlations were established between particulate zinc and clay percentage ($r = 0.39$, $P < 0.05$) for the entire dataset period of the study, only. Ultimately, the highest correlation was established between particulate chromium and silt percentage ($r = 0.51$, $P < 0.05$) in base flow condition. No distinct relationship was detected between nickel and PSD. The variation of contributing areas in generation of runoff during base flow and flood conditions, changes in rainfall centroid, as well as changeability of mechanisms in soil erosion due to different types of runoff can be regarded as acceptable reasons for controlling SSC–HMCs relationships (Sadeghi and Saeidi 2010; Sadeghi *et al* 2010). Investigating the relationship between HMC and PSD verified the highest correlation coefficient between HMC and silt percentage due to its easy, high surface specific area and rapid transportation (Singh *et al* 2005), which is in agreement with other studies (Taylor 1996; Martin 2004) that reported weak correlation between HMC and clay percentage. It can also be attributed to the high variability of silt content with a coefficient of variation of 140.8% compared to those reported for sand and clay as 8.9 and 24.4, respectively. It is then accepted that fine-grained suspended (silt and clay with particle size <63 μm) accumulate greater concentrations of contaminants (particularly those with low water solubility) than coarse particles (particle size >63 μm) as mentioned by Forstner (2004). It is finally understood from regression models (tables 1 and 2) that the equations developed among study variables are often non-linear. It clearly verifies the general tendency of hydrological variables relationships towards non-linearity as reported by Singh (1992) and Sadeghi *et al* (2008).

4. Conclusion

The transport rate of suspended sediment is of considerable interest in studying watershed hydrology. The present research was conducted to assess the feasibility of estimation of heavy metal (Fe, Zn, Cr and Ni) content using suspended sediment in base and flood runoffs from the Research and Educational Forest Watershed of the Tarbiat Modares University, northern Iran. The results of this study have proved the non-linear reliable regression models between HMC and SSCs. The highest correlation was established between chromium content in water and SSC. Meanwhile, the weakest relationship was found between SSC and nickel content. The present study has achieved a reasonable interaction between SSC and HMC under consideration. However, there is clearly a need to further investigate the relationship between SSC and other HMC and to evaluate the accuracy of measurement by other methods in the same watershed. Further research is required to evaluate the applicability of developed models in other watersheds to draw final conclusions.

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