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Dating of late Pleistocene and Holocene fluvial sediments Using OSL, Uranium series and ^{14}C methods in the Saqqez River, Iran

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ABSTRACT

Fluvial sediment dating surveys are one of the quaternary researches which can help to explicit many environmental issues. In the present study, samples taken to direct age determination belong to a depositional profile located in one of the tributaries of Saqqez River. This profile is located at southwest of Saqqez city and at the outlet of Saqqez basin. The OSL method was used to determine the age of three samples including sandy lens, floodplain and conglomerate sediments. ^{14}C and U/Th methods was applied to dating of the charcoal in conglomerate sediments and pedogenic/ cement carbonates respectively. The age of the unit is constrained by OSL dating of a sandy lens, at the base of the profile, at 68.35±9.50 ka and the overlying floodplain deposits at 26.76±6.96 ka (i.e. Late Pleistocene). Floodplain consolidated sediment (conglomerate) has been OSL dated at 5.79±1.88 ka which belongs to Mid-Holocene period. Concerning the fluvial sediments ages by the U/Th method, it should be noted that since the ratio of thorium to uranium was much higher than its standard value in both pedogenic and cement carbonate samples, it is not possible to determine the age of carbonate samples using the uranium series method. The calendar age reported by ^{14}C is around 1995, looks enigmatic considering the context from which the sample has been collected and the time required for crystallization of the enclosing carbonate cements. Therefore, it seems that among the different methods of fluvial sediment absolute dating methods, luminescence is more suitable for quaternary fluvial sediments dating in Saqqez River basin.

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1. Introduction

There are numerous routinely employed chemical and physical techniques which provide qualitative and quantitative insights into both contemporary and ancient fluvial systems. These methods have undergrown rapid progress in the past few decades and, in addition, whole new suites of techniques relating to cosmogenic and short0lived isotopes have been developed in response to improvements in both theory and instrumentation.

These approaches vary, in addition to other factors, in the time range applicable, the precision of the resulting age estimates, and the nature of the event being dated (Colman and Pierce, 2000).

Thus, for direct and absolute dating of fluvial sediments we are left with a limited subset of chemical, isotopic and radiogenic methods. These include radiocarbon dating, luminescence-based techniques, and dating via incorporated short-lived radio-isotopes.

In fluvial systems, there is often a complete lack of material suitable for radiocarbon dating; where it is possible, large uncertainties for young samples (<500 years), and the limitations of a 35-50-ka maximum age limit its applicability to latest Pleistocene and Holocene fluvial deposits. Fortunately, over the past few decades there have been considerable advances in short-lived isotopic and radiogenic, particularly luminescence dating, methods. These relatively new and developmental methods provide a great potential for the future age evaluation of fluvial depositional systems (Kondolf and Piegy, 2003). Until recently, the dating of fluvial sediments has been problematic. The only way would have been radiocarbon dating of organic material found in the fluvial deposits. While this may be straightforward for many Holocene deposits, it is only rarely possible for sediments formed during glacial periods, where organic remains are only infrequently found. Furthermore, radiocarbon dating is limited to the last 50 ka due to the half-life of the ^{14}C isotope. Among the difference dating methods, radiocarbon dating can only be used for up to 60 ka years when perorate sediments must contain the appropriate organic matters. Because the samples oxidation and biological impurities, the use ^{14}C method is often not suitable for sediments that are close to the earth's surface (Mahan and Brown, 2007). Other methods, such as Potassium-Argon, Argon-Argon and Uranium series require volcanic and calcareous deposits in site (Lian and Roberts, 2005). However, among the various dating methods, luminescence is more suitable for Quaternary sediment dating (Stokes and Hutt, 1993; Ollerhead et al., 1994; Huntley and Clague, 1996). A relatively new method that overcomes these problems is Optically Stimulated Luminescence (OSL), which allows the direct determination of sediment deposition ages (Wallinga, 2002; Preusser et al., 2008). Most problems encountered with the method, such as the identification of incompletely bleached sediments, have been largely solved and an increasing number of studies have used OSL for establishing chronological frameworks in the past decade (Wallinga et al., 2004; Rittenour et al., 2005). However, two major problems remain with the dating of fluvial sediments. The first is related to the absence of suitable sand layers for OSL dating in some fluvial environments, especially in high fluvial energy regimes in mountainous areas and proglacial settings. The second is related to the upper dating limit of OSL. Although it should be, in principle, possible to date back to several 100 ka by OSL (Schokker et al., 2005; Rhodes et al., 2006), it is highly dependent on the radioactive dose rate in any particular setting. In sediments rich in radioactive elements, the saturation dose of the OSL signal may be reached in about 150 ka or even less. Furthermore, to confirm OSL beyond 200–300 ka, independent age control is desirable because many samples approach saturation and fitting of growth curves gets less precise. Besides OSL and the recently tested approach of burial dating using cosmogenic nuclides (Hauselmann et al., 2007), age control for fluvial sediments beyond 100 ka may be possible by U/Th methodology. Luminescence dating has recently been used for river deposits around the world (Bourke et al., 2003; Cheong et al, 2003; Rittenour et al., 2005; Eriksson et al., 2006; Hanson et al., 2006), especially in Europe (Colls et al., 2001; Fuchs and Lang, 2001; Nador et al., 2007). In Central Europe, Rhine-Mass drainage system has also been extensively studied particularly in Upper Rhine Graben and Lower Rhine area (Wallinga, 2001; Busschers, 2008; Frechen et al., 2008; Lämmermann-Barthel et al., 2008; Lauer et al., 2010). In fact, the chronology of river depositional periods is very important to better understand the river's response to climate, tectonic and eustatic changes. River sediments are suitable for application of optically stimulated luminescence (OSL) (Aitken, 1998; Wallinga, 2002; Busschers, 2008). This method makes it possible to determine the time elapsed since the latest exposure to light, which is related to the time of river sediments aggradation. In Iran, Thermoluminescence method is performed in the Archeological Research Institute for archeological purposes and has not been used in geomorphology and earth sciences. Limitation of this method is the reliable maximum age (about 200 ka years) which can determine (Moeini et al., 2009). Determination the ages of Quaternary terraces in Taleghan basin is the first study which reports the direct ages of this landform in Iran. The dating method used in this study by Moeini et al (2009) is Thermoluminescence that was performed in Taleghan basin (100 km from northwest of Tehran). About Trojan War terrace Thermoluminescence dating yielded 4560-5200 years, Worm's terrace 9100-15600 years and Riss's terrace approximately 100000 years. Despite all attractions that the results of river sediments dating approach can have for the researcher, research about Quaternary period is very limited in Iran and not considerable compared to other geological periods. Applied studies in natural resources and environmental science began in 1975 for three years in Taleghan basin by Ahmadi in collaboration with Renal, a professor of Strasburg University in France, and the naming of terraces done based on similarities with French Alps terraces, especially French's extensive studies in north Africa (Moeini et al., 2009). In this study, although the age of terraces was estimated, their direct age was not specified. Subsequent researches by other researchers have been conducted on the basis of other countries experiences or on the preliminary studies of Ahmadi and Renal. Therefore, fluvial deposits dating studies are one of the researches disciplinary can help clarify many environmental issues, including climate changes, tectonic processes and morphological interpretations related to these issues. Accordingly, the ages of floodplain unconsolidated (sandy lens) and consolidated (conglomerate) sediments also alluvial sediments affected by pedogenic processes was calculated using absolute dating methods including OSL, ^{14}C and U/Th in Saqqez river basin.

