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Authors' contributions

EOD and AAA jointly designed this study; EOD carried out archaeobotanical analyses, photographing of plant remains, consideration of archaeoentomological data and contributed to the environmental background and to the discussion with archaeobotanical and ethnobotanical aspects: AAA, RK, and ET carried out the detailed archaeometrical analyses of the ancient buckthorn pyrenes and handled the chemical interpretation; Kİ conducted the recent archaeological investigations at the study site, provided all the archaeological data, archaeobotanical remains, and the historical background for the manuscript

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Competing interests

No competing interests have been declared.

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Ancient plant remains with special reference to buckthorn, *Frangula alnus* Mill., pyrenes from Dascyleum, Balıkesir, NW Turkey

Emel Oybak Dönmez¹*, Ali Akın Akyol², Recep Karadağ³, Emine Torgan^₄, Kaan İren^₅

¹ Department of Biology, Faculty of Science, Hacettepe University, 06800 Beytepe, Ankara, Turkey ² Material Research and Conservation Laboratory (MAKLAB), Department of Restoration and Conservation of Cultural Properties, Faculty of Fine Arts, Gazi University, 06830 Gölbaşı, Ankara, Turkey

³ Natural Dye Research Laboratory, Department of Textile, Faculty of Arts, Marmara University, 34660 Acıbadem, İstanbul, Turkey

⁴ TCF and Armaggan Company, Cultural Heritage Preservation and Natural Dyes Laboratory, Keyap Çarşı Sitesi, A2 Blok, No. 14, 34775 Y. Dudullu, Ümraniye, İstanbul, Turkey ⁵ Department of Archaeology, Faculty of Art and Science, Muğla Sıtkı Koçman University, 48000 Kötekli, Muğla, Turkey

* Corresponding author. Email: polen@hacettepe.edu.tr

Abstract

Carbonized plant remains recovered from the ancient city Dascyleum (Daskyleion) in the province of Balıkesir in northwestern Turkey provide an outline of several phases of plant use in archaic, Hellenistic, and medieval times. At the study site, various crop plant remains of Near Eastern agriculture, including cereals (barley, *Hordeum vulgare* L. and bread/durum/rivet wheat, *Triticum aestivum* L. / *T. durum* Desf. / *T. turgidum* L.) and pulses [bitter vetch, *Vicia ervilia* (L.) Willd.; grass pea, *Lathyrus sativus* L. / *L. cicera* L.; fava bean, *V. faba* L.; and chickpea, *Cicer arietinum* L.] were found. Drupaceous fruits and pyrenes of buckthorn (*Frangula alnus* Mill.) were also found, probably representing dyes and/or medicines used by the inhabitants of the mound.

Archaeometrical analyses of the ancient buckthorn pyrenes by high performance liquid chromatography with photodiode array detector (HPLC-PDA) provide chemical evidence for traces of ancient mordants remaining until the present day.

Some of the pulse seed remains retrieved from the medieval layers at the study site were found to have been infested by bruchid beetles (Bruchidae).

Keywords

archaeobotany; archaeometry, ancient buckthorn; Daskyleion; Turkey

Introduction

The acropolis of Dascyleum (Daskyleion), situated on Hisartepe Höyük, lies south of the Marmara Sea and Manyas Lake (Kuş Lake), near Ergili village of Bandırma, in Balıkesir Province in northwestern Turkey (Fig. 1a,b). The top of Hisartepe covers an area of 2.7 hectare and lies 36 m above lake level.

The study site was first identified by Kurt Bittel in 1952 [1]. Archaeological excavations started in 1954 under the direction of Ekrem Akurgal and lasted until 1959 [2,3]. Tomris Bakır and her team restarted excavations in 1988 [4]. The site has been excavated under the direction of Kaan İren since 2009 [5]. The tumuli (ancient burial mounds) around Manyas Lake correspond to the burials of Lydian, Persian, Macedonian, and local elites. Archaeometrical studies by Akyol et al. [6] and Özdemir et al. [7] have been conducted at one of these tumuli, called the Koru tumulus, near Dascyleum.

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Fig. 1 Location of Dascyleum. a Location map of Dascyleum. b General view (from the northeast) of Dascyleum on Hisartepe (arrowed).

