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A Microfacies Toolkit for Revealing Linkages Between Cultural Discontinuities and Exceptional Geogenic Events: the Tell Da'de Case Study (NE Syria)

Marie-Agnès Courty · Eric Coqueugniot

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Abstract High-resolution multi-archive studies have been promoted to reveal abrupt discontinuities that would express rapid social reactions (days to decades) to sudden exceptional geogenic phenomena (climatic, volcanic, tectonic, geomorphic, or cosmic). We expose here how we have adopted this approach for examining the causal linkages between a series of cultural discontinuities encountered through the cultural deposits of Da'de (Syria) and cosmic airbursts. Cosmic events, known to be rare, erratic, and unpredictable, are traced by exceptional debris that formed from aerosols when violently compressed during entry through the earth atmosphere of hypervelocity cosmic collider. They consist of singular organo-mineral materials in the form of volatile-rich hydrocarbon fuel with resistant metal-rich carbonaceous composites and colorful polymers. The archaeological sequence comprises Da'de phases 0 to III that represent a nearly continuous accumulation for 1,000 years during the Pre-pottery Neolithic period (ca. 9,900–8,900 years BP) and two short-term occupation phases during the pre-Halaf (Da'de IV) and early Bronze Age periods (Da'de V). Study of the exceptional debris in virgin soils and occupation deposits at Da'de has allowed to distinguish an intact debris assemblage that pulverized at the ground following an airburst (type 1 natural singular signal) with the ones that have been processed by humans (type 2 anthropic singular signal). Type 1 singular organo-mineral materials were found intact at the contact of the virgin soil with the earliest Pre-pottery Neolithic A (PPNA) occupation layers (Da'de phase 0, area [SB]), at the top part of the virgin soil with the PPNA occupation layers (Da'de phase Ia,

M.-A. Courty (✉)
CNRS-UPR 8521 PROMES, Procédés et Matériaux Solaires. Rambla de la Thermodynamique, Tecnosud, 66100 Perpignan, France
e-mail: Marie-Agnes.Courty@promes.cnrs.fr

M.-A. Courty
Institut Català de Paleoecologia Humana i Evolució Social, Universitat Rovira i Virgil, Plaza Imperial, C/ESCORXADOR, s/n, 43003 Tarragona, Spain

E. Coqueugniot
CNRS-UMR 5133—"Archeorient", Maison de l'Orient et de la Mediterranee—Jean Pouilloux, 7 rue Raulin, 69365 Lyon Cedex 07, France

area [B]) and just under the lower slabs of the EB III/IV burials (Da’de phase V, area [SB]). This has allowed to establish a strict contemporaneity between cosmic airbursts and remarkable historical events: initial settlement at phase 0, establishment of the magnificent communal building at phase 1, EBIII/IV cemetery after a long period of abandonment. Evidence for the collection of the singular materials (type 2) and meticulous processing of the related hydrocarbon fuel in occupation floors, earthen construction, coloring materials, and objects have been traced from the initial settlement (phase 0) through the successive cultural periods. This correlation has revealed a long-lasting transmission of the value of memorial natural resources which were only available from time to time in the surroundings. The close timing between changes in the use of the singular organo-mineral materials (preparation techniques, types of by-products) and cultural phases has enabled us to identify social discontinuities which correlate to distinctive geogenic events and search of the related exceptional resources. Based on the long-term record from Da’de, cosmic airbursts are suggested to be considered as a new class of dual-discontinuous signal of historical dimension, which have led to sudden (days to months) geogenic shifts in the availability of singular fuel resources and, simultaneously, to rapid cultural responses in their exploitation and use, depending on reactivity of local landscapes to the induced changes at short time scales (decades).

Keywords Geogenic · Anthropic · Cosmic airburst · Event · Soil microfacies · Hydrocarbon fuel · Carbonaceous polymorphs · Polymers · Exceptional resources · Memory · Neolithic · Early Holocene

Introduction

Over the last two decades, revealing how sudden environmental shift due to climate, volcanic, cosmic, or geomorphic factors might have played a role on remarkable cultural discontinuities has greatly stimulated multi-disciplinary research efforts at the crossroads between anthropology and geosciences (i.e., Bar-Yosef and Belfer-Cohen 2002; Belcher and Belcher 2000; Diamond 2005; Jousse 2006; Nicoll 2004; Rosen 2007; Turney and Brown 2007; Zhang *et al*. 2007). Therefore, identifying the historical reality of these dual events by their related geogenic and anthropogenic manifestations, with a suite of diagnostic proxies, has remained a challenging perspective (Berger 2006; Holliday and Meltzer 2010; Meltzer and Holliday 2010; Rosen and Rivera-Collazo 2012). This critical issue is well illustrated by the debate on the 4-kyr BP exceptional aridity crisis that was identified in a certain range of soil-sedimentary records by a remarkable dust spike of large-scale extent and which was suggested to have led civilizations from the Mediterranean region to west Asia to a sudden decline (Weiss *et al.* 1993; Courty 1998; Cullen *et al.* 2000; Wang *et al.* 2004). In contrast, other records and proxies have provided the alternative perspective of gradual climate change with marked regional gradients (Marchant and Hooghiemstra 2004) along to an overall continuity of settlement pattern (Kuzucuoglu 2007a, b; Mercuri *et al.* 2011). Meanwhile, our refined characterization of the 4-kyr BP dust event has provided increasing evidence to reinforce its exceptional dimension both from natural and cultural perspectives. We suggested to explain the anomalous loose micro-aggregated soil facies of the 4-kyr BP dust layer, with its assemblage of singular micro-debris (carbonaceous species, melted clasts, metals), as the result of violent pulverization at the ground surface of atmospheric aerosols, which would have been possibly induced by a cosmic impact (Courty *et al.* 2008, 2012a). These natural records helped us to identify human transformation of the singular assemblage for deliberate purposes (Courty 2012b).

Within the same perspective, the exact nature of geogenic and cultural events that were recorded during the Younger Dryas and their possible linkages has, so far, remained highly controversial. This critical period has been for long viewed as a remarkable abrupt cooling and dust increase in the Northern hemisphere, showing complex regional expressions, without evidence of severe hazards, which would have stimulated the adaptive potential of highly mobile hunter–gatherer societies across the New and the Old World continents (Broecker *et al.* 2010; Ballenger *et al.* 2011; Strauss 2012). In contrast, the hypothesis of a catastrophic cosmic impact has been proposed to explain the sudden initiation of the Younger with severe consequences on ecosystems and humans: destabilization of the Laurentide Ice Sheet, extensive biomass burning with atmospheric disturbances, food deprivation followed by extinctions of Pleistocene megafauna, and a presumed termination of the Clovis lithic technology/culture (Firestone *et al.* 2007; Kennett *et al.* 2009; Anderson *et al.* 2011; Tian *et al.* 2011; Israde-Alcántara *et al.* 2012). From this view, the Younger Dryas has been presented as an extended stratigraphic discontinuity that was widely traced over North America and parts of South America, Europe, and Syria by a thin sedimentary layer, called the YD boundary layer, and well defined by anomalous materials just like impact by-products: glassy and iron-rich microspherules, siliceous scoria-like objects, and singular carbon forms, such as nanodiamonds, glass-like carbon, aciniform soot, fullerenes, carbon onions, and carbon spherules (Kennett *et al.* 2009; Tian *et al.* 2011; Israde-Alcántara *et al.* 2012; Bunch *et al.* 2012). However, the stratigraphic evidence for a unique major impact with devastation of natural resources and human populations at continental scale has faced severe refutation (Holliday and Meltzer 2010; Pinter *et al.* 2011). The lack of a significant gap or discontinuity in the radiocarbon record was concluded to indicate an overall continuity of the Paleo-Indian occupation (Buchanan *et al.* 2008). In the absence of the distinctive fingerprints left by large impacts, i.e., presence of crater structures in regions showing impact debris or shocked materials, the Younger Dyras impact hypothesis was severely rejected (Boslough *et al.* 2012). The singular carbon forms were suggested to have possibly derived from contamination by modern carbon or from an unknown source that could not be related to impact processes, due to the widespread occurrence of similar nanodiamonds in carbon spherules within surface soils in Belgium (Tian *et al.* 2011).