2. Material and Methods

2. 1. Case study

In this study, samples taken to direct age determination belong to a sedimentary profile located in one of the tributaries of Saqqez River. This profile is located at southwest of Saqqez city and at the outlet of Saqqez basin. Saqqez basin as a study area of this research is 835 km2 in area in west Iran, from 36° to 36° 17´ and from 45° 46´ to 46° 19´ (Fig. 1).

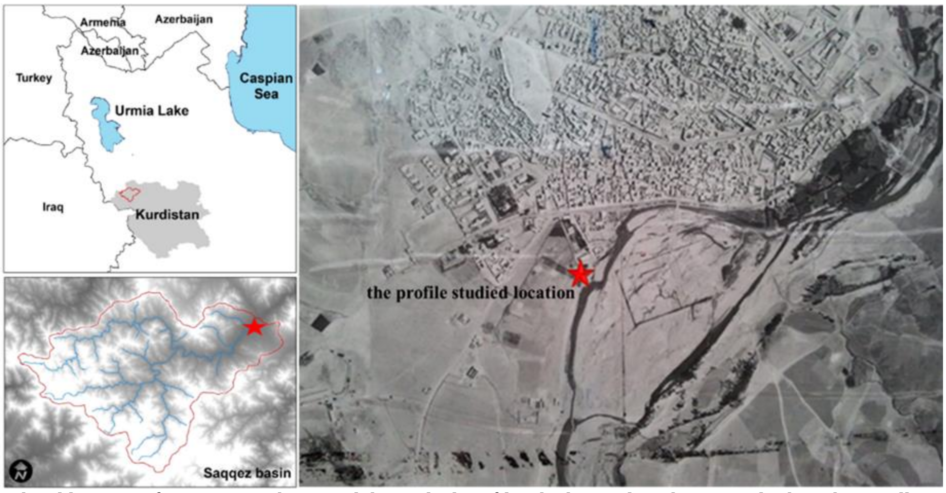

Fig. 1. Geographical location of Saqqez river basin and the studied profile which samples where samples have been collected to sediment direct dating (the year of aerial photos is 1963).

The basin is located in Kurdistan Province and Saqqez County. The study area has a mountainous climatic condition with harsh cold winter of snow and temperate summer. The minimum temperature in winter is reduced to -30˚C and the maximum in summer is 40˚ C. The highest point of that is 2700 m at the Vazeneh Mountain. The average of annual precipitation in the area is 350 to 650 mm and relative humidity is ranged from 14% to 95%. This climate condition developed forest and rangelands in the region mainly in the south and southwest parts of the study area; the north and northeastern parts are devoid of vegetation cover except along the rivers near the villages and springs. River (Cham) of Saqqez is originated from the high mountainous areas, Pirbodagh and Vazeneh Mountains, near Baneh city; it is flowing in a southwest-northeast direction towards Saqqez city and connected to Zarrinehrood River in a long distance from the city. In terms of tectonic setting, the area is located in northwestern margin of the transformed strip of Sanandaj-Sirjan. It is, in fact, this zone acts in junction with the tectonic zones with the tectonic zones of Khoy-Mahabad and Alborz-Azerbaijan. Thus, the geologic units in the area are somewhat similar to Sanandaj-Sirjan (Precambrian metamorphic rocks) and in some parts they have similarities in lithology to tectonic zones of Alborz-Azerbaijan (carbonate clasts of Precambrian and Paleozoic and Mesozoic rocks). The middle parts of the basin mainly composed of Cretaceous igneous rocks has a gentle topography with low height eroded flat hills (Derafshi et al., 2017).

