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Sediment contribution from different geologic formations and land uses in an Iranian small watershed, case study

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ABSTRACT

Many intra and extra problems occurred due to unsustainable human use of natural resources leading to increasing sediment loads in the watersheds. However, few studies have been comprehensively conducted in progressing countries to prioritize sediment sources from different points of views, particularly in some countries like Iran where such valuable information is essential for proper watershed resources management. The present study was therefore planned to assess the importance of potential sediment sources viz., spatial sources (geologic units) and source types (land use units) in sediment yield in Idelo watershed as one of the important sub-watersheds of Sefidrood large Watershed in Zanjan Province, Iran, using composite fingerprinting. In addition, the results of the sediment fingerprinting approach were compared with those of field measurement data obtained from studying soil erosion types (viz., sheet, rill and gully erosion). Toward this attempt, 16 tracers were detected in different geologic units and land uses and the sediment yielded at the watershed outlet. The results showed that the composite fingerprints of the different geologic units comprising As, N, Cu, Zn, OC and Co tracers could correctly distinguish 86% of the sediment source samples. The red gypsiferous marl contributed 85 percent in sediment yield. In regard to source types, the optimum composite fingerprint encompassed only N and Cu and provided a discriminatory efficiency of 90%. Besides that, the rangelands with 48.8% study area coverage had a significant contribution of 88% in sediment yield. The field measurements confirmed the reliability of results of fingerprinting approach in apportioning watershed scale sediment sources on the base of consistency of the two sets of results. It was also understood from the results, besides successful applicability of composite fingerprinting in assessing the provenance of the sediment yielded at the watershed outlet that the geologic formations and land use types played different roles in sediment yield. Such information helps managers and decision makers to properly regulate appropriate and adaptive management approaches in the study watershed.

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1. Introduction

Concern for the impact of accelerated rates of soil erosion and sediment yields on watersheds, resulting from land clearance and poor land management, has generally divided on their effects in terms of on and off site problems. In many areas of the world, particularly in developing countries, control of soil erosion, sediment delivery to watercourses is seen as being of great importance in reducing diffuse sediment and dealing with aforesaid problems. During few last decades, the soil and water resources of Iran have faced some serious degradation problems mainly due to soil

erosion (Davudirad et al., 2016; Sadeghi & Hazbavi, 2015; Sadeghi & Tavangar, 2015). In response to these sediment-related environmental and watershed management problems, the design of effective management strategies to achieve meaningful reductions in sediment loads and yields is required because such strategies should target the key sources, if sediment fluxes are to be significantly reduced (Adhami & Sadeghi, 2016; Jenns et al., 2002). But, targeting sediment management and control policies are significantly constrained by the scarcity detailed information on both the nature and relative importance of the primary sediment sources within a catchment (Slimane et al., 2016; Walling et al., 2008). Traditional techniques for assembling such information involve many operational problems and sampling constraints. The fingerprinting technique, as the direct method, was accordingly developed to overcome problems mentioned above. It offers an

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alternative approach which has been increasingly employed to identify the relative importance of potential catchment sediment sources and avoids many of these problems (Aksoy et al., 2016; Buendia et al., 2016; Collins & Walling, 2007; Collins et al., 1997a, 1997b, 1997c; Walling, 2005; Walling & Woodward, 1995; Zhao et al., 2016). The problems are much complicated in developing countries due to population growth and increasing demand for food, economic dependence on primary production and chaotic urban growth causing widespread land degradation (Adhami & Sadeghi, 2016; Davudirad et al., 2016), while due to the financial constraints, there is a need to focus conservation and controlling measures on key sources.

The application of sediment fingerprinting approach has been founded on some main assumptions. First, that potential sediment sources can be discriminated on the basis of measurements of their diagnostic properties of fingerprints. Second, a comparison of suspended sediment and catchment source samples in order to determine sediment provenance is applicable using these properties with multivariate mixing model (Collins & Walling, 2007; Collins et al., 2001, 2010a; Walling et al., 1999). In addition, the use of sediment source fingerprinting techniques necessarily assumes that the sediment tracers used behave conservatively within the fluvial system and also that the tracers of source material and sediment samples can be directly compared. It is often difficult to confirm the conservative behavior of tracers directly, but, in many studies it is emphasized that for successful modelling, the property values of the suspended sediment samples must fall within the range of behavior displayed by the values chosen to represent the sources. The accuracy of the methodology can also be influenced by errors in source definition, chemical alteration during transport or size selective transfer of different sources. Enrichment and depletion effects associated with the grain size composition and organic matter content caused by selective mobilization and transport can be viewed as a form of nonconservative behavior. A number of different approaches have been employed to deal with this potential problem. These include, first, processing both source material and sediment samples to focus on the same grain-size fraction, and, second, use of correction factors to take account of contrasts in organic matter content and grain-size composition (Mukundan et al., 2012; Walden et al., 1997). On these bases, many pioneer researches have traditionally used single component signatures, encompass mineralogy (Klages & Hsieh, 1975; Wood, 1978), color (Grimshaw & Lewin, 1980; Martinez-Carreras et al., 2010), mineral magnetic (Hatfield & Maher, 2009; Walden et al., 1997; Wang et al., 2011), fallout radionuclides (Olley et al., 1993; Owens et al., 1999; Wallbrink et al., 1998) and organic matter (Santiago et al., 1992) properties. But using single property or component signature is likely to prove unrealistic and spurious linkage between source materials and sediment (Collins & Walling, 2002), so most fingerprinting studies now employ composite fingerprint.

Previous fingerprinting studies were significantly different in several respects comprising size of the studied areas, sampling design, classification of sediment sources, method and component of analyses. The sizes of the studied areas have encompassed from small sizes about 0.5–2 km² (Li et al., 2003; Mizugaki et al., 2008; Walling, 2005) to thousands of km² (Collins et al., 1996, 1997b; Juracek & Ziegler, 2009; Wallbrink et al., 1998; Walling et al., 1999). Sampling designs also varied in several aspects. For instance, depth of sampling ranged from < 1 cm (Motha et al., 2003; Wallbrink et al., 1998) to 5 or 10 cm (Gruszowski et al., 2003; Juracek & Ziegler, 2009) but it was generally < 2 cm for surface materials (Collins et al., 2010a, 2010b, 2010c, 2010d; Walling, 2005; Walling et al., 2008) and exceeded up to 30 cm for subsurface materials (Gruszowski et al., 2003). The number of samples per sediment source reported from less than 10 to 50 samples per