Meanwhile, our high-resolution study of Neanderthal occupation surfaces and combustion deposits from various regions has allowed us to identify an assemblage of remarkable components (glassy debris, native metals, and carbonaceous polymorphs) that is sharing certain similarities to the singular materials of the 4-kyr BP dust layer and of the Younger Dryas Boundary layer (Courty 2012a). The structural and chemical properties of graphitic vitreous carbon and polymer species, which so far have not been reported in archaeological or geological deposits, lead us to question the exact origin of these recurrent singular materials with their intriguing hydrocarbon by-products. The surprising occurrence of highly resistant pyrolytic carbon and high-temperature minerals that were not compatible with common human or natural firing products leads us to envisage a possible connection between ancestral pyrotechnology and the occasional delivery by cosmic airbursts of exceptional hydrocarbon fuel (Courty 2012a). The physical processes and environmental consequences of cosmic airbursts that are known to have recurrently occurred along the course of human history (Boslough and Crawford 2008) yet remain to be clarified by appropriate research on natural records. Meanwhile, we intend here to further elucidate the possible link between exceptional geogenic phenomena that would be defined by the occasional delivery of singular natural resources, most likely in response to cosmic events, and cultural discontinuities.

The study is based on the high-resolution facies study of microstratified occupation deposits that we have developed to decouple the interfering signals of natural and anthropic processes in archaeological deposits. We have selected the sequence of Da'de in northern Syria (Fig. 1) that is representative of a large number of situations encountered in archaeology, with regard to two aspects: (1) considerable data can be

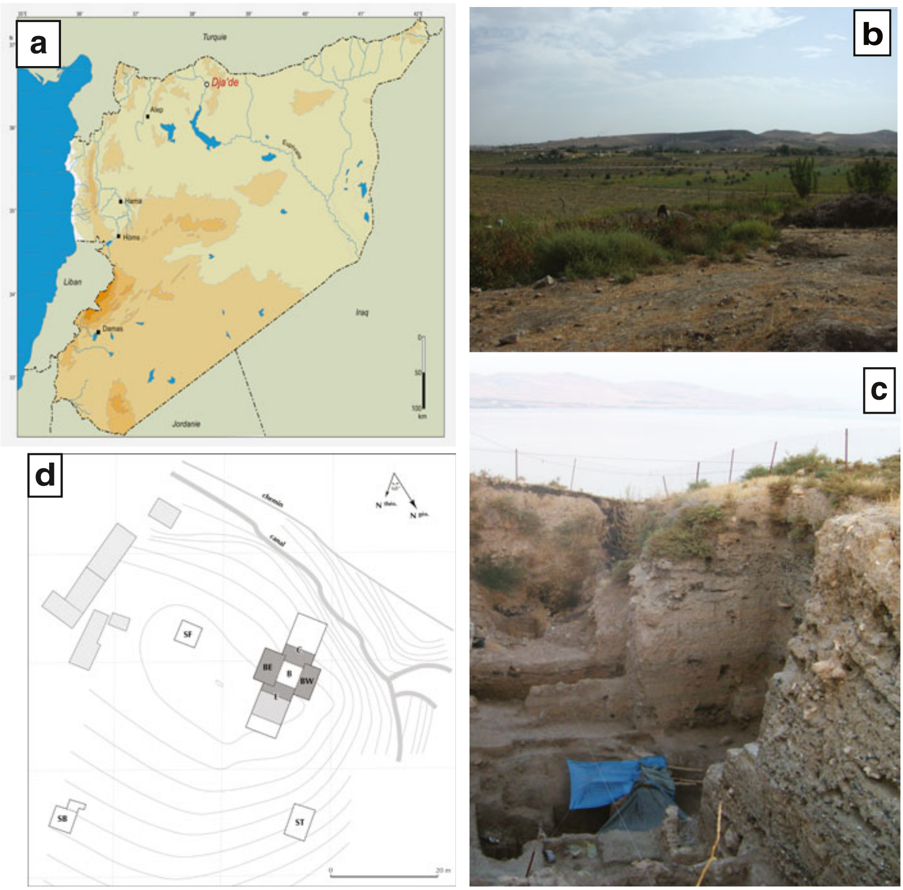


Fig. 1 **a** Location map of Da'de el Mughara. **b** West view of the tell showing the shallow contact with the surrounding flood plain formed by an ephemeral stream. **c** East view from the tell showing the excavated sequence from area [B] and [C] and, in the back, the dam on the Euphrates River. The *blue plastic cover* indicates the position of the painted construction and position of the virgin soil 2.5 m lower. **d** Location of the excavated areas; in *gray*, the ones mentioned in the text

gathered at one location, but there is limited possibility of carrying out regional correlation; (2) diverse occupation rhythms are clearly expressed both in the stratigraphic records and in the characteristics of the material culture, i.e., one millennium continuous occupation with gradual shifts, long abandonments, and short occupation periods.

We focus on the two most challenging issues raised by the Da'de sequence: how to define the diagnostic signatures of a particular type of exceptional geogenic events, possibly the ones of cosmic origin, in cultural deposits, and how to establish a possible causative linkage between these natural phenomena and the tempo of cultural changes.

Context, Materials and Methodology

The Da'de Site

Twenty years of excavation at Da'de (Northern Syria) that is located on the west bank of the Middle Euphrates (Fig. 1) river has provided a comprehensive perception of the cultural sequence and of the site configuration in its surroundings floodplain (Coqueugniot 1999, 2000, 2011). The first cultural layers (ca. 9,700–9,500 years BP, Table 1) that are part of the PPNA, defined as Da'de I, is part of early agriculture and management of natural resources (Miller 2011; Zeder 2011) by proto-sedentary communities which were seasonally established in villages with circular architecture (Stordeur *et al.* 1982; Stordeur 2000). The Da'de I phase has revealed a circular central building formed of three massive structures/peers in the form of bull heads with a geometric polychromic motif (Fig. 2), which aggregates a dense imbrication of common houses that are built upon the soil at its periphery (Coqueugniot 2011). This construction is a remarkable example of the exceptional architecture that is encountered throughout the PPN period, which is assumed to have implied a collective effort in the direct continuity of the previous Natufian period (Valla 2003; Edwards *et al.* 2004; Stordeur 2006; Watkins 2008). The Da'de I phase ends with partial destruction of the painted walls which rose above the external floor, deposition of a human burial directly on the last soil, a careful filling of the entire circular structure, then an extended sealing by a thick pise floor. This distinctive transition to the Da'de II phase is followed by an architectural change with the construction of small-sized earth-made quadrangular households that are associated with a sequence showing the alternation of finely stratified living floors and destruction/remodeling episodes. Da'de (DJ) II phase (9,500–9,300 years BP), which corresponds to the Early Pre-pottery Neolithic B (EPPNB), shows a great diversity of combustion structures and, particularly, a series of thick pit deposits of auroch and equid burnt remains, which could possibly relate to ritual feastings. The transition to Da'de III phase (ca. 9,300 years BP) is marked by a different spatial pattern with small-sized rectangular rooms that are separated by open activity areas and the construction of a small, but unique, rectangular building that was frequently repaired. A remarkable change is shown by the close association of funerary activities to the habitation, with the repeated burials of human remains below living floors that comprise primary and secondary burials of more than 70 bodies, and isolated skulls in the so-called *House of the Dead* (Coqueugniot 2000).