The excavations revealed that Dascyleum occupied a key strategic position among regions of the Thrace, Straits, Marmara Sea, Black Sea, and Phrygia in ancient Anatolia. It had a multicultural character ranging from Lydian–Phrygian to Byzantine, including the Anatolian satrapy of the Persian emperor from 546 to 334 BC.

During excavation seasons from 1989 to 2011 on the mound, carbonized plant remains were collected from several areas of the mound burnt in ancient times. Plant remains are represented by kernels of cereals and seeds of pulses, and fruits and pyrenes (seeds surrounded by hard endocarp) of buckthorn (*Frangula alnus* Mill.). The discovery of buckthorn fruits and pyrenes is of special interest because these remains are rare in Near Eastern archaeobotanical assemblages, and the fruits and seeds are known to be a traditional source of natural dye containing various substances such as emodin and rhamnetin, and medicines [8].

This paper primarily deals with the archaeobotanical analyses of plant remains obtained from several strata (cultural layers), including the Archaic, Persian, Hellenistic, and medieval layers, at the site of Dascyleum with the aim of characterizing ancient plant use on the mound. The present study also focuses on the archaeometrical analyses of ancient buckthorn pyrenes from the medieval layers of the site by high-performance liquid chromatography with photodiode array detector (HPLC-PDA), with the aim of identifying ancient mordants.

Environmental background

Dascyleum is situated on Neogene limestones (called Hisartepe) south of Manyas Lake [9]. A recent paleopalynological study of the surroundings of the Marmara Sea by Biltekin [10] documents vegetation and climatic change in the region of Dascyleum from the late Miocene up to the present day, spanning approximately the last 7 million years.

From 1.8 million years ago to the present day, herbaceous ecosystems of Amaranthaceae/Chenopodiaceae, Poaceae, Asteraceae and mugwort (*Artemisia* L.) steppes developed, and relict plants such as hickory (*Carya* Nutt.) and hornbeam (*Carpinus orientalis* Mill.) persisted in the region of Marmara. The chestnut (*Castanea* Mill.) – oak (*Quercus* L.) forest at the mountain vegetation level in the Kazdağı (Mount Ida) massif in the west of the region gradually declined from the eleventh century BC onwards, probably owing to human impact, which resulted in the spread of black pine (*Pinus nigra* J. F. Arnold) forests [11].

In the study area of Dascyleum, during the late Quaternary the main palaeogeographic elements were the Karadere Stream, an outlet from the lake, and the Kocaçay Delta in the Manyas Lake area [12]. Then, the lake areas enlarged and the Karadere Stream formed vast flood plains.

Dascyleum City center was situated on a small peninsula and the city itself was surrounded by a river and its marshy area. Regional tectonic movements lasting from approximately 4000 to 3000–2800 years BP (uncalibrated) (4104 calibrated BP to 3169 cal. BP – 2908 cal. BP) resulted in the formation of open lacustrine conditions in the Manyas Lake area. This formation was accompanied by a decrease in the Marmara Sea level [13]. The dates of around 800 or 1000 AD have been linked to the end of the Beyşehir Occupation Phase. The historical event at 460 AD that destroyed various settlements in the Manyas Lake area has been associated with a seismic event or the early Byzantine tectonic paroxysm and the 447 AD seism over the entire Marmara Sea region [13].

Kazancı et al. [14] suggested that deforestation created high rates of sedimentation in the basins and/or strong denudation of the region during the late Holocene. A recent study of the Hellenistic layers of Dascyleum City by Yaman et al. [15] revealed that some wood species, including beech (*Fagus cf. orientalis* Lipsky), elm (*Ulmus* L.), and oak (*Quercus*), were employed in construction and also possibly used as fuel at the site.

The present day climate of the study area is typically Mediterranean with hot, arid summers and moderately cool and moist winters. In the area, the warmest months are July and August (43.2°C and 43.7°C) and the coldest month is February (–18.8°C). The mean annual precipitation is 615 mm, most of which falls in December [16].

Today, the mesophytic forest formations of the mountains of the Black Sea Region in the north extend into parts of the southern Marmara region, depending on relief and climate [17–19]. These formations can be found in mountainous areas in the vicinity of Dascyleum, especially on Kazdağı (1774 m) in the west and Uludağ (2543 m) in the south.