source (Collins & Walling, 2002; Collins et al., 2010a; Najafi & Sadeghi, 2013; Walling, 2005) emphasizing on 15 samples for using multivariate Discriminant Function Analysis (DFA) (Hair et al., 2010). In some studies (e.g. Collins et al., 1996; Walling et al., 1999; Zhang et al., 2012), the spatial location of the main sediment sources has been designated within a drainage watershed, while in others, relative importance of different categories of potential sources, such as individual source types including surface soils from areas of different land use and channel banks (Russell et al., 2001; Walden et al., 1997), unmetalled and damaged roads (Collins et al., 2010b; Gruszowski et al., 2003), geologic zones (Walling et al., 1999; Walling & Woodward, 1995) and land uses (Owens et al., 1999) has been taken into account. A modified mass balance model incorporating the Monte Carlo approach and genetic algorithm were also applied for representing the uncertainty surrounding source and sediment sampling, taking account of the within-source variability and discriminatory power of individual tracer by Collins et al. (2010a, 2010b, 2010c, 2010d, 2013), Haddadchi et al. (2014) and Stone et al. (2014). Sadeghi et al. (2014) also successfully quantified the contribution of potential sediment sources, i.e., sheet, rill and gully erosion, in Idelo watershed in Zanjan Province, Iran, using composite fingerprinting. They reported that sheet, rill and gully erosions contributed some 56%, 44% and 0% in sediment yield from the study area, respectively. It therefore showed that gully erosion had the least contribution in sediment yield in the study watershed. It might be due to controlling effects of different types of check dams viz., gabions, wire and brush constructed to establish gullies. Recently, Jaramillo et al. (2016) measured bed and suspended loads on the river system to determine the impact of a 20-ha limestone quarry as sediment source. They reported that the quarry stood to make a disproportionately large contribution ($\approx 92\%$) to suspended sediment load at the outlet. The contribution of bed load sediments from the quarry was also estimated $> 75\%$ at a section located 1.2 km downstream.

There are limited comparative studies on designation of contribution of different sediment sources in sediment yield using fingerprinting techniques and direct field measurement according soil erosion types, particularly in developing countries like Iran where such valuable information is essential for proper watershed resources management. This research has been therefore conducted for Idelo watershed as one of the important sub-watersheds of Sefidrood large Watershed, Iran, to distinguish key sediment source in the base of different spatial sources including geologic units and different land uses in sediment yielded at the outlet of the watershed using fingerprinting technique. The objectives of this study were therefore to (1) determine which discriminative properties could differentiate the sediment sources in the study area; (2) identify important geologic and land uses source types in sediment yield of the study watershed and (3) compare results of the sediment fingerprinting approach with field measurement data. Such types of intensive field and lab works have been rarely conducted in field of sediment yield study in developing countries like Iran where drawing a valuable baseline for designating proper management of soil erosion and land management are essentially required.

2. Materials and methods

2.1. Study watershed

The Idelo is one of the main watersheds drains into the Sefidrood large watershed located in northwestern Iran. It comprises an area of about 20 km² underlined by Tertiary and Quaternary geologic formations which divided to three main

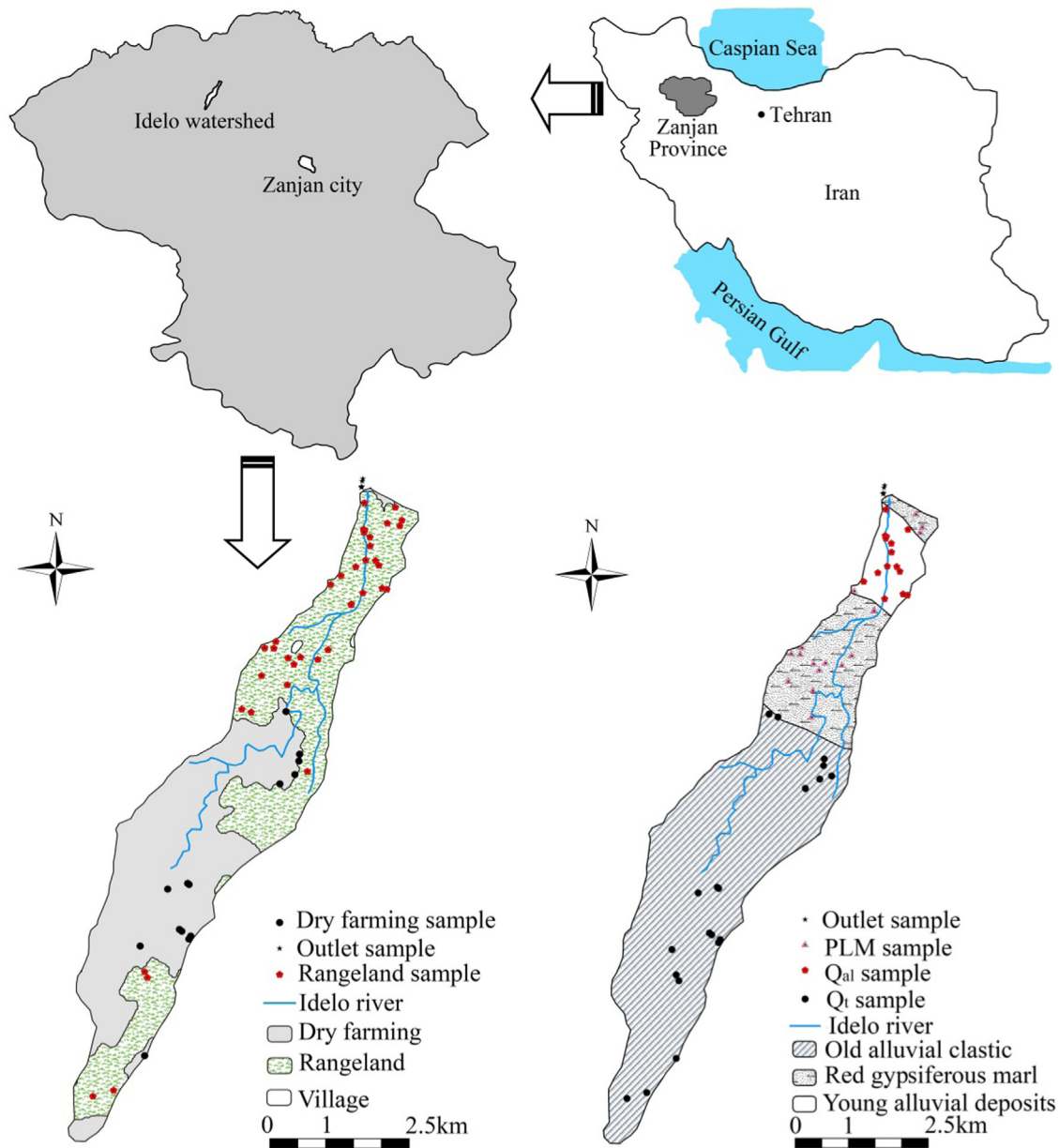


Fig. 1. The location of the study watershed in Zanzan Province and Iran bottom, geologic map (upper right) and land use (upper left).