2.2. OSL dating

The radiation to which a grain is exposed during burial derives from a number of different sources, and consists of alpha and beta particles, and gamma rays. Quartz grains contain very little internal radioactivity, so the majority of the radiation they receive is derived from uranium, thorium, and potassium contained within the surrounding sediment, as well as a small level of cosmic radiation. The radiation flux at a sampling location is termed the environmental dose-rate, and can be obtained either by field or by laboratory measurements. The amount of radiation that a grain has received during burial can be determined by measuring the OSL signal from the natural dose (i.e. that received during burial), and the OSL signals from a series of laboratory irradiations of known dose. The laboratory measurements are used to calibrate the OSL signal derived from the natural dose, and thus determine the laboratory dose that is equivalent to the dose received by the grains in nature; this is termed the equivalent dose (De), with the unit of measurement in Grays (Gy). Since the OSL signal is bleached on exposure to light during sediment transport, the De normally represents the amount of radiation received following deposition and burial after the last transport event. By deriving the burial dose (De) of a sample, and the environmental dose rate at the sampling location, the age of a sample can be found using the equation 1.

Age (Ka) = Equivalent Dose (Gy) / Dose Rate (Gy/Ka) (1)

Where the equivalent dose (in Grays) is the laboratory radiation dose that produces the signal equivalent to the natural luminescence signal for the grains being measured (Duller, 2004). The dose rate (in Grays per thousand years) is the annual dose received by those grains in their depositional environment (Duller, 2004). This annual dose rate refers to the rate at which mineral grains in sediment absorb energy from the surrounding flux of radiation which comes from the naturally occurring radionuclides 232Th, 238U and 235U and their daughters, and 40K and 87Rb as well as cosmic radiation. Ideally, the OSL signal is derived from a single type of mineral grain such as quartz or potassium feldspar mineral separates. The dating technique can be applied to the age range from present to approximately 300 000 years ago and precision varies from ~3-10% of age for heated materials and ~5-20% for sediments (Wintle et al., 1993). Initial studies of the application of OSL dating to sediments concentrated on depositional environments where the transport process ensured that sufficient exposure to sunlight to bleach the material had occurred, such as aeolian deposits (e.g. Huntley et al., 1985; Stokes, 1992). In some other depositional settings (e.g. fluvial or colluvial), not every grain receives light exposure of a sufficient strength and/or duration to bleach fully the dose from the previous burial period. If a residual trapped charge remains in the grains, the sediment is regarded as partially or incompletely bleached. Where partial bleaching is present, careful assessment of the distribution of De values is necessary to obtain the correct age. Simply taking some measure of the average from the De values for a partially bleached sample is not appropriate because the grains with residual trapped charge will cause overestimation of the age (Wallinga, 2002; Kim, 2009). In this study, three samples of fluvial consolidated conglomerate, floodplain and fine-grain fluvial sediments (sandy lens) were collected using the plant of appropriate metal pipes among the sediments to OSL dating (Fig. 2). At the time of sampling, control conditions were considered such as kept the sedimentary contents from sunlight, their depth from the earth surface (the minimum sample depth from the earth surface must be 2 meters to be protected from the effected of cosmic rays and estimate accurate proportion of these rays from the luminescence signals) and the adequate sand content of samples. Locations and characteristics of the sample sites are given in figure 3.

Fig. 2. Implantation of suitable metal pipes in river sediments (sandy lens) to determine OSL age

The samples have been sent to OSL laboratory in Witwatersrand University of Johannesburg, South Africa, to determine their D_e and OSL dating. All luminescence measurements are conducted on Risø Reader Model –DA-15 as an automatic measurement system that can measure both TL and OSL for the quartz and feldspar. Common procedure to calculate De is SAR (Single Aliquot Regenerative) protocol (Murray and Wintle, 2003) that its stages are presented in Table 1. The information about dose rate for detached materials of volume samples has been achieved by high precision Gamma-Ray Spectrometer (GRS) in iThemba Laboratory, South Africa, in order to determine isotope abundance of major radionuclides. To do this, the dose rates of quartz grains have been calculated using the final transformation factors (Guerin et al., 2011). Cosmic dose rate has also been calculated by Hutton (1994) equations as a function of elevation, latitude, longitude, and depth from the earth surface. With equivalent dose and dose rate available, we can finally calculate the ages by OSL.