At higher elevations facing the north, fir [*Abies nordmanniana* (Steven) Spach subsp. *equi-trojani* on Kazdağı and *Abies bornmulleriana* Mattf. on Uludağ], beech (*Fagus orientalis*), chestnut (*Castanea sativa* Mill.), hornbeam (*Carpinus betulus* L.), lime (*Tilia tomentosa* Moench), and oak [*Quercus petraea* Mattuschka (Liebl.)] prevail. In the south of Balıkesir Province, drier forest formations, including brutia pine (*Pinus brutia* Tenore) and black pine (*Pinus nigra*), become common. The bush formation that developed following human impact on forests is characterized mainly by mock privet (*Phillyrea latifolia* L.) and Greek strawberry tree (*Arbutus andrachne* L.)

In the region, a subalpine zone is found mostly above the timberline from 2000 to 2100 m [20]. Juniper (*Juniperus communis* L. subsp. *nana*) and prickly thrift (*Acantholimon* Boiss.) are characteristic associations of the subalpine zone. Above 2300 m, alpine grasslands prevail. The pseudo-alpine zone on Kazdağı is rich in species, consisting of 189 vascular plant taxa [21].

The main species of maquis vegetation in the southern coastal belt of the Marmara Sea are Greek strawberry tree (*Arbutus andrachne*), strawberry tree (*A. unedo* L.), weaver's broom (*Spartium junceum* L.), and laurel (*Laurus nobilis* L.) [17]. The present vegetation of Manyas Lake and the adjacent marshy areas comprise a rich

flora represented by many species such as common spike-rush [*Eleocharis palustris* (L.) Roem. & Schult.] and common duckweed (*Lemna minor* L.) [22].Today, the main human activity in the study area is agriculture; bread wheat, sun-

flower, maize, chickpea, melon, and watermelon are grown locally. Manyas Lake and several tributaries support agricultural activities by irrigation.

According to the Turkish Plants Data Service [23], in Anatolia, *Frangula* from the buckthorn family (Rhamnaceae) is represented by two subspecies, *Frangula alnus* Mill. subsp. *alnus* and *F. alnus* Mill. subsp. *pontica* (Boiss.) Davis et Yalt. The former occurs from northern, western, southern, and central to eastern Turkey, whereas the latter, which is endemic to Anatolia, occurs only in eastern Turkey. Based on location records by the Turkish Plants Data Service, *Frangula alnus* is not currently present in the Balıkesir Province where the study site Dascyleum is situated.

Historical background

We have eight cultural strata on the acropolis of Dascyleum (Tab. 1). Stratum VIII covers roughly the Bronze Age. The earliest findings from Dascyleum are dated back to the third millennium BC. Some pottery shards, stone axes, and "8" idols seem to confirm this dating. However, undisturbed Bronze Age layers are still absent. There

Tab. 1 Cultural strata of Dascyleum.							
Strata	Period	Dates	Cultures				
Ι	Modern	13th cent. AD – today	Ottoman/Turkish				
II	Medieval	12th-13th cent. AD	Byzantine/Seljuk				
III	Late Antiquity	4th–5th cent. AD	Roman/Byzantine				
IV	Hellenistic	334–100/50 BC	Macedonian/Bithynian				
V	Classical	546-334 BC	Persian				
VI	Archaic	700–546 BC	Phrygian/Lydian (?)				
VII	Early Iron Age	1200–700 BC	Phrygian (?)				
VIII	Bronze Age	3rd millennium BC	\$				

is no evidence for the second millennium BC from the settlement, except for an Old Babylonian seal. One may assume a hiatus in the settlement during the Bronze Age, ending in the Early Iron Age.

Sometime in the Early Iron Age (approximately 1200–700 BC) newcomers settled on the acropolis (upper city; Stratum VII). They were Phrygians, or another people who developed Phrygian cultural aspects. The acropolis was surrounded by a wall.

Stratum VI developed between approximately 700 and 546 BC. In this period, the Lydian culture penetrated into the city. Various imported pottery from the Aegean region was widespread in the city and its buildings were constructed on the layers of the previous stratum.

The earliest tumulus (570–530 BC) hitherto excavated at Dascyleum is contemporaneous with Stratum V. This stratum came to an end in a fire, which may be associated with the arrival of Persians in 546 BC [24,25]. In this period, Dascyleum became a center of a satrapy of the Persian Empire. The Persian satrap was a governor who ruled a state, called satrapy. The city reached a peak during this period [26]. The archeological findings, including an archive building, shed light on the administrative aspects of the site [27]. Most of the large tumuli strewn around the Manyas Lake are from this period.