geologic units namely Red gypsiferous marl (PLM), old alluvial clastic (Q_t) and young alluvial deposits (Q_{al}) with respective coverage of some 21%, 71% and 8% area. Soils are mainly in Solonchak and Rendzina classes as well as carbonate rocks with sand clay loam texture. The mean annual precipitation is 275 mm. Land use of the watershed is dominated by dry farming (51.2%) and rangeland (48.8%) distributed in southern and northern part of the watershed, respectively. Rangelands are located on red gypsiferous marl and young alluvial deposits areas versus dry farming lands that occupied old alluvial clastic. It has been extended from 1281 m amsl at the outlet to 1699 m amsl in the upstream areas with steep slopes dominantly 15 percent (Najafi & Sadeghi, 2013; Sadeghi et al., 2014). A schematic view of the study watershed has been shown in Fig. 1.

2.2. Sample collection

Field sampling involved the collection of representative samples of the main potential suspended sediment sources identified

based on geologic and land use units. Description of the geomorphologic properties of the sampling points have been shown in Table 1. A total 50 source material samples were systematically collected from the entire of the study watershed including 15, 15 and 20 samples from Red gypsiferous marl, young alluvial deposits (with rangelands) and old clastic (with dry farming land use), respectively. The mixed samples were taken from upper 2, 2–30 and below 30 cm depths. Aforesaid depths represent sheet, rill and gully erosions, respectively (Gruszowski et al., 2003; Sadeghi et al., 2014) for all geologic and land use units. The spatial distribution of the sampling points in the study watershed was designated based on variability of slope steepness, extension and severity of different types of soil erosion and different geologic formations. In addition, a range of locations was ultimately sampled within the watershed to represent areas with good connectivity to the watercourse system (Collins et al., 2010a, 2010b; Walling et al., 2006) to minimize the uncertainty. Accordingly, most of the sampling points have been distributed in the northern half of the study watershed. All samples were collected using a stainless steel

Table 1
Geomorphology properties of the sampling points in different Geologic units.

Red gypsiferous marl (PLM)					Old alluvial clastic (Q_2)					Young alluvial deposits (Q_{a1})				
Sample number	Slope (%)	Type of erosion	Distance to the river channel	Distance to the outlet	Sample number	Slope (%)	Type of erosion	Distance to the river channel	Distance to the outlet	Sample number	Slope (%)	Type of erosion	Distance to the river channel	Distance to the outlet
1	0–5	Sheet	602	849	1	0–5	Sheet	120	5272	1	0–5	Sheet	20	701
2	0–5	Sheet	640	809	2	0–5	Sheet	3948	11,955	2	0–5	Sheet	10	742
3	5–15	Sheet	431	3373	3	5–15	Sheet	601	4579	3	5–15	Sheet	380	682
4	5–15	Sheet	126	3002	4	5–15	Sheet	648	4601	4	5–15	Sheet	549	1824
5	0–5	Sheet	500	573	5	5–15	Sheet	500	5620	5	5–15	Sheet	420	1613
6	5–15	Rill	591	3895	6	0–5	Sheet	286	7924	6	5–15	Sheet	160	1268
7	5–15	Rill	494	3417	7	0–5	Sheet	859	8769	7	5–15	Sheet	240	1378
8	5–15	Rill	261	3210	8	5–15	Sheet	1271	9338	8	0–5	Rill	10	710
9	5–15	Rill	80	3228	9	5–15	Sheet	1698	9731	9	5–15	Rill	316	1798
10	0–5	Rill	45	4314	10	5–15	Sheet	1785	9806	10	5–15	Rill	417	1835
11	0–5	Gully	228	3287	11	0–5	Rill	1051	8806	11	5–15	Rill	10	1862
12	5–15	Gully	367	3334	12	0–5	Rill	907	8772	12	0–5	Gully	80	840
13	5–15	Gully	302	3443	13	0–5	Rill	107	8166	13	0–5	Gully	160	1394
14	5–15	Gully	108	3840	14	0–5	Rill	310	7933	14	0–5	Gully	0 ^a	1002
15	5–15	Gully	0 ^a	2098	15	5–15	Rill	3213	11,161	15	5–15	Gully	0 ^a	1260
					16	0–5	Gully	260	5127					
					17	0–5	Gully	252	4991					
					18	0–5	Gully	340	5379					
					19	0–5	Gully	1075	8877					
					20	5–15	Gully	4174	12,214					

^a Indicates samples taken from the river bank.

trowel, which was regularly cleaned to avoid inter sample contamination (Collins & Walling, 2002). Attention was further taken to ensure that only material prone to mobilization and transportation by erosion was collected. Furthermore, since there was no human-made at the end of the sub-watersheds and because of impossibility of considering inter/intra variation of fluvial behaviour of the study watershed, just six composite averaged samples were collected from 0 to 20 cm depth of materials deposited at the vicinity of the watershed outlet. The direct field measurements were also organized during 2010–2011 to allow comparing two sets of results. Towards this, 9 unit plots, 12 rills with over 10 m length and 3 gullies were monitored and measured to measure the main portion of entire soil erosion (Sadeghi et al., 2014). To further reduce the uncertainties in sampling processes, the mean sediment contributions from sediment sources bounded by 95% confidence limits were also considered.

2.3. Laboratory analysis

All samples were analyzed for 16 diagnostic properties encompass As, Al, Cd, Co, Cr, Cu, Ni, Pb, Se, V, Zn, Fe, Mn, C, N and total P on the basis of available analytical equipments and successfully used in previous studies to discriminate sediment sources (Collins et al., 1997a; Collins & Walling, 2002; Foster & Walling, 1994; Gruszowski et al., 2003; Walling, 2005). All samples were initially air dried, slowly disaggregated using a pestle and mortar and dry sieved through a 63 μm mesh prior to analysis to ensure sample consistency. The concentration of all metals were determined using Inductively Coupled Plasma Optical Emission Spectrometry, VISTA-PRO model (ICP-OES), after direct and complete digestion with H_2SO_4 , HClO_4 , HNO_3 and HF acid (Marin et al., 2001; Matusiewicz, 2003). Total P was determined using UV–visible spectrophotometry after extraction with perchloric acid. Concentration determination of organic C and N was undertaken using Walkely–Black and Kjeldahl procedures.

