

ANALYSIS OF SOIL PHOSPHATE AS A TOOL IN ARCHAEOLOGY, CASE STUDY, RIVI, NORTH KHORASAN, IRAN

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Abstract: Phosphate is unique among the elements in being a sensitive and persistent indicator of human occupation. It has long been of interest to archaeologists because of its potential to inform them about the presence of past human activity and to offer clues regarding the type and intensity of human occupation. In fact, the soil of settlements is part of the phosphorus reservoir. The reconstruction of the human activities areas of archaeological sites using soil phosphate analysis is a well-known technique. This study aims to identify and compare the activity area at ancient mounds of Rivi through the measurement of the quantity of phosphates in the soil. In this study, 29 soil samples were collected from the study area, and phosphorus quantity was measured using Spectrophotometry. Multivariate statistical methods were used to classify the obtained results. The results showed that the phosphorus concentrations in the ancient areas were higher than in the control area, and among the ancient areas, the phosphorus quantity related to Rivi B was higher than in other mounds. Previous studies have shown that the Rivi area has been inhabited from around 2900 years ago to the last Sassanian years (1,500 years ago), and in the middle of the Islamic age (1000 years ago), it was a great village. In total, the archaeological site of Rivi was inhabited during the Iron Age, Achaemenid, Parthian, Sassanid, and Islamic periods, and that is why there was a high concentration of phosphorus in the Rivi area compared to the control area.

Keywords: Phosphorus, Chemical Analysis of Soil, Rivi mounds, Ancient Human Activity, North Khorasan.

چکیده: فسفر در میان عناصر که نشانگر حساسیت و فعالیت‌های پایدار انسانی است، منحصر به فرد می‌باشد. باستان‌شناسان به دلیل پتانسیل فسفات در آگاهی بخشیدن به آن‌ها در مورد فعالیت‌های انسان در گذشته و ارائه سرنخ‌هایی در مورد نوع و شدت فعالیت انسانی، به مدت طولانی علاقه‌مند بوده‌اند. در واقع خاک سکونت‌گاه‌ها بخشی از مخزن فسفر است. بازسازی فعالیت‌های انسانی در مناطق باستان‌شناسی با استفاده از تجزیه و تحلیل فسفات خاک شیوه‌ای شناخته شده است. هدف از این تحقیق بررسی و مقایسه مکان‌های فعالیت در تپه‌های ریوی، استان خراسان شمالی با استفاده از آنالیز شیمیایی فسفات خاک می‌باشد. جهت انجام مطالعه ۲۹ نمونه خاک از منطقه مورد مطالعه برداشت شد و غلظت فسفر با استفاده از دستگاه اسپکتروفتومتری اندازه‌گیری شد. برای طبقه‌بندی نتایج به‌دست‌آمده از روش‌های چند متغیری آماری استفاده شد. نتایج نشان داد که غلظت فسفر در مناطق باستانی بسیار بیشتر از منطقه شاهد است و در بین مناطق داخل محوطه، مقادیر نمونه‌های مربوط به تپه ریوی B بیشتر از تپه‌های دیگر بود. تحقیقات پیشین نشان داد که محوطه ریوی از حدود ۲۹۰۰ سال پیش تا سال‌های پایانی دوران ساسانی (۱۵۰۰ سال پیش) با کاربری شهری مسکونی بوده است و در دوران میانه اسلامی (۱۰۰۰ سال پیش)، روستایی بزرگ بوده است. در مجموع محوطه ریوی در دوره‌های آهن، هخامنشی، اشکانی، ساسانی و اسلامی مسکونی بوده است. در حقیقت غلظت بالای فسفر در محوطه ریوی نسبت به منطقه شاهد به همین دلیل است.

کلمات کلیدی: فسفر، آنالیز شیمیایی خاک، تپه‌های ریوی، فعالیت‌های انسان باستان، روش آنالیز اسپکتروفتومتری، خراسان شمالی.