It is possible to divide Stratum V into three phases: c, b, and a, or late, middle, and early. Stratum Vb ended in a destruction layer, which may be associated with the attack by the Spartan commander Agesilaos [28]. A new architectural activation emerged in phase Va after the destruction. New buildings with new plans were reconstructed on the debris. Under the Persian rulers, Phrygians, Lydians, and other ethnicities continued to live in Dascyleum [26]. This stratum ended with the arrival of the army of Alexander the Great.

During the period of Stratum IV, the newcomers reused the stones of the destroyed Achaemenid buildings for their own structures. A road reaching the acropolis, several houses, and a megaron (great hall of palace complexes), which was probably built for the new Macedonian satrap, are among the most remarkable architectural features of this period. The tradition/custom of burying the elites in tumuli continued. This period is roughly the Hellenistic period and ended in the first century BC.

Small findings from Late Antiquity (fourth and fifth centuries AD) are not absent in Dascyleum [29], but the architectural remains of Stratum IIId were not discovered until now. One may assume the presence of a Late Roman farm somewhere on the hill.

Stratum II is mainly medieval. In the 12th century AD, a Byzantine castle was constructed against Turkish attacks on the top of the hill, reusing the stones of the ancient buildings. The castle was active in the 12th and 13th centuries [29], after which Dascyleum was abandoned forever.

The latest stratum (Stratum I) is the Turkish epoch and is represented by shepherd houses.

Material and methods

Archaeobotanical analyses

Carbonized plant remains from several contexts of various cultural levels in the trenches, such as building floors and pithos (large storage jar), were collected by the excavation team. A total of 19 archaeobotanical samples were handled. Dates for these samples have been estimated, based on the associated archeological finds. Sample numbers have been given by the present authors according mainly to the chronology of the contexts.

The plant remains were identified and the well-preserved remains were measured with a stereomicroscope. Photographs were taken by a digital camera connected to the stereomicroscope. The remains were identified using the reference collections in the Department of Biology at Hacettepe University. The remains of buckthorn were also compared with modern fruits and stones/pyrenes of herbarium specimens of the genera from the Rhamnaceae family found in Turkey. These include the species of *Paliurus* Mill., *Zizyphus* Mill., *Sageretia* Brongniart, *Frangula* Mill., and *Rhamnus* L. deposited at Hacettepe University Herbarium (HUB).

For nomenclature of barley, collective name *Hordeum vulgare* L. and for wheat and pulses, traditional classification given in Zohary and Hopf [30] were used. For nomenclature of the buckthorns (Rhamnaceae), the Turkish Plants Data Service (TÜBİVES) [23] was followed.

Tab. 2HPLC analysis is performed using the following elution.

	Time	(min.)							
	0.0	1.0	20	25	28	33	35	40	45
H ₂ O – 0.1% TFA (v/v)	95	95	70	40	40	5	5	95	95
CH ₃ CN – 0.1% TFA (v/v)	5	5	30	60	60	95	95	5	5

Archaeometrical analyses of Dascyleum buckthorn pyrenes by HPLC-PDA

For archaeometrical analyses of the Dascyleum buckthorn pyrenes retrieved from a pithos (from Stratum II; medieval, 12th–13th centuries AD), HPLC-PDA was used.

Chromatographic experiments were performed using an Agilent 1200 series system (Agilent Technologies, Germany; computer hardware: Hewlett Packard, USA) including a G1329A ALS autosampler and a G1315A diode-array detector. Chromatograms were obtained by scanning the sample from 191 to 799 nm with a resolution of 2 nm and chromatographic peaks were monitored at 255, 268, 276, 350, 491, 520, 580, and 620 nm. A G1322A vacuum degasser and a G1316A thermostated column compartment were used. The data were evaluated with Agilent Chemstation. A Nova-Pak C18 analytical column (3.9×150 mm, 4 mm, part number WAT 086344; Waters, USA), protected by a guard column filled with the same material, was used.

Analytical and guard columns were maintained at 30°C. Chromatographic separations of the hydrolyzed samples were performed using a gradient elution program using two solvents: A, H_2O : 0.1% trifluoroacetic acid (TFA) and B, CH_3CN (acetonitrile) : 0.1% TFA. The flow rate was 0.5 mL/min and the applied elution program is described in Tab. 2.