Table 1 ^{14}C Radiometric dating of the Tell Da'de sequence

	Lab. no.	Date BP	Cal. BC	Phase	Provenance
33	Beta 268168	9,560±50	9,180–8,750	DJ0	DJ09-B-850 virgin soil
34	LTL 3332A	9,562±65	9,210–8,740	DJ I	DJ07-SB-bottom (below the initial occupation)
2	Ly-2603 (Poz)	9,560±65 (AMS)	9,214–8,652	DJ I	DJ02-ST-C6c (above the virgin soil)
50	Lyon-7795 (GrA)	9,540±45 (AMS)	9,146–8,748	DJ Ia	DJ10 B-x1 (virgin soil, prof. -945)
40	Lyon-4704	9,480±60 (AMS)	9,123–8,626	DJ I	DJ07-BW-G16 (lower filling of the painting house)
3	Ly-2604 (Poz)	9,780±65	9,307–9,167	DJ I	DJ02-B-US32 (accélérateur) (filling of the painting house)
42	Lyon-4706	9,590±60 (AMS)	9,227–8,761	DJ I	DJ07-SB-C14
43	Lyon-6902 (GrA)	9,360±45 (AMS)	8,746–8,492	DJ I	DJ09-Bs-v5 US 666 (z=850)
45	UBA-16007	9,556±34 (AMS)	9,091–8,841	DJ I	DJ09-Bs (z=850) US 666/27-behind massif 666
46	UBA-16008	9,530±39 (AMS)	9,088–8,808	DJ I	DJ09-BE (z=850) St. 668-before massif 669
5	Ly-12110	9,570±50	9,207–8,744	DJ I–II	DJ02-ST-w4-fosse450
6	Ly-11329	9,480±50	9,155–8,630	DJ I–II	DJ01-B-K1 (above the painting)
7	Ly-14032	9,420±45	8,791–8,611	DJ II	DJ06-BW-F1a (post of the burnt house)
8	Ly-14033	9,220±60	8,618–8,290	DJ II	DJ06-E-K5
9	Ly-13631	9,340±45	8,725–8,482	DJ II	DJ04-E-I5
49	Ly-15455	9,150±40	8,457–8,284	DJ II	DJ05 E-a1-K1 struct.549
10	Ly-13630	9,000±40	8,284–8,021	DJ II	DJ04-E-I2b
11	Ly-13629	9,440±45	8,812–8,621	DJ II	DJ04-E-I1e
12	Ly-11330	9,410±50	8,799–8,485	DJ II	DJ01-ST-C2b
13	Ly-13628	9,305±45	8,693–8,355	DJ II	DJ04-E-H4a
14	Ly-8842	9,370±75	8,794–8,196	DJ II	DJ96-Bsond-xy3-G3base
37	Ly-14515	9,155±40	8,463–8,284	DJ II base	DJ07-E-M3
38	Ly-15185	9,345±45	8,733–8,481	DJ II	DJ05-BW-F1a
39	Ly-15186	9,315±40	8,697–8,461	DJ II	DJ05-E-K1
41	Lyon-4705	9,380±60 (AMS)	8,787–8,487	DJ II	DJ07-C-I17a
47	UBA-16009	9,483±30 (AMS)	8,819–8,749	DJ IIb	DJ 01-B-x2/3-K4c
48	Ly-15454	9,320±45	8,703–8,462	DJ IIb	DJ10 F-E15b2 (hearth 736)
15	Ly-12112	9,290±45	8,686–8,336	DJ III	DJ02-CD-H6b
16	Ly-10849	9,210±95	8,716–8,244	DJ III	DJ00-ST-F3
17	Ly-6166	8,990±100	8,323–7,739	DJ III	DJ92-B-y3-F4
18	Ly-6164	9,070±220	8,831–7,579	DJ III	DJ92 B-x4-F4b
19	Ly-8841	9,280±60	8,413–8,108	DJ III	DJ95 B-x2-E4c
20	Ly-10845	9,175±55	8,543–8,270	DJ III	DJ00-C-G11b
21	Ly-11328	9,145±50	8,520–8,266	DJ III	DJ00-D-G11b
22	Ly-8844	9,245±65	8,394–8,089	DJ III	DJ95 B-y1/2-E3d
23	Ly-5820	9,540±290	9,128–8,262	DJ III	DJ91-C-x4-F5
24	Ly-8843	9,190±65	8,343–8,080	DJ III	DJ95 C-vw1/3-F5b

Table 1 (continued)

	Lab. no.	Date BP	Cal. BC	Phase	Provenance
25	Ly-5823	9,140±390	9,058–7,428	DJ III	DJ91-C-y4-F4
26	Ly-5822	9,160±75	8,356–8,028	DJ III	DJ91-B-y4-D5
27	Ly-5821	9,610±170	9,836–8,014	DJ III	DJ91-B-x4-D4
28	Ly-6165	9,100±80	8,338–7,976	DJ III	DJ92 B-x1/2-C4a
29	UtC-2367–2369	9,200±100 (AMS)	8,429–8,026	DJ III	DJ91 C-x4-F6
30	Ly-10846	9,250±55	8,625–8,292	DJ III	DJ00-D-H2c
31	Ly-10847	9,210±55	8,601–8,287	DJ III	DJ97-SF-D1
32	Ly-10848	9,150±55	8,528–8,265	DJ III	DJ00-SF-F11a2

The homogeneity of the material culture encountered in the different parts of the village and the coherent series of ^{14}C dates (Table 1) show the general extension of the three phases (Da'de I to III), from Late PPNA to the end of EPPNB, throughout the mound since the village was established on virgin soil. The abandonment of the village at the end of Early PPN (ca. 9,100 years BP) ends a nearly continuous sequence for approximately 1,000 years of seasonal occupation and gradual evolution of the material culture, activity patterns, and life styles. After a long break of occupation during Middle and Late PPNB, evidence for a new occupation of the site during the Early Neolithic has been encountered in the central part of the site by a series of architectural phases with an abundant pre-Halaf ceramic assemblage. This pre-Halaf occupation is represented by finely plastered pits that were dug into the PPNB without damaging the deeply buried monumental constructions, although the deeply buried buildings were no longer visible at that time.

An Early Bronze Age cemetery dated of the EB III/IV periods, which sealed the early PPNB layers on the eastern slope of the site, marks the ultimate evidence of ancient human presence on the site (Fig. 1d). The excavation performed on one nearly intact individual tomb, formed of large stone slabs, revealed the presence of funerary materials in the form of full shaped ceramics and a copper object close to the human body (Coqueugniot *et al.* 1998).

Studied Materials and Methodology

The study focusses here on microfacies analyses (Courty 2000) and characterization of soil-sediment properties (Courty 1998; Courty *et al.* 2008) from the cultural sequences that were successively exposed by the excavation conducted since 1992, and of the associated natural deposits. For the latter, particular attention was devoted to the virgin soils that appeared at 8.5 m depth in three distinctive areas below the PPNA cultural layers (Fig. 1d), and to a cumulative sequence showing the alternation of flood deposits and weakly developed soils that was revealed by a series of soundings at the periphery of the mound.

High-precision sampling at the scale of the finest microfacies—natural or anthropic—recognized during the excavation, has been performed both vertically and laterally, as previously defined by Courty *et al.* (2012b). The collection

Fig. 2 Field views from area [B]. **a** General architecture of the exceptional structure after partial removal of the painted constructions: the *yellow arrow* indicates the earliest phase Ia occupation floor and the lowermost earthen construction; the *red* one indicates the position of the painted wall of the monumental construction; the *purple* one marks the virgin soil in which the monumental construction was dug during phase Ia. **b** Bottom of the PPN sequence, just above the virgin soil showing the succession of flooded layers and painted floors with sharp contact (indicated by *red arrows*); the *upper green arrows* mark the sealing occupation floor at the phase I/II transition. **c** Spatial view of a highly degraded painted floor; the *reddish arrows* indicate spots of red pigments. **d** View showing the skeletal part of the exceptional building (*red arrow*) dug into the top virgin soil (*purple arrow*). **e** Detailed view of the top virgin soil showing concentrations of heat-dried *Arnebia* seeds (Boraginaceae family, steppic herbaceous plant) in a soft top soil with a type 1 microfacies and assemblage. **f** View of one of the polychromic painted constructions. **g** Schematic plan of the exceptional building with the polychromic painted constructions interpreted as a circular juxtaposition of three horned bull skulls