2.4. Fingerprinting sediment sources

Before using fingerprinting approach for discriminating sediment sources, all the data checked for univariate and multivariate outliers. Although, the criterion of mean \pm standard deviation is being used to identify univariate outlier data, but according to the nature of geochemical data which are somehow fully scattered, the aforesaid rule could not deliver a relevant threshold estimate for the study variables; because outliers have significant influence on the mean and standard deviation. To reduce the effect of extreme values in estimating the mean and standard deviation, robust scaling has been suggested. In robust scaling, the mean is replaced with median and the standard deviation is replaced with Median Absolute Deviation (MAD) from the median. The sample median and the MAD are simple and easy to compute but nevertheless very useful. MAD was calculated by using Eq. (1) (Hampel, 1974; Rousseeuw & Croux, 1993) in which, x_i and x_{median} denotes, respectively, study variable and median of study variable of data set. The constant of 1.482 in Eq. (1) is needed to make the estimator consistent for the Gaussian (normal) distribution. Their extreme sturdiness makes them ideal for screening the data for outliers. Therefore, the median ± 3 (MAD) criterion was used as a robust approach for detecting outliers based on normal distribution of majority of our data.

$$\text{MAD} = 1.482 \text{ Median} (|x_i - x_{\text{median}}|) \quad (1)$$

The multivariate outliers were also addressed by square Mahalanobis Distance measure (D^2) as a multivariate assessment of each observation across a set of variables. This method measures each observation's distance in multidimensional space from the centroid of all observations, providing a single value for each observation and no matter how many variables are considered. For the detection of outliers, the Mahalanobis D^2 measure was used. The Mahalanobis D^2 divided by the degrees of freedom (df) equal to the number of variables (D^2/df) was considered as a criterion to distinguish outliers. So, observations having a D^2/df value further 2.5 in samples fewer than 80 observations have been designated as possible outliers (Hair et al., 2010; Tabachnick & Fidell, 2007).

Additionally, enough number of samples per category was taken for using multivariate Discriminant Function Analysis (DFA). So that, the minimum ratio of sample size to the number of tracers of approximately five and the least number of 15–20 observations per category were considered (Hair et al., 2010). The two-stage statistical procedure proposed by Collins et al. (1997a) was employed to confirm the discrimination of the potential sediment sources in the watershed. In stage one, all fingerprint properties were tested for their ability to discriminate source types encompass different geologic formations and land uses using the Kruskal–Wallis *H*-test and One-Way ANOVA. The One-Way ANOVA and Kruskal–Wallis *H*-test were used for data with normal and non-normal distribution, respectively (Tabachnick & Fidell, 2007). In next step, DFA was employed to identify composite fingerprints prone to distinguishing spatial sources involving geologic units and different land uses. Application of DFA was based on three assumptions namely normality of independent variables, lack of collinearity among independent variables and equality of group covariance matrices that required to be initially satisfied (Hair et al., 2010). For first assumption, we assessed the univariate and multivariate normality. The Kolmogorov–Smirnov test and normal probability plots were used for normality test with attention that achieving univariate normality of individual variables will many times suffice to reach multivariate normality, as well as DFA is robust to failures of normality if non normality (violation of the assumption) is caused by skewness inverse outliers data (Tabachnick & Fidell, 2007). About the collinearity we used two criteria viz., correlation matrix and tolerance (TOL) or Variance Inflation Factor (VIF). The correlation matrix was used as the simplest means of distinguishing collinearity for the independent variables. Generally, the level of 0.9 and higher of correlation shows the first criterion of significant collinearity. But it does not ensure a lack of collinearity because multicollinearity may be happened due to the combined effect of two or more other independent variables. To overcome aforesaid issue, we used TOL and VIF (Hair et al., 2010; Tabachnick & Fidell, 2007). Tolerance indicates that two or more independent variables are highly explained by the other variables and thus it will not have high explanatory power in fingerprinting (Hair et al., 2010). In other words, tolerance denotes the proportion of the variation in the independent variables not explained by the variables already in the function. A tolerance with 0 value means the independent variable under discussion is a complete linear combination of independent variables already in the function. Generally acceptable levels of TOL and VIF (VIF is TOL inverse) are up to 0.1 and below 10, respectively, which violation these thresholds means multicollinearity among variables (Hair et al., 2010). Finally, equality of group covariance matrices was assessed with Box's *M* test that is the most common test for this assumption. In this approach it is tried to find a nonsignificant probability level which indicates that there are not differences between the group covariance matrices. When the mentioned assumption to be violate, using of the group specific covariance matrices instead total covariance matrices can minimize this effect and a significant level is not regarded as too important (Hair et al., 2010). Composite fingerprints were finally constructed using the square Mahalanobis Distance criteria in stepwise method (Hair et al., 2010). Towards that, the square Mahalanobis Distance criterion was used in order to select the variable with statistical significant difference across groups while maximizing the square Mahalanobis Distance criteria between the other closest groups. In this manner, statistically significant variables were selected to maximize the discrimination between the most similar groups at each stage. The process was continued as long as additional variables provided statistically significant discrimination beyond those differences already accounted for by the variables in the discriminant function.

Table 2
Results of checking univariate outliers using concentration of properties.^a