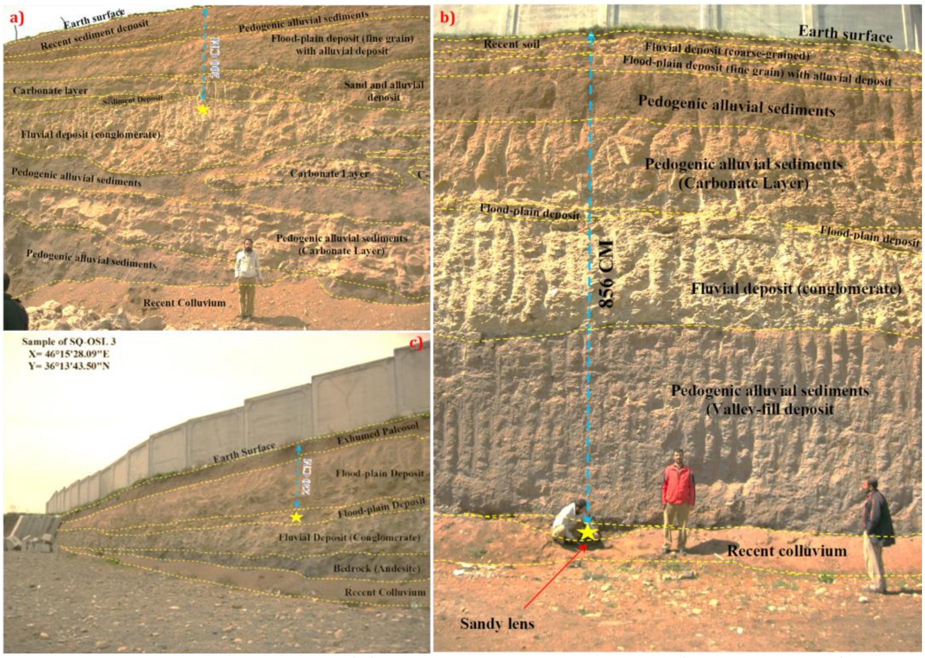

Fig. 3. a) Location and stratigraphic characteristics of fluvial consolidated conglomerate for OSL dating; floodplain unconsolidated sediments are located lower the conglomerate and more recent fluvial and also colluvial sediments upper that. The intermingled cement in fluvial sediments of the conglomerate is calcite; b) location and stratigraphic characteristics of sandy lens sediment sample for OSL dating; the sandy lens is located the lowest part of the profile and the alluvial deposits are accumulated above that; this may be resulted from sedimentation inside a Paleo-valley and c) location and stratigraphic characteristics of an unconsolidated floodplain sediment sample (from fine-sand to silt) for OSL dating; consolidated conglomerate sediments are located lower the sample and fine grain floodplain sediments upper that.

Table 1. The SAR protocol all sample for OSL dating

Step	Treatment
1	Give dose (Natural,0, 13.5, 405, 0, 13.5 Gy)
2	Preheat 260°C for 10 s (240°C for SQ2)
3	IRSL 10 s at 75°C
4	OSL 40 s at 125°C
5	Test dose (6.75 Gy)
6	Preheat 180°C for 5 s
7	IRSL 10 s at 75°C
8	OSL 40 s at 125°C
9	OSL 5s at 205°C

High resolution gamma spectroscopy was performed on material sub-sampled from the 'bulk' samples to determine the isotopic abundances of the parent radionuclides, by iThemba Labs, South Africa. From these, the dose rates to quartz grains have been calculated using the latest conversion factors (Guerin et al., 2011). Allowance was made for the attenuation of the beta dose due to grain size (Mejdahl, 1979). Dose attenuation due to water follows Zimmerman (1971), using the measured water content values. The cosmic dose rate was determined as a function of altitude, latitude/longitude and depth, according to Prescott and Hutton's (1994) equations, plus the soft component from Madsen (2005). A constant sedimentation rate was assumed for the cosmic component.

2.2. ^{14}C dating

Radiocarbon dating is a method that provides objective age estimates for carbon-based materials that originated from living organisms. An age could be estimated by measuring the amount of carbon-14 present in the sample and comparing this against an internationally used reference standard. The impact of the radiocarbon dating technique on modern man has made it one of the most significant discoveries of the 20th century. No other scientific method has managed to revolutionize man's understanding not only of his present but also of events that already happened thousands of years ago. Archaeology, geomorphology, geology and other earth and human sciences use radiocarbon dating to prove or disprove theories. Over the years, carbon 14 dating has also found applications in hydrology, geophysics, atmospheric science, oceanography, paleoclimatology and even biomedicine.

The method was developed in the late 1940s at the University of Chicago by Willard Libby, who received the Nobel Prize in Chemistry for his work in 1960. It is based on the fact that radiocarbon (^{14}C) is constantly being created in the atmosphere by the interaction of cosmic rays with atmospheric nitrogen.

The resulting ^{14}C combines with atmospheric oxygen to form radioactive carbon dioxide, which is incorporated into plants by photosynthesis; animals then acquire ^{14}C by eating the plants. When the animal or plant dies, it stops exchanging carbon with its environment, and thereafter the amount of ^{14}C it contains begins to decrease as the ^{14}C undergoes radioactive decay.

Measuring the amount of ^{14}C in a sample from a dead plant or animal, such as a piece of wood or a fragment of bone, provides information that can be used to calculate when the animal or plant died. The older a sample is, the less ^{14}C there is to be detected, and because the half-life of ^{14}C (the period of time after which half of a given sample will have decayed) is about 5,730 years, the oldest dates that can be reliably measured by this process date to around over 50,000 years ago, although special preparation methods occasionally permit accurate analysis of older samples. In this study, 14C dating method was used to calculate the age of existed charcoal in one of the conglomerates of Saqqez River (Fig. 4). The sample was sent to Poznan Radiocarbon Laboratory in Poland. The used procedure to ^{14}C dating of the sample is AMS technique which includes few steps: chemical pretreatment, production of CO_2 and graphitisation, AMS ^{14}C measurement and calculation of ^{14}C age and calibration of ^{14}C age. Methods of chemical pre-treatment generally follow those used in the Oxford Radiocarbon Accelerator Unit, as described by Brock et al., (2010). Sample of charcoal (after mechanical removal of macroscopic contamination visible under binocular) are treated with 0.25M (UV1) - 1M (UV, UW, ZR) HCl (80°C, 20+ min), 0.1M NaOH (room temperature for fragile plant remains (UV, UV1), 80°C for wood and charcoal (UW, ZR)) and then 0.25M HCl (80°C, 1 hour). After treatment with each reagent, the samples are rinsed with deionised water (Millipore) until pH=7. For the first HCl treatment, longer time (20+) is applied, if emanation of gas bubbles from sample is still visible. The step of NaOH treatment is repeated a few times, generally until no more coloration of the NaOH solution appears (coloration of solution is caused by humic acids dissolved in NaOH), but the NaOH treatment is interrupted if there is a danger of complete dissolution of the sample.