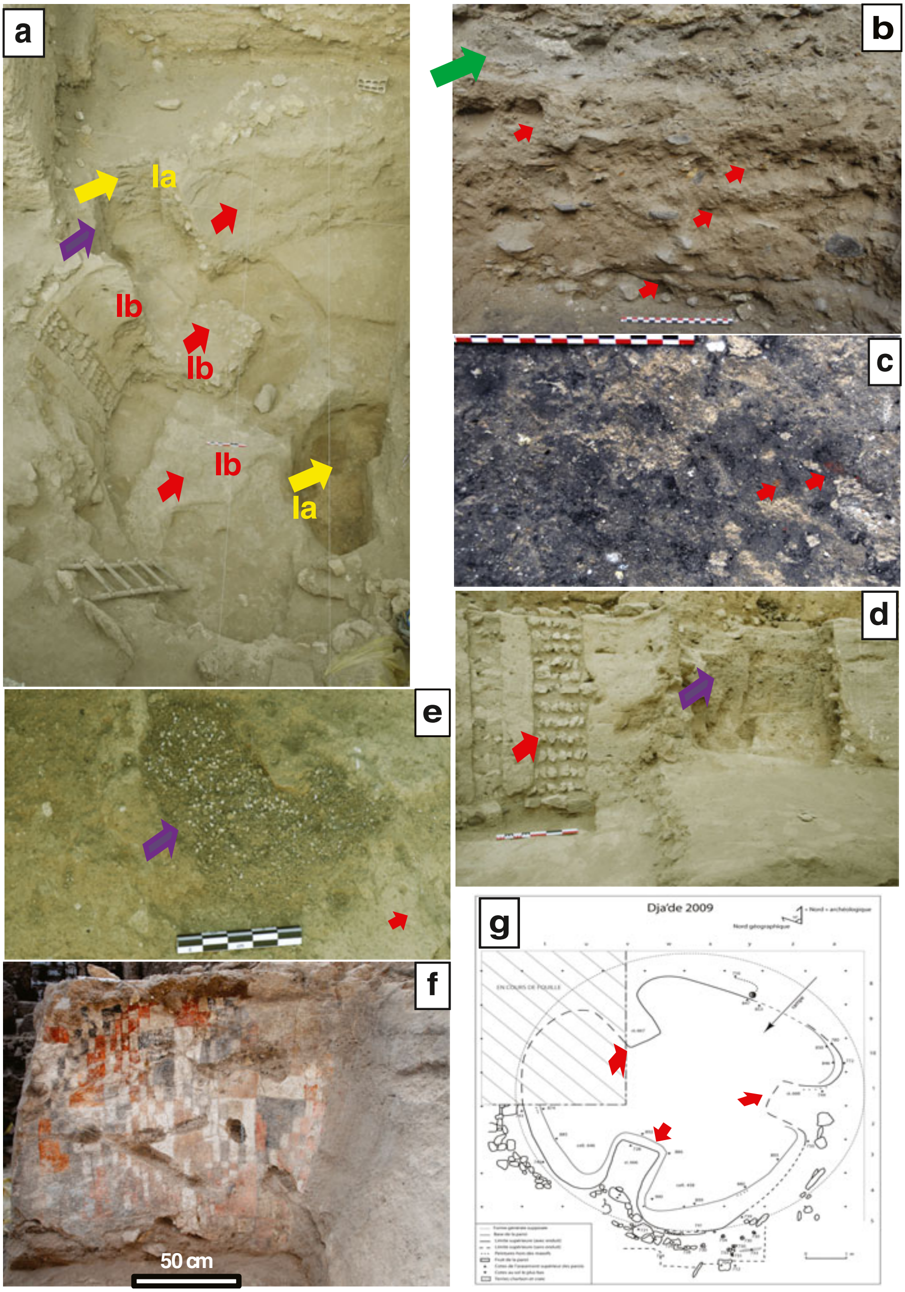
of 220 large-sized thin sections, which represent the entire sequence, has been used to distinguish in the field the stratigraphic reality of the finest anthropic event from the polyphased occupation surfaces (Gé *et al.* 1993). This help is particularly important at Da'de due to the rapid drying of the sediments that makes it difficult to widely expose individual living floor while excavating.

Selective microsampling of the bulk matrix was performed throughout a series of occupation contexts and of the related structures (Table 2). Water sieving was conducted for individual microfacies by separating five particle-sized fractions: >2 mm, 2–1 mm, 1 mm–500 μm, 500–250 μm, 250–100 μm. The coordination of the excavation and of the sorting of the water-sieved fractions under the binocular microscope has allowed to repeatedly adjust microsampling strategies to the stratigraphic complexity of the archaeological contexts. The structure and compositional range of singular components were studied by using an environmental scanning electron microscope coupled to an energy dispersive X-ray spectrometer (ESEM-EDS). The analytical data have been interpreted by comparison to our previous multi-analytical characterization of similar components (Courty *et al.* 2012b).

Results

Natural and Anthropic Singular Signals

The high-resolution microstratigraphic analysis carried out on the different deposits of Da'de has enabled us to trace the occurrence in natural and cultural deposits of unusual organo-mineral components. The latter are similar to the singular assemblage of organo-mineral materials that we previously suspected to be of cosmic origin (Courty *et al.* 2008). Marked contrast in the microfacies and spatial extent of the host soil/sedimentary matrix and in the compositional range of the singular organo-mineral materials has allowed to distinguish natural singular signals (type 1) from anthropic singular ones (type 2). The type 1 signals refer to the assemblages of unusual organo-mineral materials that were deposited by geogenic processes, which range from the intact records to the reworked ones. The first ones correspond to soil surfaces that originally recorded the thermal spray at the ground of the high-velocity pulverized terrestrial debris. They display the unusual carbon-vaporized soil



microfacies with their full range of well-preserved singular components (native metals, carbonaceous polytypes, and mineral grains) that are finely integrated to the host matrix (Courty *et al.* 2008; Courty 2012a, b). The reworked natural singular signals only display relictual features of the original ones, i.e., fragmented domains of the unusual carbon-vaporized soil microfacies, and/or a biased range of singular

Table 2 Anthropic type 2 exceptional organo-mineral assemblage in the cultural contexts of the Tell Da'de sequence

Occupation phase–location	Cultural contexts	Exceptional organo-mineral assemblage and micro-facies
Da'de 0, I (DJ 0-I)–PPNA area B, ST, SB 9310/Cal BC	Occupation floors/residual paintings. Construction materials. Hearths/combustion residues	Translucent carbonaceous filaments. Vitreous carbon rare earth phosphates–$BaSO_4$–metals/phosphatic fine fraction
Da'de I (DJ I)–PPNB area B, ST, SB 9310–8830 Cal BC	Well-prepared occupation floors and polychromic paintings of the exceptional circular building. Construction materials	Colored and translucent carbonaceous filaments; vitreous carbon. Rare earth phosphates–$BaSO_4$–metals/silicate–carbonates–sulfate–breccia clasts
Da'de II (DJ II) PPNB/8,800–8,500 Cal BC	Occupation floors/residual paintings. Construction materials. Hearths/combustion residues	Raw and polished exotic stones Translucent carbonaceous filaments. Vitreous carbon.
Da'de III (DJ III) PPNB/8,540–8,290 Cal BC	Occupation floors. Construction materials. Hearths/combustion residues. Ochered skull	Raw and polished exotic stones Translucent carbonaceous filaments. Vitreous carbon. Rare earth phosphates–$BaSO_4$–metals/ochre
Da'de IV (DJ IV) early 7th millennium pré-Halaf	Occupation deposits, ceramics	Raw and polished exotic stones Translucent carbonaceous filaments. Vitreous carbon.
Da'de V (DJ V) mid-3rd millennium BC Early Bronze III/IV	Ceramic fillings in the individual burials	Translucent carbonaceous filaments. Vitreous carbon. Rare earth phosphates–$BaSO_4$–metals/silicate–carbonates–sulfate–breccia clasts

components due to their differential preservation when the vaporized surface was exposed to post-depositional processes. The type 2 singular signals correspond to a vast range of cultural deposits, i.e., prepared floors, construction materials, combustion residues, and dumped deposits, which display all the typical characteristics of anthropic microfacies (Courty *et al.* 1989) with a biased range of the unusual components. Below, we present some of the main characteristics for each type before questioning their interference along to the occupational sequence at Da'de.