Sample number	Al	As	Cu	Mn	P	C
1	0.16	1.55	22.35	255.70	15.96	0.774
2	0.27	1.75	24.49	276.54	10.92	0.052
3	0.23	1.24	19.82	279.83	0.00	0.113
4	0.21	0.71	18.69	276.64	7.56	0.258
5	0.30	1.84	23.32	458.14	8.40	0.162
6	0.20	1.58	19.25	392.68	7.98	0.052
7	0.14	1.42	15.35	226.90	4.62	0.144
8	0.36	1.62	24.17	286.79	0.00	0.165
9	0.11	1.80	16.70	180.75	7.14	0.000
10	0.15	1.24	16.17	259.64	13.02	0.340
11	0.42	2.01	23.29	309.06	2.94	0.475
12	0.30	1.15	19.57	316.35	8.82	0.000
13	0.31	2.11	21.27	340.98	5.46	0.227
14	0.17	1.72	16.86	261.33	10.71	0.000
15	0.33	3.35 ^b	23.30	336.53	3.36	0.113
16	0.15	0.21	13.25	169.50	7.14	0.051
17	0.34	0.96	25.46	375.43	15.96	0.722
18	0.22	0.88	24.31	321.60	0.00	0.438
19	0.61 ^b	0.50	39.15 ^b	516.49 ^b	0.00	0.350
20	0.24	0.84	24.67	341.85	7.98	0.232
21	0.24	0.78	29.20	282.13	36.12 ^b	1.300
22	0.28	0.94	25.76	221.43	0.00	1.424
23	0.17	1.10	30.24	364.68	11.34	1.445
24	0.19	1.38	24.80	331.44	1.05	1.413
25	0.35	1.80	30.16	253.64	2.94	1.341
26	0.18	0.61	18.15	203.15	0.00	0.268
27	0.33	0.45	22.65	178.10	10.08	0.413
28	0.34	0.57	25.83	404.18	0.00	2.477 ^b
29	0.27	0.58	34.18	430.24	0.00	0.000
30	0.47	0.48	33.89	441.93	26.04	0.098
31	0.25	0.99	15.36	276.80	10.71	0.258
32	0.29	1.16	25.97	440.89	10.08	0.052
33	0.41	0.63	22.71	425.59	0.00	0.103
34	0.23	1.53	16.04	320.06	9.66	0.103
35	0.35	1.13	15.03	275.14	14.49	0.557
36	0.23	1.65	15.92	264.81	17.22	1.084
37	0.31	1.23	19.34	261.11	4.62	1.062
38	0.28	1.19	18.01	294.11	5.88	1.135
39	0.52	1.53	20.61	373.53	1.89	1.032
40	0.22	1.37	14.42	233.06	0.84	0.928
41	0.36	1.52	15.89	238.41	14.49	0.629
42	0.83 ^a	1.72	23.04	383.93	1.68	0.402
43	0.50	1.37	22.47	356.28	2.94	0.670
44	0.21	1.27	14.82	239.15	8.82	0.619
45	0.19	1.18	18.96	293.70	1.89	0.670
46	1.09 ^a	1.42	16.18	280.44	0.42	0.908
47	0.15	0.84	13.67	225.74	5.46	0.412
48	0.09	0.88	14.10	220.48	6.51	0.825
49	0.34	1.43	19.35	387.60	1.26	0.846
50	0.17	1.09	17.92	245.90	12.18	0.185
Median	0.27	1.24	20.22	284.46	6.20	0.41

^a C is expressed by percent and unit of other elements is $\mu\text{g g}^{-1}$.

^b Outliers data.

2.5. Source quantification

As the final step of the source fingerprinting method for estimating the relative contribution of each potential source to the sediment samples collected at the watershed outlet, the fingerprint of the sediment was compared with that of the potential sources. This was achieved using a multivariate mixing model as given in the following (Collins & Walling, 2007; Collins et al., 2010c, 2010d; Minella et al., 2008; Walling et al., 2006; Walling & Woodward, 1995):

$$C_i = \sum_{j=1}^n a_{ij}p_{ij} \quad (i = 1, 2, \dots, m) \quad \text{and} \quad (j = 1, 2, \dots, n) \quad (2)$$

Constrained to:

$$0 < P_j < 1 \quad (3)$$

Table 3
Results of applying multivariate outlier criterion of D^2/df for outlier designation.

Sample	D^2	D^2/df	Sample	D^2	D^2/df
1	13.16	1.01	29	12.40	0.95
2	8.15	0.63	30	15.69	1.21
3	6.65	0.50	31	5.01	0.38
4	7.14	0.55	32	13.24	1.02
5	18.81	1.45	33	10.51	0.81
6	19.44	1.50	34	10.27	0.79
7	9.06	0.70	35	7.43	0.57
8	8.61	0.66	36	7.24	0.56
9	9.50	0.73	37	9.42	0.72
10	9.89	0.76	38	9.52	0.73
11	12.82	0.99	39	9.20	0.71
12	6.80	0.52	40	4.66	0.36
13	5.79	0.44	41	5.20	0.40
14	5.61	0.43	42	22.30	1.71
15	29.16	2.24	43	11.89	0.91
16	23.53	1.81	44	11.39	0.88
17	5.13	0.39	45	5.75	0.44
18	6.98	0.53	46	40.44	3.11
19	18.31	1.40	47	6.86	0.52
20	25.18	1.93	48	7.62	0.58
21	24.81	1.91	49	10.34	0.79
22	17.28	1.33	50	14.71	1.13
23	11.46	0.88	51	8.62	0.66
24	8.36	0.64	52	7.25	0.56
25	22.74	1.75	53	16.59	1.27
26	9.66	0.74	54	25.11	1.93
27	10.28	0.79	55	13.80	1.06
28	31.87	2.45	56	6.29	0.48

Table 4
Result of univariate test of normality by Kolmogorov–Smirnov method.

Property	Statistic	Significant level
Al	0.21	0.000**
As	0.08	0.200
Cd	0.18	0.121
Co	0.09	0.200
Cr	0.08	0.200
Cu	0.10	0.200
Fe	0.10	0.200
Mn	0.12	0.060
Ni	0.09	0.200
Pb	0.14	0.200
Se	0.23	0.200
V	0.12	0.075
Zn	0.08	0.200
P	0.16	0.003**
OC	0.15	0.009**
N	0.12	0.000**

** Represents statistically significant at 1%.

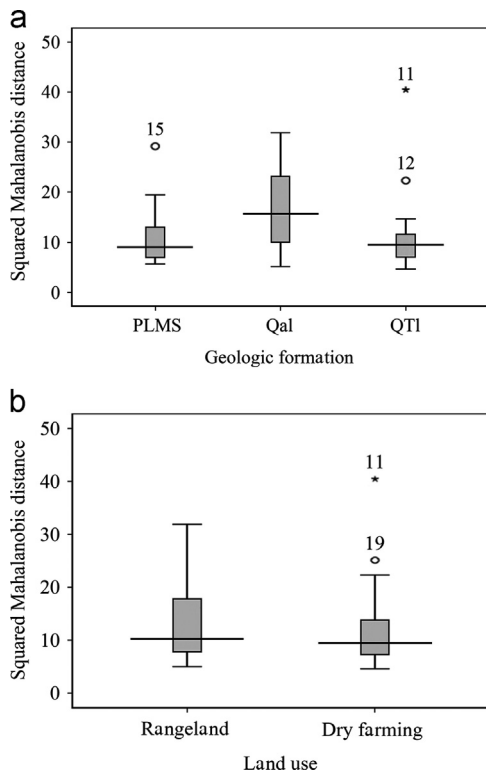


Fig. 2. Box plots of square Mahalanobis Distance measure (D^2) for geologic (a) and land use (b) units for outlier detection.

$$\sum_{j=1}^n p_j = 1 \quad (4)$$

where C_i is the concentration of the element i in the sediment sample; a_{ij} is the mean concentration of fingerprint property i in source j ; p_j is the optimized percentage contribution from sources;