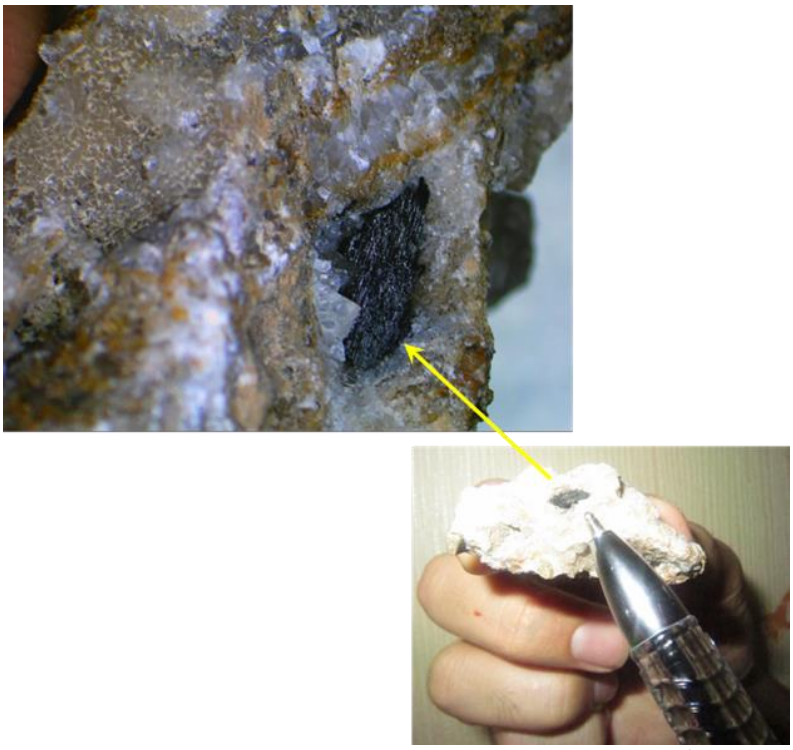

Fig. 4. A sample of charcoal that sent to 14C dating at Poznan Radiocarbon Laboratory; surrounding of the charcoal piece has covered by calcite carbonate that some parts of it have been replaced by dolomite carbonate.

In case of organic samples, CO_2 is produced by combusting the sample. Combustion of organic samples is performed in closed (sealed under vacuum) quartz tubes, together with CuO and Ag wool, in 900°Cover 10 hours. CO_2 from carbonate samples is leached by treating with concentrated orto-phosphoric acid (H_3PO_4) in a vacuum line. The obtained gas (CO_2 + water vapour) is then dried in a vacuum line, and reduced with hydrogen (H_2), using 2 mg of Fe powder as a catalyst. The obtained mixture of carbon and iron is then pressed into special aluminium holder, according to the description provided by Czernik and Goslar (2001). Measurements described in this point, are performed in the AMS ^{14}C Laboratory of Mickiewicz University in Poznan. Content of ^{14}C in a sample of carbon is measured using the spectrometer “Compact Carbon AMS”. The measurement is performed by comparing intensities of ionic beams of ^{14}C, ^{13}C and ^{12}C measured for the sample. Conventional ^{14}C age is calculated using correction for isotopic fractionation, basing on ratio $^{13}C/^{12}C$ measured in the AMS spectrometer simultaneously with the ratio $^{14}C/^{12}C$. Uncertainty of calculated ^{14}C age is determined using uncertainty implied from counting statistics, and also spread (standard deviation) of partial $^{14}C/^{12}C$ results, whichever is bigger. Calibration of ^{14}C age is performed using the program OxCal ver. 4.2 (2014). Calibration is performed against the newest version of ^{14}C calibration curve i.e. INTCAL13.

2.3. U-series dating

Difference types of carbonate cements can be observed in fluvial terrace gravels at basin downstream. Therefore, therefore, it is possible to focus the study on well-developed calcite cements in gravels that can be used for age measurements. The organic matter aggradation can contribute to the depletion of calcites and lead to the formation of stratified sediments, which are suitable materials for determining age using uranium (U/Th) series. In this study, two samples of conglomerate sediments and fluvial sediments affected by pedogenic (paleosols) were used to calculate calibrated age using U-series. 0.1-0.2 g of carbonate cement of the fluvial conglomerate sediment were mechanically separated by binocular microscope (Fig. 5). Carbonates of the fluvial sediment affected by pedogenic was also separated using mass weight method. The prepared samples were sent to the Department of Environmental and Earth Sciences at Queensland University in Australia to determine the age of the uranium series.

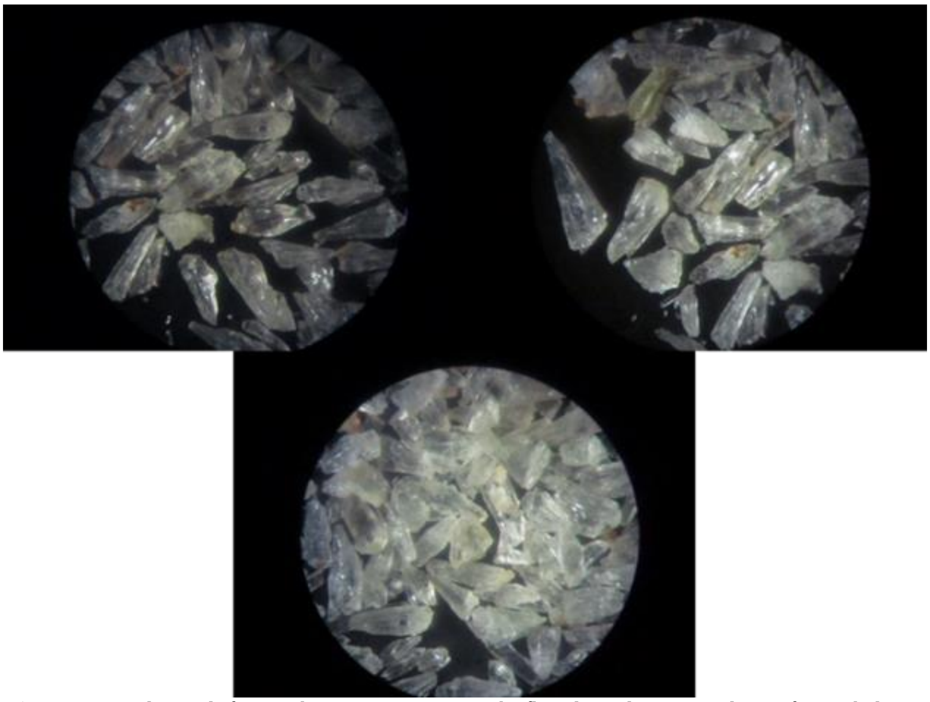

Fig. 5. Separated crystals from carbonate cement among the fluvial conglomerate sediment for U/Th dating