I. Introduction

At the beginning of the Neolithic period, humans played an essential role in changing soil properties as an important factor. Over time, the results of transformational farming activities, from natural soils to arable soils, and the direct and indirect effects of livestock, both in terms of fertilizer production and in a harmful way (overgrazing of animals), changed the physical and chemical properties of soils. These include human changes, soil erosion, changes in height and elevation to create different buildings, soil drainage, salinization of soil or the addition of organic nutrients to soil, soil compaction, etc. Studies over the past few decades have shown that human settlement, even in its short-term and temporary form, has significant effects on soil. For example, soil samples from two contemporary fishing camps in western Alaska were chemically decomposed in an archaeological study. In reviewing the results of this study, although one of the camps had been established for 30 years and the other

had been inhabited for one year, both camps showed significant traces of human contact in the soil. Different amounts of Al, Ba, Ca, Fe, K, Mg, Mn, P, Sr, Ti, and Zn were measured in each camp (Knudson *et al.*, 2004). Today, most urban dwellers may have lost their physical and emotional connection to the soil, but in the not-so-distant past, the soil was the first environment in which people worked, intervened, and lived off the soil. Increasing awareness of the importance of soil in several respects reveals the fact that soils alone are worth preserving for the future, and soils can be considered archives (Blum *et al.*, 2006). It can be argued that much of the cultural heritage is preserved in the soil, and on a much larger scale than it can be obtained by storage in museums. Soil is now crucial for human societies as well as the environment. Given that soils can easily be used as archives of information about human affairs in the past (Linderholm, 2007).

Many of the activities that we humans do remain biological and physical as time goes on. The main part

of these works is enclosed at the molecular level. Archaeologically, the soil should not be considered a discrete entity because soil (chemical) data reflects ongoing processes. Soil is a large mass that is constantly changing, but the rate of change is different. Soil information has a strong explanatory power that enables temporal interpretations of human activities and behaviors. The impact of the human environment on soil and sediments in terms of pollution is a well-known phenomenon because industrialization has intensified over historical periods and left its mark on pollution. However, humans have always stored or disposed of various materials and compounds related to their habitats for livelihood, but these early effects are not always available depending on the state of soil and sediment protection. Biological elements are in constant contact with humans and their habitats, especially phosphate, which is evident in this case. This was already the case in 1930 when iron age settlements were identified through high phosphate levels in soil phosphate mapping. With successive stages of human activity, the soil is like a manuscript on which writing is erased and rewritten (Entwistle *et al.*, 1998).

As a heterogeneous substance in nature, soil is generally a relatively complex object to study. Similarly, soils are studied in various scientific fields, such as physics, chemistry, biology, geology, soil science, geography, agriculture, forestry, and many others. For this reason, different analytical methods and techniques are used to study various soil phenomena. There are big differences in how we relate to the concept of soil due to the scientific context, and the methods of observing soil are also different: as sediment or as a growth medium for plants and organisms. Quaternary geology has a starting point in sediments and sediment formation. Soil science is dedicated to soil formation, focusing on plant-living interactions. General agricultural and forestry research is directed towards productivity, and geotechnology considers soils and sediments as building materials (Retallack, 2001). Adding archaeology to this list provides another angle from which the formation of sediments and soils must be recognized from a human cultural perspective.

Today, most studies related to soil chemical decomposition focus on phosphate groups because phosphates are a biological factor (including humans) in the sedimentary system and the concentrations and changes associated with them indicate places of human activity (Rapp and Hill, 1998). Studies show that some human activities increase soil phosphorus, while other activities reduce it or have no effect on soil phosphorus (Holliday and Gartner, 2007). Sources of phosphorus related to pre-industrial human activities include animal and human wastes, bone residues and waste, meat, fish and plants, burials, animal manures used in agriculture, and fire ash (Gurney, 1985; Farrell, 1997; Holliday and

Gartner, 2007). Therefore, it is natural that the amount of phosphate in places such as kitchens and food processing places, food storage points, slaughterhouses, stoves, cemeteries, fertile lands, stables, pastures, garbage dumps, gardens Fields, battlefields, industrial sites, passages, and routes where waste is dumped, are above normal levels (King, 2007; Farrell, 1997; Terry *et al.*, 2000). Natural phosphate levels in the soil are determined by the soil standard of each area as well as by sampling from a pristine area.