Sample preparation for HPLC analysis

Buckthorn pyrene samples were hydrolyzed in 400 mL of a solution mixture of 37% HCl : MeOH : H_2O (2:1:1, v/v/v) in conical glass tubes for exactly 10 min in a water bath at 100°C to extract organic dyestuffs. After rapid cooling under cold running water, the solution was evaporated just to dryness in a water bath at 55–65°C under a gentle stream of nitrogen. The dry residues were then dissolved in 200 mL of MeOH : H_2O (2:1, v/v) and centrifuged at 4000 rpm for 10 min, and 100 µL of the supernatant was injected into the HPLC equipment.

Results

The results of the analyses of the plant remains are presented in Tab. 3–Tab. 6 and Fig. 2. Most of the archaeobotanical samples are represented in Stratum II. Four periods are represented: one sample from Stratum VI (700–546 BC), one sample from Stratum V (546–334 BC), two samples from Stratum IV (334–100/50 BC), and 15 samples from Stratum II (12th–13th century AD).

The archaeobotanical assemblage contains cereals, pulses, and buckthorn (Fig. 3a-h). Some of the pulse seeds were found to had been infested by pests (Fig. 3i). The dimensions of the well-preserved plant remains are given in Tab. 7. The HPLC-PDA analyses of buckthorn pyrenes from the site revealed traces of mordants (Fig. 4a-e).

Botanical and entomological remains

Cereals. On the site were found only grains of these species: barley (*Hordeum vulgare* L.; Fig. 3a) and free-threshing wheat; bread/durum/rivet wheat (*Triticum aestivum* L. / *T. durum* Desf. / *T. turgidum* L.; Fig. 3b).

All grains of barley were hulled and symmetrical, suggesting two-row barley. The identification of the free-threshing wheats, bread, durum, and rivet wheat, to the species level was based on chaff remains. Because there was no chaff material in the

Tab. 3Plant remains from Dascyleum.					
Sample No.	1	2	3	4	5
Trench	Ι	F5	T21-S21	24	BS
Context	Building B, floor	Ashy layer, N	Building A, floor	Floor, NE	Rampart N, burnt layer
Stratum	VI	Vb	IV	IV	II
Period	Archaic, 7th– 6th cent. BC	Classical, early 4th cent. BC	Hellenistic, 3th cent. BC	Hellenistic, 3th cent. BC	Medieval, 12th-13th cent. AD
Cereals (grain)					
Hordeum vulgare – barley		4			2
<i>Triticum aestivum/durum/turgidum –</i> bread/durum/rivet wheat		22			12
Pulses (seed)					
Cicer arietinum – chickpea				1	
Lathyrus sativus/cicera – grass pea	26				5
<i>Vicia ervilia</i> – bitter vetch		37			
Fruit plant					
<i>Frangula alnus</i> – buckthorn fruit			6		

Tab. 4 Plant remains from Dascyleum.					
Sample No.	6	7	8	9	10
Trench	BS Rampart	BS	BS	Archive	19c
Context	NW of Pithos 1	Pithos 1	Burnt layer	Building N, Pit I	Pithos
Stratum	II	II	П	II	II
Period	Medieval, 12th–13th cent. AD	Medieval, 12th–13th cent. AD	Medieval, 12th-13th cent. AD	Medieval, 12th–13th cent. AD	Medieval, 12th-13th cent. AD
Cereals (grain)					
<i>H. vulgare –</i> barley		1	4	15	
<i>T. aestivum/durum/turgidum</i> – bread/ durum/rivet wheat	1	1	11	2	
Pulses (seed)					
L. sativus/cicera – grass pea					
<i>V. ervilia</i> – bitter vetch	18	28			
<i>V. faba</i> – fava bean	1			10	
Fruit plant					
<i>F. alnus</i> – buckthorn pyrene	3				54