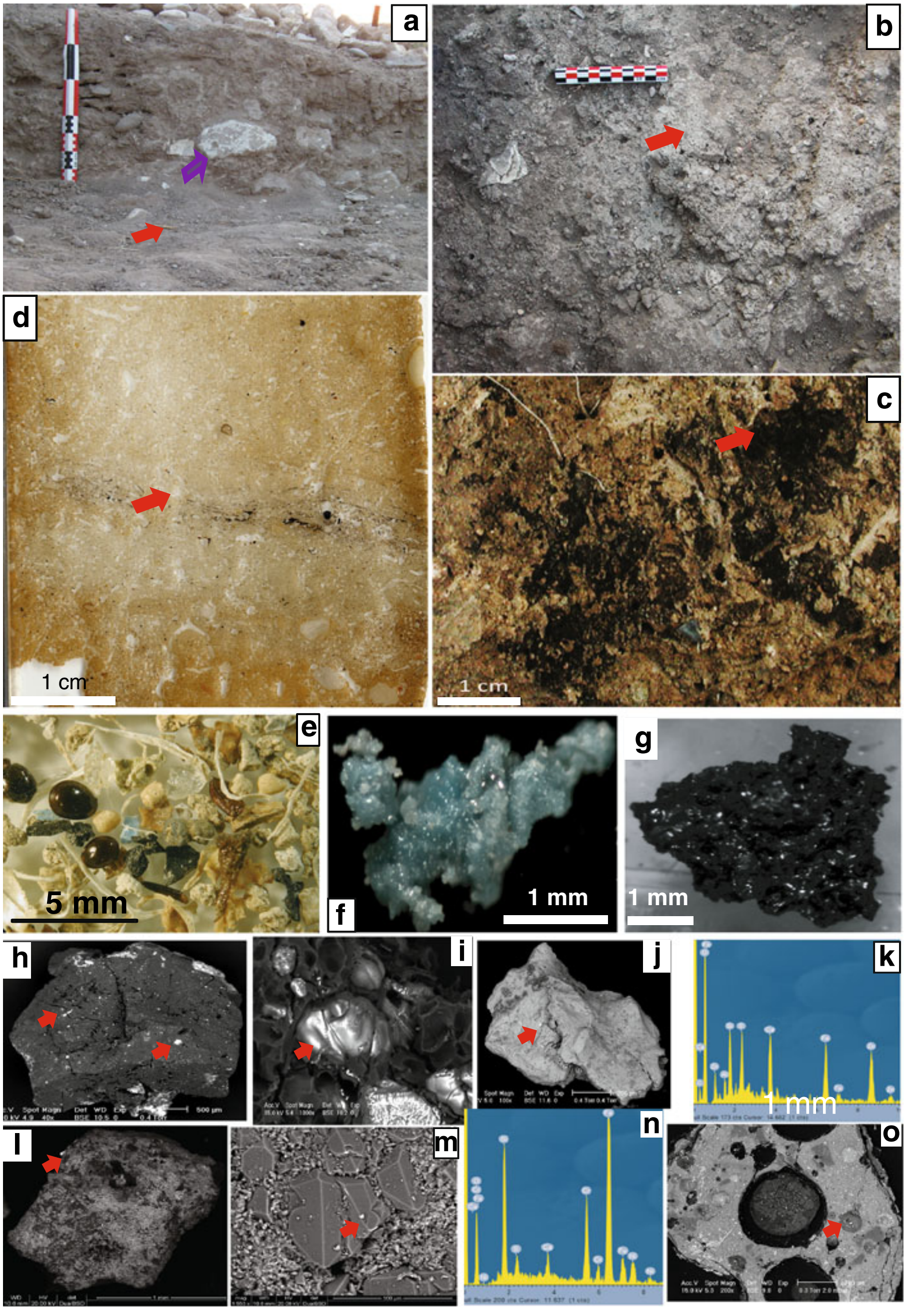
Context, Facies Pattern, and Characteristics of the Intact Type 1 Signal

Three situations with nearly intact records of type 1 natural exotic signals were identified during the excavation: (a) in area [SB], associated with the Early Bronze Age III/IV burials (Figs. 3a, b); (b) in area [B], at the contact of the virgin soil with the PPNA occupation layers, defined as Da'de phase Ia (Fig. 2a); and (c) in area [SB], at the contact of the virgin soil with the earliest PPNA occupation layers (Da'de phase 0, Figs. 5a, b). A test over 4 m^2 revealed the remarkable continuity of the Bronze Age III/IV type 1 dark carbonaceous surface in area [SB] that is interrupted only here and there by root channels and ant galleries. Its darkening is linked to the occurrence of a shiny graphitic thin film with a discontinuous microstratification (Figs. 3c, d). Careful examination revealed its penetration throughout the underlying top soil as thin coatings on soil microaggregates, roots channels, and ant galleries. This top soil that developed within the last PPNB occupation layers, after abandonment of the site, displays a thin slaking crust which was formed by long-term aerial exposure. The fine penetration of the graphitic film gives an impression of a gradual contact from the section, although the surface when exposed in the field was forming a distinctive stratigraphic discontinuity. The latter is fully confirmed by the occurrence of a singular assemblage of uncommon organo-mineral components, mostly concentrated within the carbonaceous surface: carbonaceous spherules, polymer agglutinates, colored to translucent polymer filaments, vesicular vitreous carbon, angular quartz and fine sandstones showing metal and carbonaceous sprayed deposits, glass shards, yellowish gray scoria-like glassy bead-shaped clasts, and igneous-like breccia with metal and carbonaceous inclusions (Figs.3e–o). This assemblage contrasts with the basic components of the local soils and is totally absent in the underlying archaeological strata. The range of carbonaceous polymorphs and their link to the host matrix suggest that they originated by pulverization at the soil surface of a debris jet with volatile and solid components deriving from hydrocarbon fuel and associated aerosols. These forming conditions are considered to express the very first manifestation that would be recorded at the soil surface during a cosmic airburst. The lack of suitable charcoal has not allowed us to obtain a reliable ^{14}C date. However, the painstaking excavation of the carbonaceous surface has shown its remarkable preservation just under the lower slabs of the Early Bronze Age III/IV cemetery and more evidence of degradation by roots and ants where the slabs are not present. These observations suggest that the type 1 signal with its carbonaceous microfacies formed at around 4 kyr BP, just before the Bronze Age III/IV occupation, and was rapidly sealed by the graves.

The Late PPNA type 1 signal was revealed in area [B], at the top part of the virgin soil in which the monumental building with its exceptional painting was partly excavated (Figs. 2a, d, e). It occurs as a weakly compacted thin yellowish brown carbonaceous strata formed by a dense network of fine root channels, locally interrupted by large root channels with a dark brown loose microaggregated infilling. The excavation on 1 m^2 has revealed concentrations of well-preserved seeds of *Arnebia* showing a calcinated external part and a partly carbonized inner part (Fig. 2e). Their occurrence in the root channels showed that the weeds were part of the natural vegetation which was covering the mound at the time of the first PPNA occupation. The presence of a singular assemblage consisting of uncommon organo-mineral components was detected in the thin compacted strata and in larger amounts in the filling of the root channels: polymer filaments, vitreous carbon, marine clasts, glass shards, angular quartz and sandstones, and scoria-like spherules (Fig. 4a). This assemblage contrasts with the basic components of the underlying local soils. The overlying deposits (Da'de phase Ia) are formed by anthropic facies showing the succession of prepared mud floors and trampled units with scattered fragments of construction materials, charcoal, flints, and bones. In area [SB], the type 1 signal was identified as a dark brown carbonaceous surface sealing the virgin soil, just at the interface with the initial PPNA occupation layers (Fig. 5a). In contrast to the area [B], the latter, defined as Da'de 0, which are weakly trampled anthropic facies without evidence of well-prepared mud floors or buildings, appeared to have formed from the decay of small earth-made structures associated to firing activities (Fig. 5b). The first