Olof Arrhenius was the first to discover the relationship between soil phosphate content and man-occupied sites. In the early 1930s, Arrhenius established a link between high levels of phosphorus and the presence of human cities and began a systematic analysis of phosphorus in soil samples. This type of analysis has become a useful tool for finding archaeological sites and research in human activity areas. The use of phosphate analysis in archaeology is possible because past human occupations and activities have increased soil phosphorus content in the regions. Phosphorus accumulated in archaeological sediments remains constant over time and can be measured through soil sampling and chemical analysis, making it a valuable tool for archaeology that can be used for many research conditions.

The application of soil phosphate analysis in archaeology can be divided into four general areas:

First: Phosphate levels are determined prior to excavation to identify archaeological sites prior to excavation, which is the most common use of phosphate analysis in archaeology.

Second: Researchers use phosphate analysis as a tool to explore specific places, such as cooking, and to help guide excavations.

Third: Phosphate analysis is used as a tool in landscape archaeology to study the location of agriculture.

Fourth: Description of characteristics and areas of activity through phosphate analysis during and after drilling (Parnell, 2001).

In general, a chemical element is only suitable for such research if it has the following characteristics:

First, human activity must have altered the natural concentration of that element in the soil of the ancient site.

Second, this change must be detectable in comparison with the normal concentration of the element.

Third, the element needs to be stabilized in a stable form in the soil (Entwistle *et al.*, 1998).

Heavy elements are naturally scarce in the soil, and their high concentrations are limited to certain minerals and activities. In the past, the most important way for heavy elements to enter the soil was to work with metals and other human activities, which play a major role in

the amount of heavy elements in the soil. Therefore, knowing the amount and type of them in this regard is very important and the results of this study can be of valuable help in archaeological studies.

II. Materials and methods

II. 1. Area of study

The study area is located in the Samalqan plain, between latitude 56 degrees and 32 minutes to 56 degrees and 49 minutes east longitude and 37 degrees and 27 minutes to 37 degrees and 37 minutes north latitude (Fig. 1). Samalqan plain is located in the west of

Bojnourd County and the southern part of the middle Atrak basin. This plain is limited to the main valley of Atrak from the north, the plain of Bojnourd from the east, the plain of Shoghan from the south, and the plain of Qarahmidan from the west and northwest. Samalqan plain is one of the second-degree catchments of the Atrak river, which has an area of more than 1148 square kilometers. This plain has also reached the national register and has a certificate. It is also located next to the Aladagh, Yaman Dagh, and Qorkhod heights (Rouhani, 2020).