Sample No.	11	12	13	14	15
Trench	J-7	J-8	Southern Cultic Road	Southern Cultic Road	F6 Eastern Trench
Context	Burnt layer	Rampart, ashy layer	Pit 6	Pit 1	Hearth
Stratum	II	II	II	II	II
Period	Medieval, 12th–13th cent. AD	Medieval, 12th-13th cent. AD	Medieval, 12th–13th cent. AD	Medieval, 12th–13th cent. AD	Medieval, 12th–13th cent. AD
Cereals (grain)			·	·	
H. vulgare – barley		17			7
<i>T. aestivum/durum/turgidum</i> – bread/ durum/rivet wheat	1	25			18
Pulses (seed)					
<i>V. faba –</i> fava bean		45		22	
Fruit plant					
<i>F. alnus</i> – buckthorn pyrene			8		

Tab. 5	Plant remains from Dascyleum.
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Tab. 6 Plant remains from Dascyleum.				
Sample No.	16	17	18	19
Trench	J7	F7-F9	18a	F5, north
Context	Floor	Floor	Floor NW	Floor
Stratum	II	II	II	II
Period	Medieval, 12th–13th cent. AD	Medieval, 12th–13th cent. AD	Medieval, 12th–13th cent. AD	Medieval, 12th–13th cent. AD
Cereals (grain)				
<i>H. vulgare</i> – barley	7	22		7
<i>T. aestivum/durum/turgidum</i> – bread/ durum/rivet wheat	17	2		24
Pulses (seed)				
<i>V. ervilia –</i> bitter vetch			2	



Fig. 2 Total numbers of plant remains recovered from cultural strata of Dascyleum.



Fig. 3 Plant remains from Dascyleum. **a** Grain of barley. **b** Grain of bread/durum/rivet wheat. **c** Seed of chickpea. **d** Seed of bitter vetch. **e** Seed of grass pea. **f** Seed of fava bean. **g** Fruit of buckthorn. **h** Pyrene of buckthorn (raphe arrowed). **i** Infested fava bean seed with a borehole and an adult of bruchid beetle (arrowed). Scale bars = 2 mm.

····· I						
	Stratum	n	Length	Breadth	Thickness	Diameter
<i>Hordeum vulgare –</i> barley grain	II	19	5.2-6.08-7.2	2.9-3.61-5	2.4-2.9-3.3	
<i>Triticum aestivum/durum/turgi- dum</i> bread/durum/rivet wheat grain	IVb	22	4.9–5. 52–6.3	3.3-3.86-4.8	2.6-3.12-3.8	
<i>Cicer arietinum –</i> chickpea seed	IV	1	5	5	4.5	
<i>Lathyrus sativus/cicera</i> – grass pea seed	VI	26	3.8-4.48-5	3.6-4.17-5	3.2-4.74-6.2	
<i>Vicia ervilia</i> – bitter vetch seed	II	18	2.9-3.11-3.8	2.5-3.16-3.4	2.7-3.42-4	
<i>V. faba –</i> fava bean seed	П	22	5-6.25-7.2	5-6.21-7.5	8.5-10.08-12	
<i>Frangula alnus</i> – buckthorn fruit	IV	6				8.2-9.7-13
F. alnus – buckthorn pyrene	П	22	5.1-5.79-7	3.7-4.14-5		

Tab. 7 Dimensions of plant remains from Dascylium (in mm).

n – number of remains measured.



Fig. 4 a HPLC chromatogram of medieval buckthorn pyrenes from Dascyleum. **b**–**e** HPLC spectra of medieval buckthorn pyrenes from Dascyleum for mordant determination.

Dascylium samples, identification of the free-threshing wheat grains was limited to *T. aestivum/durum/turgidum*.

Pulses. The seeds of pulses belonged to chickpea (*Cicer arietinum* L.; Fig. 3c), bitter vetch [*Vicia ervilia* (L.) Willd.] (Fig. 3d), grass pea (*Lathyrus sativus* L. / L. *cicera* L.) (Fig. 3e), and fava bean (*V. faba* L.; Fig. 3f). Seeds of grass pea shaped like the head of an ax and almost triangular in cross section were designated as *Lathyrus sativus* / L. *cicera*.

Fruit plant. Drupaceous fruits and free pyrenes of buckthorn (*Frangula alnus*) were found at the study site (Fig. 3g,h). The circular fruits were severely deformed or flattened. One fruit was deliberately broken in order to extract its seeds. It yielded some pyrene fragments. The free pyrenes were obovate and slightly convex with fine longitudinal edge along ventral side, corresponding to raphe.