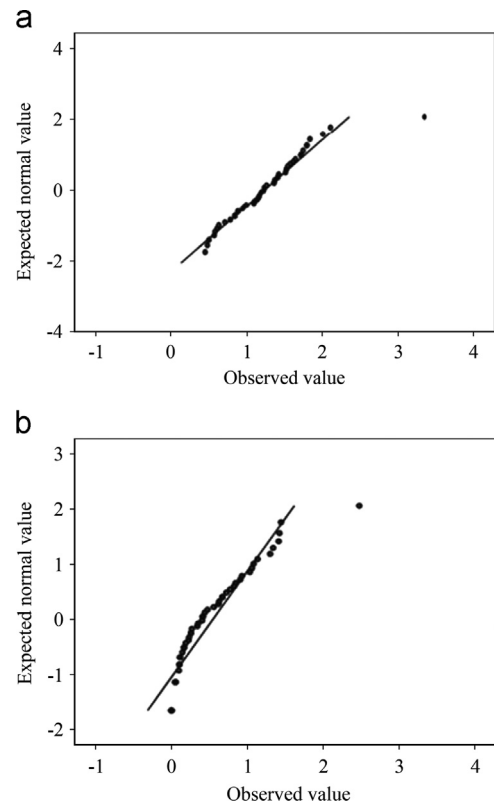


Fig. 3. Examples of normal probability plot As (a) and OC (b).

m is the number of fingerprint properties comprising the optimum composite fingerprint and n is the number of sediment source type categories.

Since the model is over determined, it must be fitted iteratively by minimizing an objective function. Accordingly, the objective function was optimized by minimizing the sum of the squares of the deviations (R) of the predicted property concentrations from the measured values, to determine the relative contribution of each potential source. Although, some correction factors like tracer specific weighting, tracer conservative and particle size correction factors have been used in many studies, but, in the present study, the sieving technique was applied for both source and sediment samples through a 63-micron mesh due to equipment constraints. Eq. (5) shows objective function that was executed using the

Table 5
Correlation matrix and mean values for sediment source properties.

Variable/Source	Al	As	Cd	Co	Cr	Cu	Fe	Mn	Ni	Pb	Se	V	Zn	N	OC	P
Al	1.00															
As	0.09	1.00														
Cd	0.10	0.00	1.00													
Co	0.35	-0.40	-0.09	1.00												
Cr	0.32	-0.03	0.09	0.85	1.00											
Cu	0.26	-0.16	-0.40	0.64	0.55	1.00										
Fe	0.30	0.28	0.01	0.48	0.39	0.02	1.00									
Mn	0.39	-0.01	-0.19	0.83	0.66	0.65	0.47	1.00								
Ni	-0.06	0.24	-0.34	0.55	0.66	0.72	-0.08	0.39	1.00							
Pb	0.28	0.29	0.63	0.01	0.15	-0.26	0.20	-0.22	-0.23	1.00						
Se	0.43	0.43	-1.00	-0.61	-0.60	-0.58	0.04	-0.48	-0.72	0.17	1.00					
V	0.48	0.20	0.11	0.85	0.81	0.57	0.47	0.76	0.38	0.21	-0.45	1.00				
Zn	0.28	-0.38	-0.26	0.61	0.62	0.58	0.17	0.52	0.68	-0.08	-0.70	0.48	1.00			
N	0.27	0.10	0.41	0.19	0.32	-0.18	0.19	-0.04	0.06	0.57	-0.46	0.20	0.18	1.00		
OC	0.10	-0.10	-0.25	0.15	0.21	0.14	-0.02	-0.03	0.30	-0.03	-0.03	0.05	0.29	0.38	1.00	
P	-0.21	-0.10	0.32	-0.12	-0.09	0.04	0.00	-0.08	0.02	-0.13	-0.92	-0.18	-0.03	0.01	-0.03	1.00
Mean values of sediment source properties of geologic and land use units																
Units	Al ^a	As	Cd	Co	Cr	Cu	Fe	Mn	Ni	Pb	Se	V	Zn	N	OC	P
Geologic																
PLM	0.24	1.67	nd	9.41	27.90	20.30	0.98	297.19	21.54	nd	nd	61.19	26.66	0.06	0.19	7.13
Q _t	0.35	1.26	nd	10.20	31.41	17.99	1.22	301.84	21.82	nd	nd	63.11	35.82	0.09	0.62	6.55
Q _{ai}	0.29	0.81	nd	10.34	32.46	26.76	0.77	322.39	28.69	nd	nd	63.28	42.81	0.07	0.80	7.91
Land use																
DF	0.36	1.31	nd	10.09	31.91	17.65	1.15	286.55	22.14	nd	nd	63.01	34.28	0.10	5.74	5.76
RL	0.27	1.21	nd	9.97	30.14	22.90	0.96	315.20	24.50	nd	nd	62.40	35.55	0.06	0.45	7.73

PLM: Red gypsiferous marl, Q_t: Old alluvial clastic, Q_{ai}: Young alluvial deposits, DF: Dry farming, RL: Rangeland
nd: none detected in sediment samples.

^a OC and N are expressed by percent and unit of other elements is $\mu\text{g g}^{-1}$.

Table 6
Results of One-Way ANOVA and Kruskal–Wallis *H*-test as well as Mann–Whitney and *T*-tests application to the geologic and source type fingerprint properties data sets respectively and data multicollinearity.

Property	Geologic units				Land use units					
	One-Way ANOVA		Kruskal–Wallis <i>H</i> -test		<i>T</i> -test		Mann–Whitney test		Multicollinearity	
	<i>F</i> -value	<i>P</i> -value	<i>F</i> -value	<i>P</i> -value	<i>F</i> -value	<i>P</i> -value	<i>F</i> -value	<i>P</i> -value	TOL	VIF
Al	–	–	2.24	0.320	–	–	-0.60	0.54	0.59	1.70
As	15.37	0.000*	–	–	-0.77	0.44	–	–	0.53	1.89
Cd	2.93	0.085	–	–	-1.20	0.24	–	–	–	–
Co	1.37	0.260	–	–	-0.24	0.82	–	–	0.11	9.10
Cr	1.65	0.200	–	–	-0.78	0.44	–	–	0.12	8.60
Cu	16.98	0.000*	–	–	4.14	0.00*	–	–	0.19	5.10
Fe	4.72	0.014*	–	–	-1.38	0.17	–	–	0.45	2.20
Mn	0.42	0.650	–	–	1.12	0.25	–	–	0.20	4.90
Ni	12.31	0.000*	–	–	1.4	0.17	–	–	0.16	6.20
Pb	2.38	0.120	–	–	2.05	0.09	–	–	–	–
Se	5.15	0.110	–	–	-0.95	0.40	–	–	–	–
V	0.17	0.840	–	–	-0.18	0.86	–	–	0.12	8.50
Zn	14.60	0.000*	–	–	0.4	0.69	–	–	0.33	3.10
P	–	–	0.87	0.650	–	–	-0.46	0.64	0.71	1.20
OC	–	–	12.30	0.002*	–	–	-2.97	0.00*	0.84	1.40
N	–	–	13.73	0.001*	–	–	-4.17	0.00*	0.54	1.80

* Represents statistically significant at 5% level.