The MC-ICP-MS method, a special appropriate protocol provided in detail by Zhou et al, (2011) and Clark et al., (2014) was used to U/Th dating of the carbonates. The samples spiked with a mixed $^{229}Th/^{236}U$ spike, dissolved in HCl, taken to dryness and then converted to the nitrate form. Organic material, if present, was attacked with 0.5 ml of H_2O_2 þ HNO_3. U and Th were separated on anion columns using 0.5 ml Eichrom U-TEVA resin. Matrix was removed with 7 M HNO3; Th was eluted with 5 N HCl and finally U with 0.5 N HCl. After evaporation, U and Th fractions were treated in oxygen plasma to destroy organic remnants from the resin. U and Th mass spectrometry was done as described by Fleitmann et al., (2007), using a Nu Instruments multi collector ICP-MS equipped with an ESI Apex_ desolvating system without membrane and using a self-aspirating nebulizer. With an uptake rate of ca. 50 ml/min, the ion yield for U and Th was about 70 V/ppm. U measurements were done from 0.5 N HNO_3 solutions in static mode, whereby masses 236 and 234 were measured in parallel electron multipliers and 235 and 238 in Faraday cups. Baselines were taken on either side of peaks and interpolated. The electron multiplier yield was calibrated every 2 samples by running an NIST U050 solution. The 238U/235U ratio was used for instrumental fractionation correction if the 238U signal was greater than 1 V (10_11 A); if smaller, the fractionation factor was input from bracketing standards. Normal washout time for U between samples was 5 min with 0.5 N HNO3 ($<1^0/_{00}$ memory); longer washout times were used where significant isotope differences between samples were expected. Runs on the NIST U960 standard yielded d ($^{234}U/^{238}U$) -37.2±

2.1$^{0}/_{00}$ (1SD, n=35) where the equilibrium ratio is after Cheng et al., (2000). The measurements were made from 3 N HCl solutions in a two-cycle multicollector dynamic mode, whereby one electron multiplier, equipped with a WARP filter, alternatingly measured masses 229 and 230. U standard was added to Th run solutions for two reasons, first, to enable correction for instrumental mass fractionation, and second, to provide a reference isotope (238) to eliminate the effects of plasma flicker in obtaining the $^{229}Th/^{230}Th$ ratios. Variations of U and Th signals during the run are fully correlated if no organic matter is present. Baselines were measured at 229.5 and 230.5 for samples and standards with significant (>10^{-12} A) ^{232}Th. Washout time was 5 min to 1$^{0}/_{00}$ of the Th signal, if the capillary and nebulizer were free of organics. Ultimately, the U/Th ages were estimated using the Isoplot/Ex 3.75 program (Ludwing, 2012).

3. Results and discussion

Currently, there are various methods for determining the direct age of fluvial sediments in the world, including OSL, ^{14}C, and U/Th. In the study of paleoenvironmental conditions of the Saqqez basin, the OSL method was used to determine the age of three samples including sandy lens, floodplain and conglomerate sediments. ^{14}C and U/Th methods was applied to dating of the charcoal in conglomerate sediments and pedogenic/ cement carbonates respectively. According to the age results obtained from OSL (Table 2), sandy lens in the base of the study profile on the bedrock is 68.35±9.5 ka of years old. The age of the alluvial deposits influenced by pedogenesis which located above the sandy lens can also be estimated and considered as the oldest sediments of Saqqez River. The function of weathering processes on Plio-Quaternary basal unit and carrying its products has probably been during at the time of Quaternary period when the fluvial regime has not yet formed at the basin and sediment transport has occurred by extremely high energy water flows (flood flows). As the studied samples of alluvial sediments have similar mineral and textural composition and there is no specific stratification in the samples, it seems the deposits have aggregated as a mass or debris flow. However, due to the non-uniform patterns and intensive weathering of mineral components in the samples, the manifestations of weathering are probably inherited from the sedimentary deposits origin, but the impregnated of the context with iron oxides and formation of quartz and iron oxide in the samples is the result of pedogenic processes. The most obvious effect of pedogenic processes is the formation of pedogenic carbonate layers among these alluvial sediments.