Archaeoentomological remains

Some of the pulse seeds from the medieval layers of the study site were infested by pests. In several bitter vetch and fava bean seed boreholes made by seed beetles were recorded. In some fava bean seeds bruchid beetles (Bruchidae) were found (Fig. 3i).

Traces of mordant in Dascyleum buckthorn pyrene remains

In the mordant analysis, four different peaks were determined by HPLC. The most pronounced peak, at 30.176, may represent emodin (Fig. 4a–e).

Discussion

Plant use

The present archaeobotanical analysis of the samples from Dascyleum gives some general information about plant use at the site in ancient times, ranging from the Early Iron Age to the High Medieval period (Strata VII–II).

The study area of Dascyleum is located in the fertile plains of northwestern Anatolia. Several sites of earlier periods, such as Neolithic/Chalcolithic Kumtepe of the Troad in Çanakkale [31] and Early Bronze Age (EBA) Yenibademli Höyük on Gökçeada [32] in the west, Neolithic Aktopraklık Höyük [33] and Late Neolithic – Early Chalcolithic Ilıpınar [34] near Bursa in the east, have yielded evidence of prehistoric farming activities in the region.

At Dascyleum, various crop plant remains of ancient Near Eastern agriculture, including cereals (barley and free-threshing wheat) and pulses (bitter vetch, grass pea, fava bean, and chickpea), were recorded. In addition to archaeobotanical findings, grinding stones found in Stratum II (medieval; 12th–13th centuries AD) provide evidence of agricultural activities and food preparation at the site. Also, large crop storage contexts, such as pithos and pits, recovered in Stratum II suggest dense settlement on the mound and extensive cultivation of crop plants in the fertile plains of the area. The agrarian economy of Byzantium consisted mainly of agriculture and animal husbandry; there was a surplus of some crops and large numbers of flocks [35].

The presence of crop plants through several cultural levels of Dascyleum suggests that agricultural activities continued during the historical periods of the site. Similar crop assemblages have also been reported from almost contemporary levels of other sites in Anatolia, for example Miletos (750–650 BC) in Aydın and Sardis (540 BC) in Manisa [36] in the west, Gordion (800 BC) in Polatlı – Ankara [37] in the north-center, Tille Höyük (700 BC) in Adıyaman [38] in the east, and Byzantine Beycesultan in Denizli in the west [39].

Barley grains are abundant in archeological contexts, often in kitchens, suggesting that barley was used as food in the past [40]. Today, barley is grown in many areas of the temperate regions as fodder and for beer making. However, barley appears to have assumed importance as food in Anatolia, even until the twentieth century. In the account of the Turkish agricultural system in the 1920s, Zhukovsky [41] noted that barley was of secondary importance after wheat, and was grown throughout Anatolia. At Dascyleum, wheat would have been used as food, while barley would have been used as food as well as for beer making and animal feed. There has been to date no archaeozoological investigation at the site.

The presence of bitter vetch, fava bean, and grass pea at the study site is of special interest because seeds of these pulses can be toxic after prolonged and intensive consumption. But it is assumed that their seeds were used by humans as food since prehistoric times only after adequate cooking to remove the toxin [42]. The intensive consumption of these pulses, particularly in the Near Eastern Bronze Age, may have been associated with cultural preferences and/or environmental factors [43]. In the agrarian economy of Byzantium, pulses were grown as part of the alternation of cultures [35]. Today, the seeds of bitter vetch are eaten only by very poor people or in times of famine, and the plant was grown mainly as a fodder plant at least since Roman times [30]. In Anatolia, powdered seeds are used to treat bone fracture, stomach ulcer [44], blood pressure, and diabetes [45]. Prolonged and intensive consumption of fava bean causes favism in people with a certain enzyme (G6PD) deficiency and that of grass pea causes lathyrism, a neurological disorder that can cause paralysis [46,47]. Today, cooked pods and seeds of fava bean are consumed as food, especially in western Anatolia, and its flowers are used to reduce kidney stones [48]. Assyrian medical texts mention the use of grass pea seeds in the medical prescriptions, whereas late lexical texts mention the use of its seeds as fodder [8].

Another pulse type recovered at Dascyleum is chickpea, seeds of which have also been consumed by humans since prehistoric times. For example, seeds were found in Late Bronze Age Troad [31] and Assyrian Kaman Kalehöyük (1900–1700 BC) (Kırşehir) in Central Anatolia [49].