Solver function in Microsoft Excel (Walling et al., 2006; Zhang et al., 2012).

3. Results and discussion

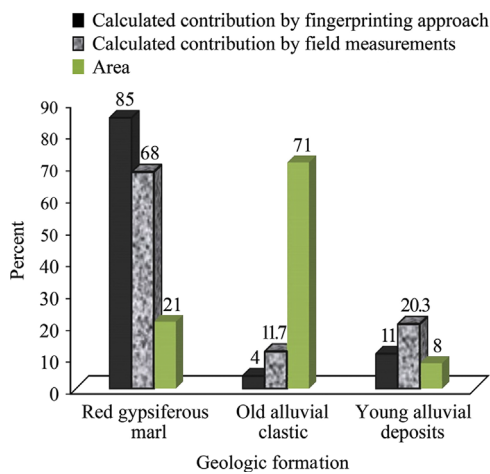
According to procedures of 2–4 section, all 16 properties were applied to designate contribution of different geologic formations and land uses in sediment deposited at the outlet of the

$$R = \sum_{i=1}^m \left\{ \frac{C_i - (\sum_{j=1}^n a_{ij} P_j)}{C_i} \right\}^2 \quad (5)$$

Table 7

Final results of the stepwise Discriminant Function Analysis (DFA) for the geologic and land use units in Idelo watershed, Iran.

Source	Step	Entered fingerprint property	Removed fingerprint property	Wilks' Lambda	Mahalanobis Distance	Cumulative percentage of source samples classified correctly
Geologic units	1	As	–	0.605	0.943	66
	2	N	–	0.389	1.965	74
	3	Cu	–	0.227	4.294	76
	4	Zn	–	0.192	6.052	76
	5	Fe	–	0.152	6.675	84
	6	OC	–	0.126	7.162	84
	7	Co	Fe	0.107	7.295	86
Land use units	1	N	–	0.572	3.425	88
	2	Cu	–	0.478	4.999	90

**Fig. 4.** Area and mean mixing model estimates and field measurements for the relative sediment contributions from each spatial sediment source to the suspended sediment sampled at the outlet of the study watershed.

watershed. The results of the study and discriminative variables are shown in Table 2.

The results of application of univariate outlier test showed that some 3 and 1 samples were laid out of the thresholds as outliers for Al and As, Cu, Mn, P and C tracers in individual, respectively as given in Table 2. Results of examination of multivariate outlier are also given in Table 3. The results showed that only one sample (No. 46) was designated as outlier data with boldface D^2/df value of 3.11 exceeding from critical value of 2.5 as mentioned by Hair et al. (2010). For further survey, the Box plots of square Mahalanobis Distance measure (D^2) for geologic and land use units are shown in Fig. 2. As it is seen in Fig. 2, only 2 samples of 11 and 19 fell out of thresholds in dry farming land uses while sample 15 and, 11 and 16 laid out from the thresholds in case of PLM and Q_t geologic formations. Scrutinizing the results of application of three outliers

test methods, just one sample was proved as outlier by two methods, so that all were retained and applied for further analysis.

Results of examination of univariate normality of data set are also shown in Table 4. Distribution of all properties except Al, P, OC and N was found normal. The normal probability plots also confirmed the same results for which two examples are given in Fig. 3.

The collinearity of data then checked by correlation matrix whose corresponding results are summarized in Table 5. As it is seen in the table, only two pair properties (Se with Cd and Se with P) had the correlation coefficient of higher than 0.90. To avoid collinearity effects on the results, the data set collected for Se was consequently deleted.

The multicollinearity criteria was also checked with the help of TOL and VIF of respective value of less than 0.10 and higher than 10. The corresponding results are accordingly shown in Table 6.

Box's M test was significant for both geologic and land use units in 0.000 and 0.008 levels, respectively, so that the group specific covariance matrices were used instead total covariance matrices. The results of applying suitable tests of ANOVA and Kruskal–Wallis H -test are also given in Table 5, based on which it can be understood that out of all 16 study elements, As, Cu, Fe, Ni, Zn, OC and N with P -values below 5% were capable to discriminate geologic units as sediment source. While, only three properties encompass Cu, N and OC could be supposed as diagnostic properties using Mann–Whitney and T -tests to recognize contributions of land use unit in sediment yield reached the study control point. Decreasing the items of diagnostic properties in land use units can reflect the fact that the geologic subareas (units) do not conform with the land use units (source types) and it is not expected that the source materials in each land use units to exhibit distinct property signatures depended geology as already mentioned by Walling et al. (1999). In other words, the rangeland unit includes each three types of geologic formations so the discriminant power of many properties is exhausted except N and OC that they are independent from geology and mainly incorporates to land cover situations.

Table 8

Total and contribution spatial sources including geologic units and different land uses in sediment yield by field measurements approach.

Land use	Geology	Area (ha)	Area (%)	Average sediment production ($t\ ha^{-1}$)	Total sediment yield (t)	Contribution in sediment yield for geologic unites (%)	Contribution in sediment yield for land use unites (%)
Rangeland	Red gypsiferous	459	23	34.4	14,023.16	68	90.3
	Young alluvial deposits	159	8	26.2	4184.14	20.3	
	Old alluvial clastic	258	13	1.5	387	11.7	
Dry farming	Old alluvial clastic	1093	52	1.83	2000.2		9.7

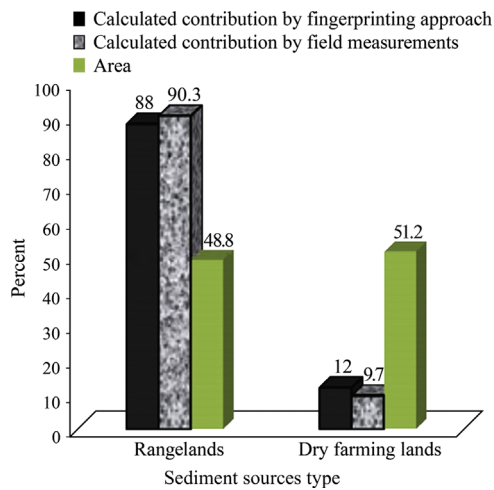


Fig. 5. Area, mean mixing model estimates and field measurements for the relative sediment contributions from each type sources to the suspended sediment sampled at the outlet of the study watershed.