Fig. 3 Area [SB], top part of the tell, surface below the Bronze Age III.IV burial (*red arrow*); the *purple arrow* indicates the stone slab on the top of one burial. **a** Field view at the top of the tell after removal of the burials sealing the carbonaceous surface (*red arrow*). **b** Spatial view showing the exposed carbon-vaporized surface (*red arrow*). **c** Distinctive black patches formed of carbonaceous concentration. **d** Scan from the thin section showing the distinct, well-preserved, finely laminated, black surface resulting from charring of the local vegetation by the airblast; the fine bioturbation of the overlying and underlying soil layers (channel microstructure) has not disturbed the carbonaceous surface. **e** View under the binocular of the water-sieved fraction. **f** Binocular view of a crumby blue polymer resulting from thermal cracking of hydrocarbon fuel. **g** Binocular view of a vitreous black carbon resulting from thermal cracking of hydrocarbon fuel; note that this type of component seems morphologically similar to the so-called "fat-derived char" described by Ligouis (2006) or Goldberg *et al.* (2009); however, the chemical and structural characteristics presented in Courty *et al.* (2012b) have established its graphitic nature and its origin from the singular volatile-rich hydrocarbon fuel. **h** ESM-BSE view of the **g** type grain; the *red arrows* indicate the metal inclusions. **i** Detailed ESM-BSE view showing calcium phosphide trapped in the degassing vesicles that is a very original feature indicating formation under highly reduced conditions. **j** ESM-BSE view of an angular quartz showing highly resistant carbon-rich metal splash that formed by hyper-velocity sputtering of molten metals on solid debris of the debris jet formed by the airblast, defined as thermal splash. **k** EDS spectra of the Cu–Zn splash. **l** ESM-BSE view of a singular sandstone clast consisting of angular quartz embedded in a carbon-rich siliceous matrix with abundant metal inclusions. **m** Detailed ESM-BSE view of **l** showing the cracked quartz with the Fe–Cr–Ni thermal splash, same forming process as defined in **j**. **n** EDS spectra of the Fe–Cr–Ni thermal splash. **o** view under ESEM-BSE of a section from a yellowish gray, scoria-like, glassy bead-shaped clast showing the diaplectic (i.e., amorphous) quartz (*gray* grains) that is typical of impact by-products—known as lechatelierite (cf. Osinski *et al.* 2008; Bunch *et al.* 2012)—and the degassing vesicles with abundant metal inclusions (*red arrow*)

signs of construction materials and prepared mud floors, defined as Da'de Ia, only appear in the overlying occupation layers (phase Ib, cf Fig. 5a). The excavation over 2 m^2 showed the poor preservation of the carbonaceous surface, most often integrated as relictual micro-aggregates within the lowermost trampled strata (Fig. 5b). A singular assemblage of uncommon organo-mineral components similar to the one of area [B] was identified, although it showed locally concentrations of scoria-like beads, charcoal and vitreous carbon aggregates, and their fine imbrication with the heated host-soil, particularly in burnt patches (Fig. 5c). The range of carbonaceous polymorphs and their link to the host matrix suggest that these two type 1 signals originally formed by pulverization at the soil surface of a debris jet with volatile and solid components deriving from hydrocarbon fuel and associated aerosols. Similarly to the situation encountered in the surface related to the Bronze Age III/IV burial, these processes can be assigned to the ones of cosmic airbursts. The marked contrast in the characteristics of the virgin soils from areas [B] and [SB], the noticeable difference in the assemblage of singular components, and their association to distinctive anthropic facies converge to suggest that the two type 1 carbonaceous surfaces might not exactly relate to the same cosmic airburst. From a stratigraphic viewpoint, the one in area [SB] would be slightly older than the one from area [SB], although this is not supported by the identical ^{14}C age (Table 1). However, in each case, a strict contemporaneity is established between their formation by exceptional geogenic phenomena and human occupation. In spite of their distinctive contexts and age, the two PPNA type 1 situations and the Bronze III/IV one display a similar assemblage of uncommon organo-mineral components (cf. Figs. 3, 4) which are broadly identical to the ones that we identified in other natural signals of supposed impact origin (Courty *et al.* 2008, 2012a; Courty 2012a). For the PPNA and Bronze III/IV type 1 situations, the sorting by sized fractions and the multi-analytical characterization clearly show the exceptional nature of the debris assemblage in contrast to the host soils, particularly their angular edges, shiny faces, water-repellent behavior



during washing, and distinctive composition (Figs. 3e and 4a). The latter is remarkable by the intimate association of three constitutive phases: native metals, carbonaceous polytypes, and mineral grains. The last ones occur as isolated crystals of various sizes (millimeter to nano-domains) within the host fine mass or as composite rock clasts, particularly polycrystalline breccia, fine-sized sandstones, mudstones and clay

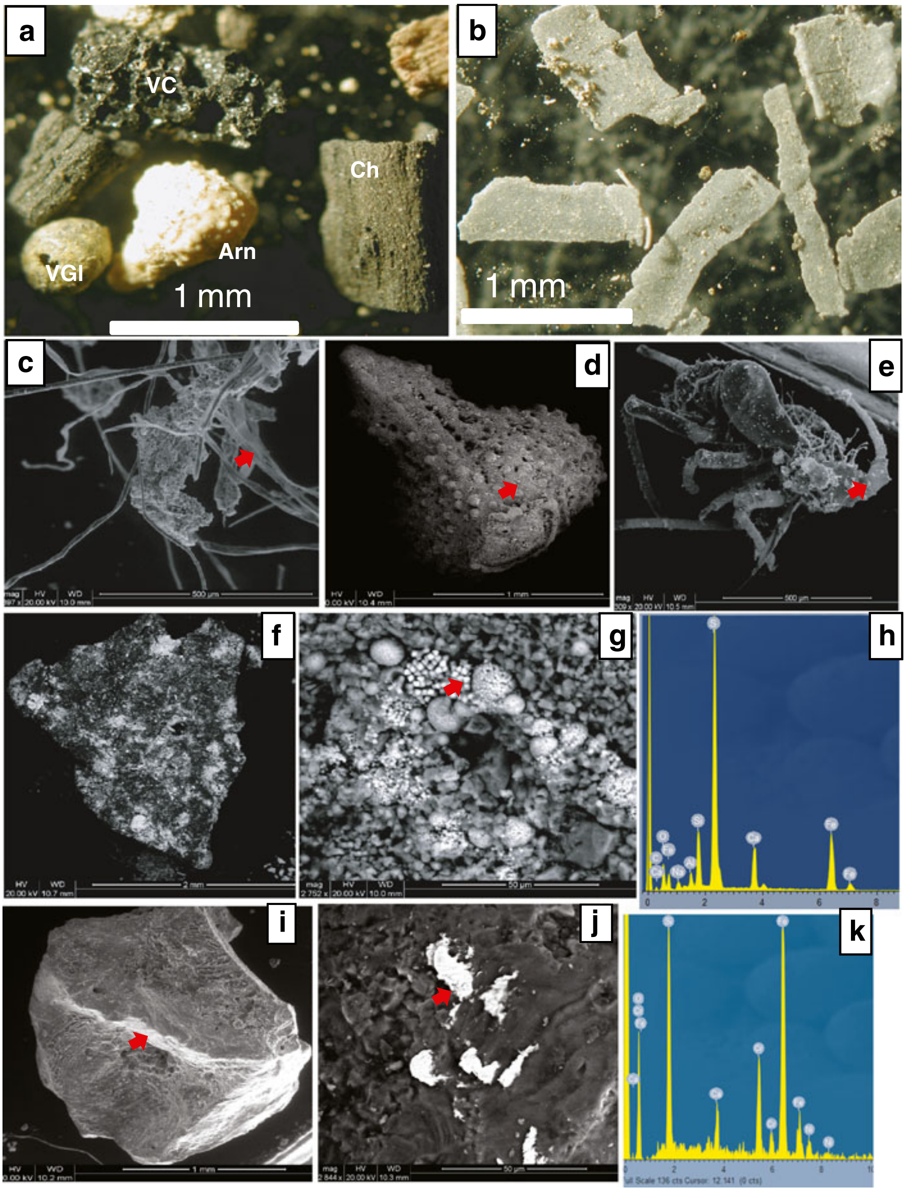


Fig. 4 Area [B], type 1 carbon-vaporized surface. **a** Binocular view showing some of the singular components and charcoal (*Ch*): vitreous carbon (*VC*), vesicular glass (*VGl*), and *Arnebia* seeds (*Arn*). **b** Binocular view of gray polymer filaments resulting from thermal cracking of hydrocarbon fuel. **c** ESM-BSE view of carbonaceous filaments embedding metal-rich mud clasts. **d** ESM-BSE view of an intact *Arnebia* grain. **e** ESM-BSE view of an intact insect. **f** ESM-BSE view of a fresh marine mud clast with metal and carbonaceous components. **g** ESM-BSE view showing well-crystallized iron sulfide concentrations in the fine silt of **f** indicating formation under anaerobic conditions; this morphology indicates that the clast is derived from fresh marine aerosols and not from lithified marine sediments which could have been reworked from the surroundings. **h** EDS spectra of the iron sulfide. **i** ESM-BSE view of a singular angular quartz with its Fe–Cr–Ni thermal splash formed by sputtering under reduced conditions (**j**), and spectra (**k**)