About the buckthorns, only a single pyrene of *Rhamnus* was recovered from EBA Tell Tayinat (3300–2200 BC) (Hatay) in southern Turkey [50]. However, previous evidence of buckthorns in Anatolia is known mostly in the form of wood charcoal from several sites in the east: for example, Early Neolithic Hallan Çemi [51] and Pre-Pottery Neolithic Cafer Höyük [52], EBA Kurban Höyük [53], medieval Aşvan Kale [54], and Gritille [55]. At Dascyleum, buckthorn wood charcoal has not been found.

Various buckthorn species were in fact economically and medicinally important in the prehistoric and historic periods of Anatolia [48,56–58]. The fruits with seeds have been used mainly as a laxative, owing to their 3-7% anthraquinone content, and as a source of natural dye containing various substances, including emodin, isorhamnetin, rhamnetin, and quercetin. The dried and stored bark has been also used in other ways, including as a laxative, antirheumatic, and diuretic [59]. In Central Anatolia, the fruits have been used to treat jaundice [60]. In the nineteen century, the city of Kayseri in east Central Anatolia became the most important center of buckthorn cultivation and fruit collection for dyeing textiles and carpets [61]. During this period, an average of 400 tons or more than 500 tons of buckthorn fruits were produced annually in the city, and most were exported to England, France, and the USA through the international harbors of Samsun in the north and İzmir in the west of Anatolia [48,61]. Dye production from buckthorn is still practiced, although locally, to make traditional handpainted scarves with a wide range of colors, called yazma in Turkish, in northern (the Black Sea Region), central (Ankara, Sivas, Yozgat, and Niğde), and east-central Anatolia (Kayseri and Kahramanmaraş) [62]. Charcoal from the wood was regarded as the best for the manufacture of gunpowder [63].

At Dascyleum, only the fruits and pyrenes of buckthorn have been found so far. The fruits have been recovered in the floor of a Hellenistic building (Building A), dating from the late fourth and early third century BC. Pyrenes have been retrieved from Stratum II; 54 pyrenes from a pithos, eight from a pit (Pit 6), and three from a pithos (Pithos 1), indicating storage, probably for dyeing and/or medicinal purposes.

Traces of mordant from buckthorn pyrenes

As mentioned above, the fruits and pyrenes of buckthorn contain mordants, including emodin, isorhamnetin, rhamnetin, and quercetin. Emodin is an anthroquinone, whereas the others are flavonoids, whose content declines within time. However, traces of these substances appeared in the spectra from HPLC-PDA analyses of Dascyleum buckthorn pyrenes, providing a chemical point of view for the ancient pyrenes and supporting the botanical determination.

Pulse seed pests

Among the pulse seeds recovered from the study site, some grains from medieval layers were infested by pests, namely bruchids (Bruchidae). In general, bruchids leave the seeds and the storeroom shortly after harvest, but occasionally their departure is delayed for several reasons: larva/adult may die naturally or emergence is delayed [64].

In case of the Dascyleum damaged pulse remains, the cotyledons with only boreholes (without pests) would represent the pests having left the seeds in the field, or in the storage location in the study site before the fire. In contrast, the presence of some fava bean (*Vicia faba*) seeds from the site with bruchid beetle adults suggest that these adults died before emergence owing to the fire on the mound, which carbonized both seeds and pests.

Earlier archaeological finds of infested pulses come from several sites in Anatolia: for example, Hacılar (5400 BC) (Burdur) [65] in the west, EBA İmamoğlu Höyük (Malatya) in the east [66] and EBA Yenibademli Höyük [67], EBA and Middle Bronze Age Troy [31] and the Old Hittite Period levels of Boyalı Höyük (1650 BC) in the north-center [67], and medieval Gritille [55].

Conclusion

From the present archaeobotanical results, it may be suggested that agricultural activities with a spectrum of Near Eastern crops and pulses continued during the historical periods at Dascyleum.

The main contribution of this study is the find of buckthorn (*Frangula alnus*) pyrenes, which are rare in archaeobotanical record of the Near East. In view of the folkloric use of buckthorn, the remains from Dascyleum are interpreted as storage, probably for use as a dye and/or medicine. In addition, the archaeometrical study of the Dascyleum buckthorn pyrenes by HPLC-PDA in this study provides chemical evidence for traces of the mordant emodin remaining to the present day and also supports the botanical determination.

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