Table 2. OSL dating of Saqqez basin fluvial sediments

Sample	*Th (ppm)*	*U (ppm)*	*K (%)*	*WC (%)*	*Dr (Gy.ka-1)*	*Over-dispersion (%)*	*De (Gy)*	*Age (ka)*
SQ-1	9.4±0.4	1.71±0.07	1.79±0.03	10.1	2.80±0.1	85±4	16.51±5.3	5.79±1.9
SQ-2	9.86±0.1	1.67±0.06	1.39±0.03	11.9	2.35±0.1	32±1	160.87±21.2	68.35±9.5
SQ-3	10.2±0.4	1.81±0.06	1.36±0.03	5.5	2.60±0.09	65±4	69.68±17.9	26.76±6.9

A layer of fine grain and unconsolidated sediments of floodplain is located above alluvial pedogenic sediments. Based on OSL dating, the age of this layer is obtained about 26.76±6.96 ka of years. After transition from cold and arid climate condition that have formed calcareous layers among old alluvial sediments, to more humid climate, the river channels system was formed in the basin which is witnessed by consolidated channels sediments as conglomerate and floodplain sediments covering old alluvial deposits with erosional boundaries. A fluvial channel deposit (conglomerate) are the third layer dated by OSL; the age of this layer is about 5.79±1.88 ka of years and related to middle Holocene. Table 3 shows the results of the U/Th analysis for two samples of pedogenic and cement carbonates. According to this, the ratio of thorium to uranium in both samples is much higher than its standard value (0.005) and it is practically impossible to determine the ages of carbonate samples using uranium series method. How much the ratio of thorium to uranium be lower, this method estimates more accurate ages. As it is currently the only suitable method for dating of carbonate materials, it was not possible to estimate the age of this type of material in the basin.

Table 3. U/Th dating of pedogenic and cement carbonates

Sample Name	Sample wt.(g)	Spike wt.(g)	U (ppm)	±2s	^{232}Th (ppb)	±2s	(^{230}Th/ ^{232}Th)	±2s	(^{230}Th/^{238}U)	±2s	(^{234}U/ ^{238}U)	±2s	Uncorr. Age (ka)	±2s	corr. Age (ka)	±2s	corr. Initial (^{234}U/ ^{238}U)	±2s
IS-UB-4	0.0094	0.00260	0.0870	0.0000	463.54	0.966	0.647	0.009	1.1353	0.0156	1.0072	0.0089	Sample contains too much detritus (Th/U = 5.3) to give an age. Material not suited for dating					
SQ-PK-CC2	0.14205	0.01604	0.8440	0.0008	84.11	0.141	38.86	0.10	1.2764	0.0029	1.2015	0.0008	555	29	554	57	1.99	0.15

About the charcoal sample dating using ^{14}C method, unexpectedly, the charcoal in the sample showed a recent age. Based on ^{14}C ages calibrated with OxCal software, with probabilities of 1.2 and 93.3%, the charcoal sample is related to 1959 and 1990, respectively, which shows the recent age (Fig. 6). The charcoal is located in the conglomerate matrix that mineralogical studies of this sample illustrate, in addition to calcite cement, there is dolomite cement in the sample and has replaced the previous cement. The existence of dolomite cement in this sample is the result of groundwater and springs that have locally created dolomite cement during short time periods; this is one of the environmental changes which has occurred a few recent decades (short-time) in Saqqez basin.

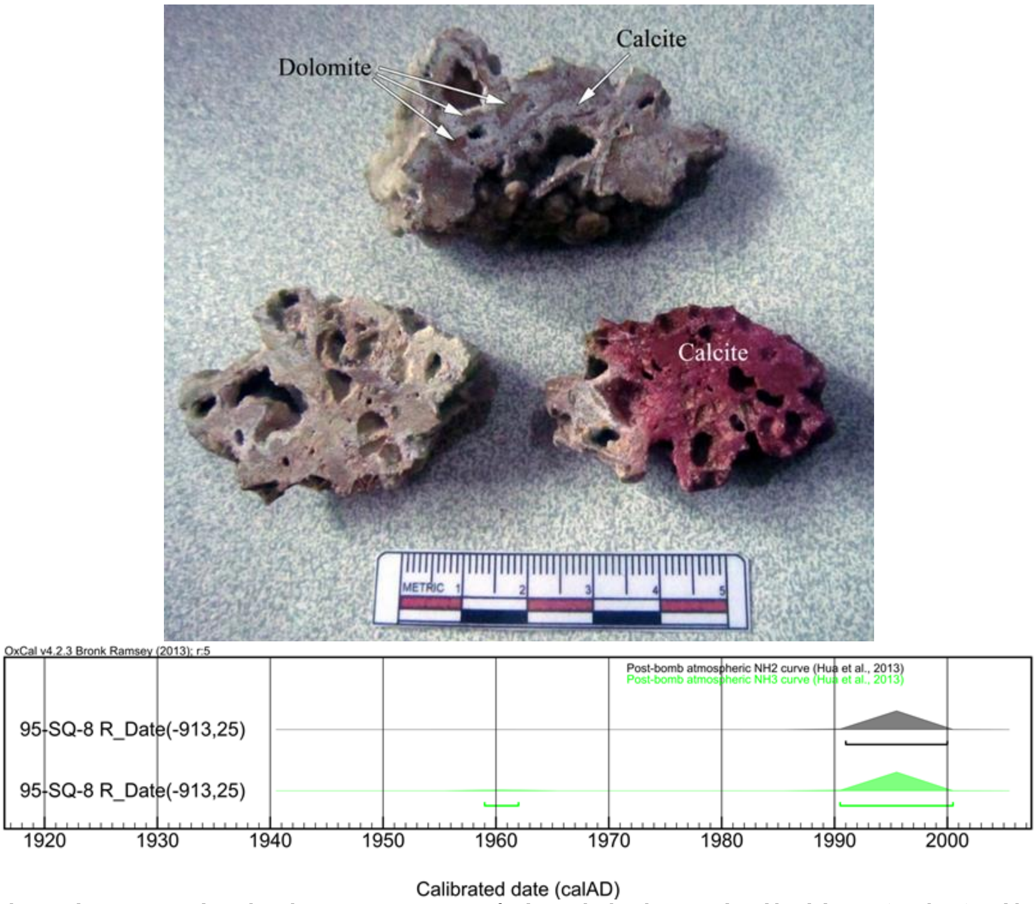

Fig. 6. The conglomerate sample with carbonate cement in type of calcite which is being replaced by dolomite (up photo); calibrated age data of ^{14}C based on NH2 and NH3 curved (down photo).