aggregates, and glassy phases (flakes, spherules, volcanic tephra). The most common compounds consist of quartz, plagioclase, pyroxene, zircon, rutile, ilmenite, magnetite, rare earth phosphates, barium and calcium sulfates, iron sulfide, iron phosphide, calcium sulfide, and soluble salts (NaCl, KCl). The carbonaceous polytypes comprise: (1) translucent to colored homogeneous masses and filaments, either adhering to the fine mass, as agglutinates of single fibers, or as a folded ribbon of densely packed fibers (Figs. 3f and 4b); (2) translucent, pale yellow, or black domains and films of agglutinated nano-sized spherules; (3) translucent to colored flakes with degassing vesicles or cracks; (4) brittle, finely cracked, shiny black, homogeneous vitreous grains; (5) vesicular black heterogeneous vitreous grains (Figs. 3g, h, i); (6) dark brown to translucent hollowed cenospheres of fly-ash type; and (7) silt-sized hexagonal graphite flakes. In a decreasing order of occurrence, the native metals are the following: Fe, Fe–Cr, Fe–Cr–Ni, Cu–Zn, Cu–Zn–Ni, Pb, Ni, Pb–Cr, Pb–As, Al, Cr, Ag, Au, Bi, Pt, W, and Co (cf. examples of EDS composition in Figs. 3k, n, and 4h, k). At meso- to micro-scales, they occur as discrete spherules, flakes, ribbon-shaped filaments, films, and splashed droplets at the surface of the exogenic mineral grains (Figs. 3j, l, m, o, and 4i, j) and as nanodomains within the carbonaceous filaments. In addition, the unusual assemblage integrates compound aggregates showing a mixing of host and singular components embedded in translucent to colored polymer filaments (Fig. 4c). Remarkably well-preserved insect and plant remains show at their surface the thin carbonaceous film with its typical unusual components (Figs. 4d, e).

Context, Facies Pattern, and Characteristics of the Type 2 Signals

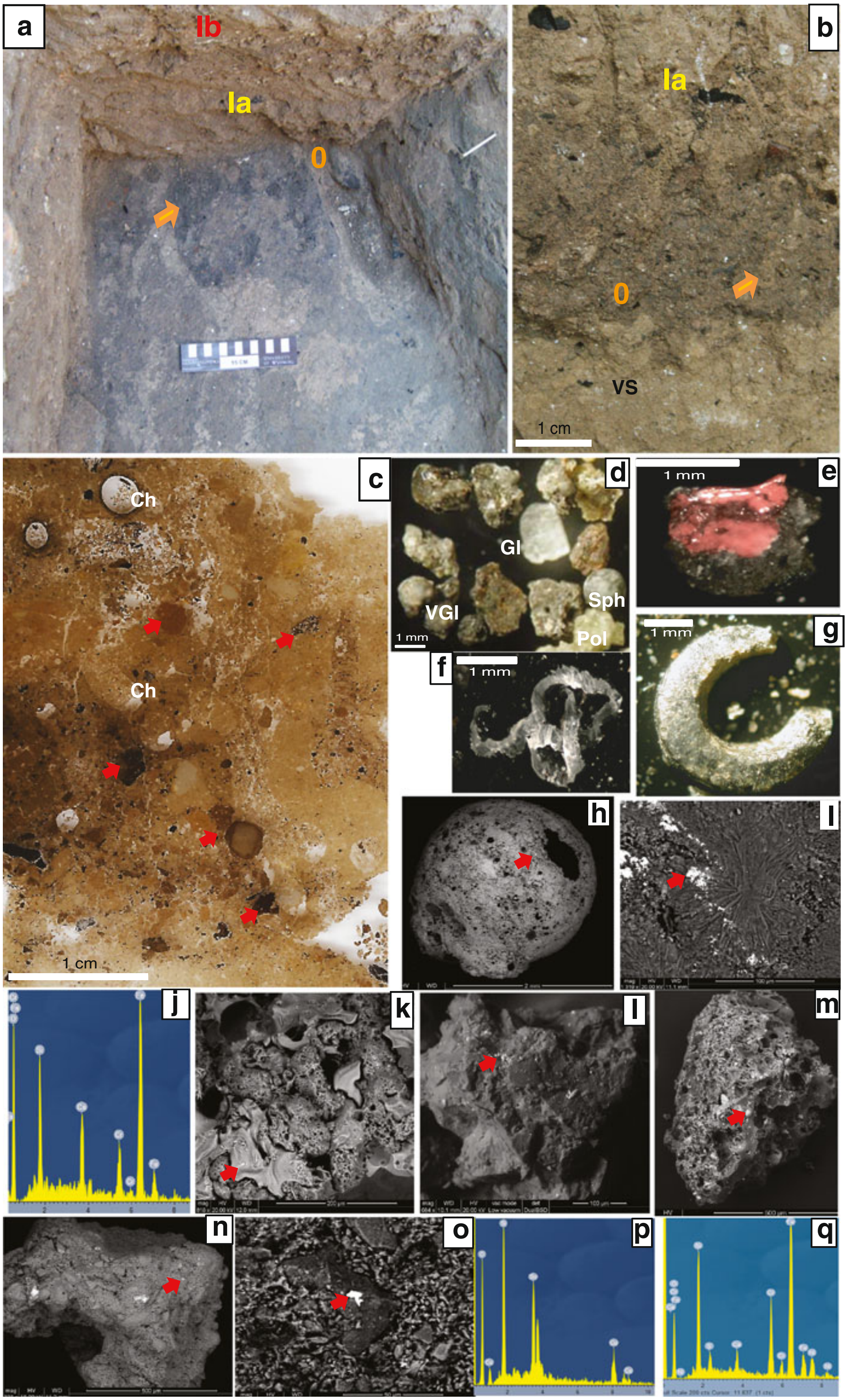
The integration of the field/multi-analytical protocol to the ongoing microfacies study has allowed to identify the occurrence of unique materials deriving from the type 1 unusual assemblage in a large diversity of habitation contexts at Da'de. These comprise millimeter- to centimeter-sized particles that are associated with all kinds of anthropic microfacies, or objects made from unusual stones or earthen materials with high content of unusual components (Table 2). Our search was greatly facilitated by their remarkable diagnostic characteristics, particularly the various carbonaceous components or the diverse metal traces, which are sharply contrasted to the local geological resources. They occur either in the host matrix as micron-sized carbon-rich clay domains with concentrations of rare earth phosphates, barium sulfate, or metal particles, or as an assemblage of organo-mineral components showing the same characteristics as the ones of the type 1 cosmic debris. Among others, the carbonaceous polymorphs with their various colored filaments and the black vitreous carbon were of great help for detecting the presence of the exceptional organo-mineral components: for example, agglutinates showing local grains embedded within paraffin translucent to colored resin that is rich in metallic micro-particles (Fig. 5e), fluffy bundles of colored to white aliphatic filaments (Fig. 5f), and angular particles of homogeneous black vitreous carbon with fine cracks, metal and sulfate inclusions, and graphitic micro-domains (Figs. 7h, i). In the following part, a series of cultural contexts showing the most intact type 2 signals are considered from the bottom to the top of the archaeological sequence.