Table 7 represents the final results of the Discriminant Function Analysis (DFA) for the study watershed. Similarly, for geologic units, totally 6 properties viz., As, N, Cu, Zn, OC and Co were selected as optimum composite fingerprint by DFA, which could correctly distinguish 86% of the sediment source samples. However, Fe in initial steps entered in the discriminant function but after adding the OC and Co, the high multicollinearity of Fe with other variables caused to be removed from composite fingerprint process. The optimum composite fingerprint for discriminating source types as defined base on land use encompassed N and Cu and provided a discriminatory efficiency of 90%. Both of the composite signatures were got for spatial sources (geologic units) and source types (land use units) comprised combination from a range of different subsets i.e., organic matter, trace and heavy metals. It confirms that multicomponent signature as a fingerprint is likely to afford the most powerful discrimination. The same findings have been reported by Collins and Walling (2002), Collins et al. (2010a), Hakimkhani et al. (2009) and Sadeghi et al. (2014).

Since the concentrations of sediment properties taken into account for estimating the relative contribution of each potential sources fall within extreme limits of the source material properties, no correction factor was used for the study. Evaluating the goodness-of-fit between the fingerprint property concentrations measured in the sediment samples and the corresponding values predicted by the model, based on the optimized contributions from the individual spatial sources and source types to those samples, indicated typical relative errors of ± 10 and 19% for spatial sources and source types, respectively. These levels of uncertainty with little connivance about source types confirmed that the optimized mixing model provided an acceptable prediction of the fingerprint property concentrations associated with sediment samples that stood within ranges reported by Collins et al. (2010b), Minella et al. (2008) and Walling et al. (2006). Though the uncertainties associated with the mixing model have been assessed with the help of the Monte Carlo approach (Collins et al., 2013).

3.1. Relative Mean contribution from different geologic formations

Fig. 4 represents the mean relative contribution from three geologic units provided by the mixing model. The most important contribution (85%) is provided by the unit underlain by red gypsiferous marl, whereas the lowest is provided by the unit underlain by old alluvial clastic (4%). Young alluvial deposits accordingly

contribute some 11% in yielding sediment at the outlet of the watershed. Aforesaid results were compared with data obtained through field measurement as confirming approach whose results are summarized in Table 8. As the results shown in Table 8, red gypsiferous with 68% is the most important sediment source of the study watershed. Totally, both results of sediment fingerprinting and field measurement approaches confirmed each other as depicted in Fig. 4. High sediment inputs from the red gypsiferous marl are consistent with susceptible soil; fine grain particles, poor vegetation, higher slopes than other units and soils with gypsum that activate different types of soil erosions which promote high sediment yields from this unit. As well as, adjacency of this unit to the main stream and good connectivity to the watercourse system mobilized eroded sediment to directly transport to the stream network as noted by Collins et al. (1997c) for Upper Severn watershed in the UK. In this catchment, the most of the area underlain by old alluvial clastic (71%) but most of the sediment load is derived from red gypsiferous marl with 21% of area which disagreed with Walling et al. (1999) who reported consistency of sediment production and the relative areas occupied by each geologic unit but agreed with findings reported by Hakimkhani and Ahmadi (2008). It was due to existing direct relationship between source type and geochemical properties of the geologic formations distributed in the study watershed. The southern area with old alluvial clastic geology despite covers most of the watershed area but it has only 4 percent contribution in sediment yield. Less contribution from this unit in sediment yield is consistent with smooth slopes, existence coarse materials resulted from conglomerate formations, far distance from the outlet and main stream and lack of complete mature water course in the geology unit similar justifications have been reported by Walling and Collins (2008), Walling et al. (1999) and Wang et al. (2011) in different part of the world.

3.2. Relative Mean contribution from different land uses

Fig. 5 represents the results of contribution of source types including rangeland and dry farming in sediment yield. There is significant contribution from rangelands with 88% versus only 12% contribution from dry farming lands. The results were also compared with data obtained through field measurements. As Table 8 and Fig. 5 show, both results of sediment fingerprinting and field measurement confirmed each other, so rangelands with more than 90% is the most important sediment source of the study watershed. The dominant contribution of rangelands in sediment yield against cultivated area is in agreement with Collins et al. (1997b), Collins et al. (2010a), Jenns et al. (2002) and Walling et al. (1999) findings whilst disagrees with Walling et al. (2006) reported cultivated areas are the most important sediment source. On the base of this fact that rangelands include red gypsiferous marl and young alluvial deposits areas so it is expected that it will be the most important sediment source that there is good agreement between these results and those for spatial sources (Figs. 4 and 5). So sediment mobilization from the rangelands is related to susceptible soil, fine grain particles, higher slopes, good connectivity to the watercourse system as well as intensive livestock grazing which entirely facilitates higher sediment rate.

3.3. Soundness evaluation of the results

The field measurements data obtained from surveying soil erosion types, i.e., sheet, rill and gully erosion confirmed the reliability of results of fingerprinting approach in apportioning geologic formations and land uses in watershed-scaled sediment sources. So that, a fair agreement with absolute and relative errors of 47.46% and 13.13%, and 28.87% and -10.58% were calculated

between estimations made for sediment contributions from geologic formations and land uses by fingerprinting technique with those assessed through field measurements data. Whereas, a good agreement with a relative error of 16% has been reported by Sadeghi et al. (2014) between two data sets calculated based on the measurement of main soil erosion types and applying the composite fingerprinting technique in the same study watershed.

4. Conclusions

This study has demonstrated the successful use of a composite fingerprinting procedure to provide a preliminary integration of spatial provenance and source type information for the contemporary sediment transported through the Idelo watershed. Overall, two fingerprint signatures including As, N, Cu, Zn, OC and Co as well as N and Cu were selected as optimum composite fingerprints to distinguish spatial sources (geologic units) and source types (land use units) from each other, respectively. Integration of spatial provenance and source type information offers in the base of the financial constraints in watershed management and operational and sampling problems about traditional methods, many of the soil erosion controlling measures appropriately placed in the watershed area. It can be inferred from the results that the conservation and control strategies in the study area would be concentrated on the rangelands underlain by red gypsiferous marl with more than 80% contribution in sediment yield. This can be practically used by watershed managers whenever they face financial and time constraints. The field measurements also confirmed the reliability of results of fingerprinting approach in apportioning watershed-scaled sediment sources. However, it seems further field measurements and insight studies are required to further increase the validity of fingerprinting results through considering mobilization, transportation and sediment delivery ratio processes to design holistic policy of soil erosion and sediment control in the study watershed and probably extended to other similar watersheds in the region.

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