4. Conclusion

Until recently, the dating of fluvial sediments has been problematic. The only way would have been radiocarbon dating of organic material found in the fluvial deposits. While this may be straightforward for many Holocene deposits, it is only rarely possible for sediments formed during glacial periods, where organic remains are only infrequently found. Furthermore, radiocarbon dating is limited to the last 50 ka due to the half-life of the ^{14}C isotope. A relatively new method that overcomes these problems is Optically Stimulated Luminescence (OSL), which allows the direct determination of sediment deposition ages (Wallinga, 2002; Preusser et al., 2008). Most problems encountered with the method, such as the identification of incompletely bleached sediments, have been largely solved and an increasing number of studies have used OSL for establishing chronological frameworks in the past decade (Wallinga et al., 2004; Rittenour et al., 2005). However, two major problems remain with the dating of fluvial sediments. The first is related to the absence of suitable sand layers for OSL dating in some fluvial environments, especially in high fluvial energy regimes in mountainous areas and proglacial settings. The second is related to the upper dating limit of OSL. Although it should be, in principle, possible to date back to several 100 ka by OSL, it is highly dependent on the radioactive dose rate in any particular setting. In sediments rich in radioactive elements, the saturation dose of the OSL signal may be reached in about 150 ka or even less. Furthermore, to confirm OSL beyond 200–300 ka, independent age control is desirable because many samples approach saturation and fitting of growth curves gets less precise: a recent study reports a systematic underestimation of quartz OSL dates in this age range (Wallinga et al., 2007). Besides OSL and the recently tested approach of burial dating using cosmogenic nuclides (Hauselmann et al., 2007), age control for fluvial sediments beyond 100 ka may be possible by U/Th methodology. Based on the above, it seems that the most appropriate method for absolute dating of fluvial sediments (especially conglomerate sediments and sand layers) is the OSL. According to the age results obtained from OSL, sandy lens in the base of the study profile on the bedrock is 68.35±9.5 ka of years old. The age of the alluvial deposits influenced by pedogenesis which located above the sandy lens can also be estimated and considered as the oldest sediments of Saqqez River. A layer of fine grain and unconsolidated sediments of floodplain is located above alluvial pedogenic sediments. Based on OSL dating, the age of this layer is obtained about 26.76±6.96 ka of years. A fluvial channel deposit (conglomerate) are the third layer dated by OSL; the age of this layer is about 5.79±1.88 ka of years and related to middle Holocene (Derafshi et al., 2017). Figure 7 shows the chronology and stratigraphy of the age-determined layers of the studied profile in this research.

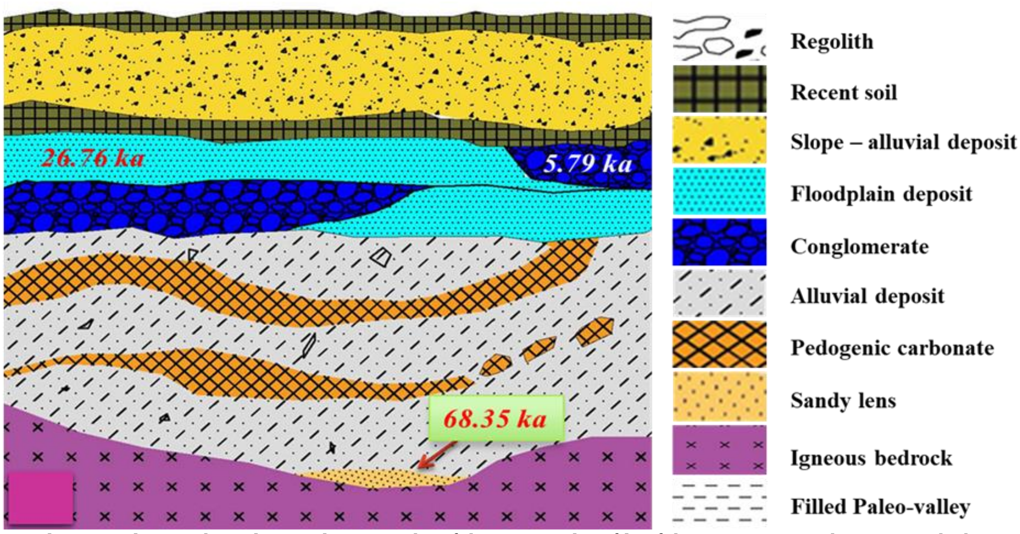

Fig. 7. Sedimentary layers Chronology and stratigraphy of the examined profile of the Saqqez river tributary near the basin outlet

Currently, the only suitable method for dating of carbonate materials in deposits is the U/Th series. How much the ratio of thorium to uranium be lower, this method estimates more accurate ages. As it is, it was not possible to estimate the age of this type of material in the study basin. As the ratio of thorium to uranium is low in marine carbonate deposit, it can be more suitable for these than fluvial sediments. About the charcoal sample dating using ^{14}C method, unexpectedly, the charcoal in the sample showed a recent age. The charcoal is located in the conglomerate matrix that mineralogical studies of this sample illustrate, in addition to calcite cement, there is dolomite cement in the sample and has replaced the previous cement. The existence of dolomite cement in this sample is the result of groundwater and springs that have locally created dolomite cement during short time periods. There is a main issue with charcoals dating in fluvial ancient sediments that they are may be deposited as a result of recent environmental and anthropogenic processes. Also, the radiocarbon method can only be used for up to 60,000 years, and sediments also must contain suitable organic materials for the age calculate. Based on mentioned states, it seems that among the various methods of fluvial sediments dating, the luminescence method is more suitable to quaternary sediments.

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