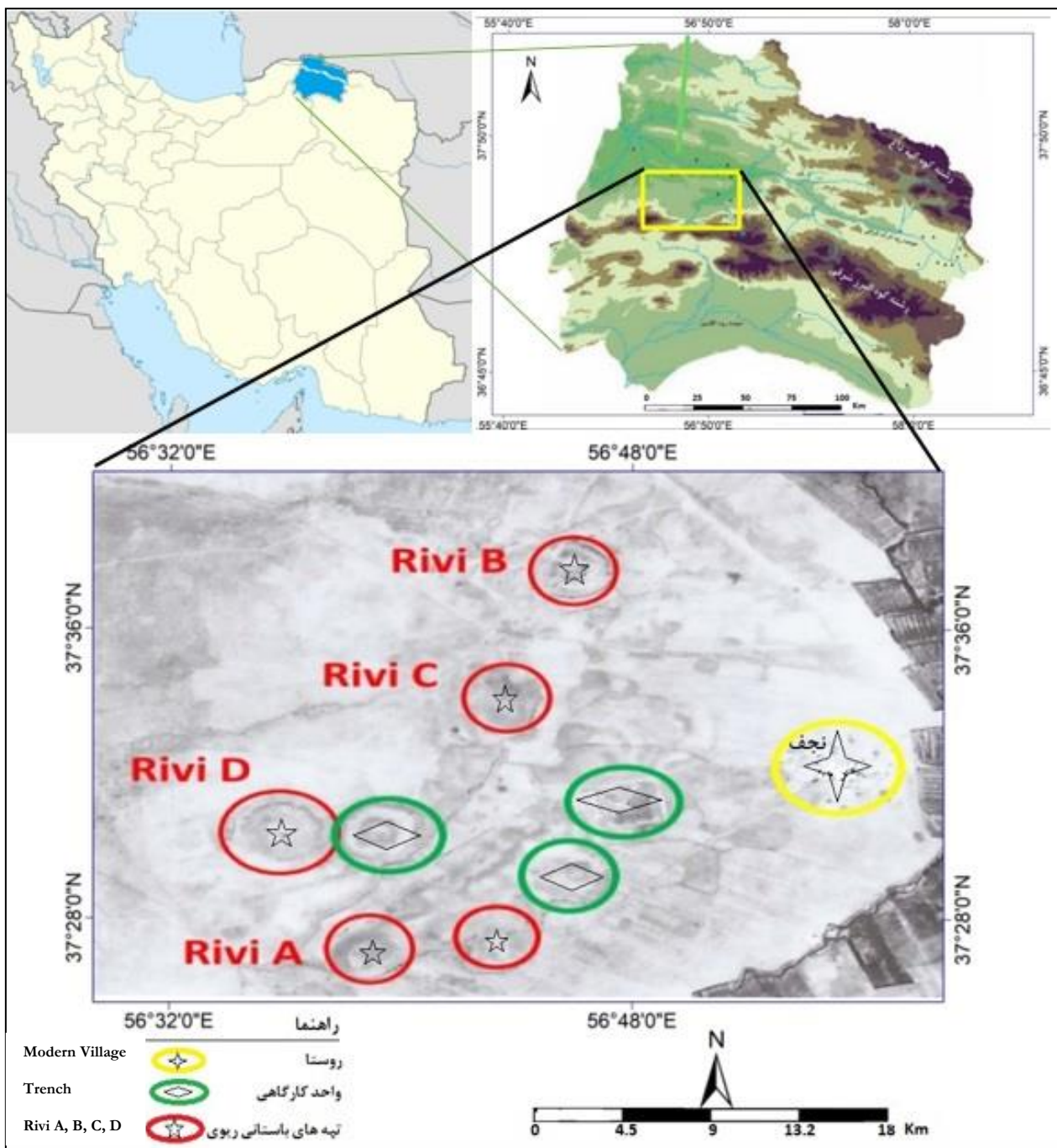


Figure 1. Location of the study area in North Khorasan and Iran.

Archaeological excavations since 2012 at the Rivi site near the village of Najaf in Maneh Va Samalqan County have uncovered the remains of a city from the Achaemenid period. Archaeological excavations in the county of Maneh Va Samalqan, which took place in 2012 and 2014 in the Rivi historical site, yielded significant results from the existence of a large historical site that has been established from about 900 BC to the late Sassanid era, about 1500 years. Its area during the Achaemenid period reached more than 110 hectares and seems to have been one of the most important cities of the Achaemenid period in the northeast of the country. According to these researches, from the Parthian and Sassanid eras, various architectural and archeological evidence have been obtained from Rivi Site, which has shown the settlement sequence in Rivi Site after the Achaemenid period. This ancient site consists of at least four hills A, B, C, and D and covers more than 110 hectares (Fig. 1) (Jafari, 2015). The central region has been damaged since the 1970s by soil collection activities of brick factories. According to satellite data, several ancient hills and features such as rectangular structures and hollow paths have disappeared over the past 40 years (Jafari and Thomalsky, 2016).

According to research conducted on this site in recent years, it was discovered.

1) Tepe Rivi B. In this map, the surface layers from zero to a depth of five meters belong to the Sassanid era (1800 to 1400 years ago).

2) Tepe Rivi A. At this point, the first meter is related to the last 500 years and from a depth of one to three meters is related to the Achaemenid period (2500 years ago).

3) Tepe Rivi D. There is a brick historical monument related to the Achaemenid period (2500 years ago), pristine and natural lower layers. In total, from the ground to a depth of 4 meters, the layers belong to the Achaemenid period and below 4 meters is virgin soil (Jafari, 2013; Rouhani *et al.*, 2021).

III. Sampling and analysis of soil samples

Soil sampling will be very important and will determine the degree of accuracy of the results obtained from the test. In this study, 29 samples were collected from 5 different areas to study and compare the changes in phosphorus concentration in soil profiles of the ancient Rivi area. In addition to the samples of Anthrosoils collected from inside the ancient site, several samples were taken as control from points located outside the ancient site with pristine soil and no ancient texture.

The purpose of collecting control samples was to compare the changes of elements in the Anthrosoil samples with the control and to measure the elemental changes outside and inside the ancient site. The classified sampling method was used to collect the samples from Rivi Site soil. This type of sampling is used when the sample units are not in the same condition. Table 1 shows the coordinates of the sampling points. To prevent contamination of the samples, tools such as brushes and stainless spatulas for collecting the samples were washed and distilled with distilled water after each sampling. The samples were poured into special plastic containers with lids, and after coding, they were transferred to the laboratory.

Table 1. Geographical location of the sampled areas.

Number	Name	Coordinate	
		Latitude	Longitude
1	Rivi A	37.34563	56.53077
2	Rivi B	37.57533951	56.88218401
3	Rivi D	37.57139356	56.87767161
4	Control	37.58231551	56.88546242

After transferring the samples to the laboratory, the samples were sieved through a 10 size 2 mm mesh sieve after drying in air. To determine the concentration, we first took 5 grams of soil, placed it in an Erlenmeyer flask, and then poured 100 ml of half a molar calcium bicarbonate. Shake the suspension for half an hour and then strain it through filter paper to obtain a clear extract. We inserted 15 ccs of the extract into a 25-laboratory flask by pipette and gently added 5cc of ammonium molybdate solution. Shake the Laboratory flask gradually to release the carbon dioxide. After this step, we added one cc of tin chloride and increased the volume of the laboratory flask to 25 ccs. We calibrated the light transmittance (I) after calibration of the standards 0.1, 0.9, 0.7, 0.3, and zero ppm at 660 nm.

Data were read by a UV-Visible model spectrophotometer (Rouhani *et al.*, 2021), and soil extraction was performed using the Olsen method (Olsen and Sommers, 1982).

In this study, statistical analysis of data was performed using SPSS 20 software, and the mean data were compared with a one-way analysis of variance, which determined the presence or absence of significant differences in the level of reliability. Duncan's test was used to compare the means.

IV. Results

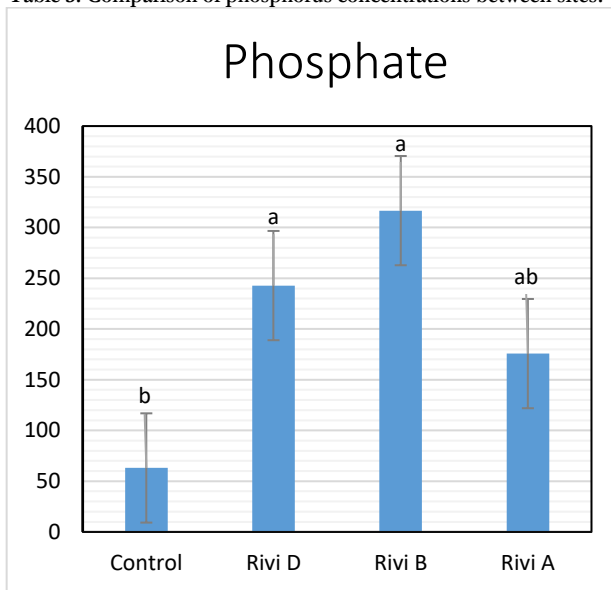
A statistical description of phosphorus concentration in the collected samples is given in **Table 2**. Its average concentration is 205.05 mg / kg. Phosphorus data were

normal according to the Kolmogorov-Smirnov test. The values obtained from the analysis of variance indicated a significant difference in phosphorus concentration in the hills of Rivi A, Rivi B, Rivi D, and the control area. The control area has the lowest concentration (63.04 mg/kg), and Tepe Rivi B has the highest concentration (316.67 mg/kg) (Table 3).

Table 2. Descriptive statistics of phosphorus concentration.

Sources of changes	Free	Average of squares	P-Value
Repeat	3	205.05	.055 ^c
Error	22	135.47	

Table 3. Comparison of phosphorus concentrations between sites.



V. Discussion

Sources of phosphorus related to pre-industrial human activities include animal and human wastes, bone residues and waste, meat, fish and plants, burials, animal manures used in agriculture, and fire ash (Gurney, 1985; Farrell, 1997; Holliday and Gartner, 2007). Therefore, it is natural that the amount of phosphate in places such as kitchens and food processing places, food storage points, slaughterhouses, stoves, cemeteries, fertile lands, stables, pastures, garbage dumps, gardens, fields, battlefields, industrial sites, passages, and routes where waste is dumped, are above normal levels (King, 2007; Farrell, 1997; Terry *et al.*, 2000).

The average phosphate concentration for the samples inside the site is much higher than the average of the control area. Undoubtedly, this significant increase in phosphorus can only be justified if we attribute it to the human impact on the soil of this hill. The lowest amount of phosphate among the samples is related to the samples in the control area. This clearly indicates a lack of human activity in such areas outside the site or at least a sharp decline. The highest

phosphate content is in Tepe Rivi B. During their research, Dr. Judith Tomalski and Dr. Mohammad Javad Jafari discovered a cemetery in this area. It is observed that the samples related to Tepe Rivi B have much more phosphate compared to Tepe Rivi A and D and the control area. Probably the main reason for this increase was the addition of human bones to the cemetery. It can be seen that the amount of phosphate in the samples taken from the site is so much that it can be stated with certainty that in these places the remnants of human activities have been added to the soil.

Phosphorus depletion may have been due to sweeping such areas immediately after the activity and cleaning them of various wastes and excreta, which ultimately prevented the stabilization and accumulation of phosphates in the soil (Wells, 2004). Such actions may prevent the deposition of organic matter residues and certain special elements in a religious building. For example, across Central America, sweeping was done primarily by women. Women have traditionally been responsible for preserving local temples in these areas, which has evoked a spiritual concept or burden for them (Wells *et al.*, 2000). The samples taken for Tepe Rivi A are from the points that have been proven during the research that these points were the royal mansion. The reason for the low concentration of phosphorus in the samples of this period could probably be due to the constant cleaning of this mansion by its crew. As a result, the accumulation and stabilization of phosphate in the soil are prevented.

VI. Conclusion

Humans can leave a geochemical artifact at an archeological site in a variety of ways. Together with other components of the biosphere, there is selective enrichment of elements in human body tissues that may lead to detectable abnormalities after death. Of the elements that are concentrated in this way, phosphorus is the most obvious. Analysis of soil samples taken from the site shows that there is a close relationship between points containing evidence of human activity and high levels of phosphorus. Some parts of the site, such as Tepe Rivi A, have low levels of elements such as phosphorus, so it can be said that in these places, there may not have been any significant activities such as cooking and food consumption, animal husbandry, industrial activities, etc., or its effects are gone. In contrast, the significant amount of phosphorus in the samples related to Tepe Rivi B indicates that certain activities such as animal and human wastes, bone waste, meat, fish and plants, burials and animal manures used in agriculture and fire ash have made in these places. Studies have shown that stoves are high in phosphate (Ryan Roth, 2002).

Soil and its formation are certainly archives, protection, and reflections of the remnants of human,

plant, and animal life, both in the present and in the past. The soil as a repository of information about cultural-natural phenomena must be protected. In natural heritage management, the focus is on conserving natural soils. "Cultural" soils, on the other hand, have received less attention. For this reason, it is suggested that the authorities prevent activities such as agriculture,

industry, etc. that cause the destruction of the site soil and do not allow agriculture in the area. Examination of phosphorus levels showed that the soil of the Rivi B site (Parthian and Sassanid period) has high amounts of P, which indicates the existence of a lot of evidence of ancient activities. It is suggested that this area be further explored.

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