Fig. 5 Area [SB], human-modified type 1 carbon-vaporized surface associated to phase 0 occupation layers. **a** Exposed black carbonaceous surface showing an irregular pattern at micro-scales that relates to differential preservation of small earthen structures (*orange arrow*); well-prepared occupation floors first appear with phase Ia and continue throughout phase Ib. **b** Vertical section in the field showing the black carbonaceous phase 0 layer with diffuse occupation layers associated to light structures; the *orange arrows* indicate the relict of a small earthen structure. **c** Scan from the thin section showing a heterogeneous assemblage of massive calcareous silty-clay, with abundant dark brown burnt domains, vitreous carbon (*red arrows*), and rare charcoal; note the channel voids with their compacted walls and loose filling that result from insect burrowing; these characteristics help to recognize the relict of small-sized earthen structures (oven?) used for transforming the singular hydrocarbon fuel. **d** Binocular view of millimeter-sized singular components: angular glass flakes (*Gl*), scoria-like vesicular glass beads (*VGl*), alumino–silicate and carbonaceous spherules (*Sph*), and Polymers (*Pol*). **e** Binocular view of a thin soft film of titanium-rich red polymer extracted by thermal processing of the hydrocarbon fuel. **f** Binocular view of a translucent carbonaceous filament produced by cracking of the hydrocarbon fuel. **g** Binocular view of a small bead made from a carbon-rich magnesian silicate, by-product from the thermally processed hydrocarbon fuel. **h** ESM-BSE view of a scoria-like vesicular glass bead showing the carbon-rich metal deposits at the surface, by-product from the thermally processed hydrocarbon fuel. **i** Detailed surface view showing the thermal Fe–Cr splash. **j** EDS spectra of **i**. **k** ESM-BSE view of the internal fabric of **h** showing the imbrication of a fluidal alumino–silicate glass (from the singular materials) and moderately fired local materials. **l** ESM-BSE view of a blocky grain of vitreous graphitic carbon showing metal inclusions on its surface, produced by cracking of the hydrocarbon fuel. **m** ESM-BSE view of a vesicular grain of vitreous graphitic carbon showing phosphate, sulfate, and metal inclusions within the degassing vesicles. **n** ESM-BSE view of an exogenous clast of silty marine mud formed of densely packed angular quartz with metal splash (*red arrow*) within carbon-rich cement. **o** Detailed surface view showing the metal thermal-splash deposit. **p** and **q** EDS spectra of **o** showing inclusions of Cu–Sn in the cement and Fe–Cr–Ni on the quartz grains

The Type 2 Singular Signals in the Da'de 0 and I Occupation Deposits

The lowermost evidence of type 2 signals of singular components was identified in the Da'de 0 initial phase of area [SB], just above the type 1 carbonaceous surface (Fig. 2), and of area [B] just above the virgin soil (Fig. 2a). In area [SB], the careful excavation over 4 m^2 of the first occupation layer has shown a mosaic of low- to high-temperature, ashy/charred/rubified microfacies, which are associated to residual small-sized hearths with diffuse contours. The great abundance of the unusual organo-mineral components as very fine-sized residues in the combustion facies suggests that they accumulated during firing treatment of the hydrocarbon fuel. The predominance of carbonaceous polymorphs, the abundance of red-colored silt-sized quartz, and the presence of fragmented processed objects—i.e., a glass-made bead (Fig. 5g)—suggests a production of coloring materials and of various objects from the processed fuel. The dark brown fine mass, the loose micro-aggregated fabric, the channel microstructure, and the lack of distinctive microstratification of the overall layer indicate that it remained widely opened to subaerial agents along to its formation. As a consequence, this early activity area seems to have taken place in an open space or, at best, in an area only seasonally covered by light structures, i.e., tents.

In area [B], a small sondage at the bottom of the painted building (phase I) has revealed the presence of an earthen structure that is sculpted within the virgin soil and is laterally associated to cultural deposits of Da'de phase 0, just at the contact with the virgin soils (Fig. 2a). The initial occupation layer is represented by a thin prepared mud floor with a dark brown graphitic carbonaceous surface that is also coating the associated earthen structure. The abundance of singular components within the graphitic carbonaceous surface and within the associated mud floor and their fine integration to the host clay matrix provide clear evidence to recognize a type 2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anthropic singular signal. In contrast to the one from the lowermost Da'de phase 0 occupation of area [SB], the incorporation of graphitic carbonaceous polymorphs, with their associated components (metals, rare-earth phosphates, barium sulfate, among others), to the clay matrix characterizes a type 2 anthropic signal of processed singular materials. The latter refers here to materials formed by meticulous mixing of local clay and by-products obtained by thermal processing of the hydrocarbon fuel, similar to the ones that have been identified in the combustion residues from the lowermost Da'de phase 0 occupation of area [SB]. In addition, the very fine integration of micron-sized singular components to the local clay suggests that the latter might have been extracted from local clay-rich raw materials with high amount of intact hydrocarbon fuel, which are suspected to have accumulated soon after the fall of fresh debris by a cosmic event (Courty 2012a). The polyphased mud plasters of the Da'de 0 earthen structure have shown marked variations in the amount of various singular components and local clay which appeared to be responsible for the distinctive color of each individual coating film. The pale-colored ones consist of a fine mixing of calcitic clay, translucent carbonaceous polymorphs, and finely crushed bones. In contrast, the dark-brown ones are rich in graphitic and charred particles within a decalcified clay matrix that contains abundant inclusions of rare earth phosphates, native metals, and barium sulfates. The construction of the Da'de phase 0 earthen structure and its successive reflections reveal a meticulous use of different types of local clay sources with a high amount of intact exceptional materials that were carefully selected in natural sources. In the juxtaposed cultural deposits, the hard setting, the heterogeneity, and the dense mixing of gravels, brickearth fragments, and sands of the coarse-textured units that are interstratified with the prepared mud floors (Fig. 3b) show that the earthen structure was repeatedly degraded by direct exposure to subaerial agents. Each event of flooding is clearly marked by the sharp contact between the graphitic carbonaceous surface that is coating each prepared mud floor and the overlying water-reworked deposits. The recognition in the field of residual patches of white, black, and red coloring materials on each carbonaceous surface has suggested that poly-chromic painted motifs were originally present on the prepared mud floor. The association of these relict painted spots to centimeter-sized hollows with a fine mud filling shows that degradation of the painted carbonaceous surface occurred under the effect of high-energy raindrops or, more likely, hailstones (Fig. 3c). This complex sequence of prepared and water-reworked occupation deposits enabled us to establish that the particular activities related to the lowermost earthen structure in area [B] were discontinuous through time and took place along Da'de phase 0 in an open space, during a period punctuated by heavy rainy events. The synchrony of the later with delivery of fresh hydrocarbon fuel and related debris along to recurrent cosmic events is suspected.

Abruptness of the transition from Da'de phase 0 to phase I is marked by a well-controlled filling of the lowermost earthen structure by gravels, pebbles, and alluvial coarse sands, and the meticulous sealing by a thick, well-prepared 10-cm-thick mud floor that forms the foundation of the partly buried magnificent building with its painted walls. The presence of early occupation layers (phase Ia), just above the virgin soil with the type 1 singular signal that was revealed behind one of the three pillars, suggests that part of the site was destroyed and/or remodeled for construction of the large building. These transformations might have occurred rapidly, possibly over a few years or at best decades, as indicated by the similar C^{14} dates obtained on charcoal from the phase 0 and I occupation layers (Table 1). In addition, the evidence for rather dry conditions that was noticed in the host matrix of the type 1 singular signal in area [B] and in the overlying occupation deposits (Ia) suggests that the heavy rainy events ended at the transition from Da'de phase 0 to phase I.

The initial mud floor of the painted building showed the greatest amount of the singular organo-mineral components, particularly the carbonaceous polytypes, with marked differences in the amount of the various species according to the types of anthropic microfacies. A preferential occurrence of the vitreous carbonaceous polymorphs with graphitized charcoal, mixed with graphitized crushed bones, has been encountered throughout the widely exposed dark-colored plastered floors at the bottom of the earthen building and within the black pigments of its painted walls (Figs. 6a, b, i, j). A high concentration of well-sorted singular minerals bearing iron

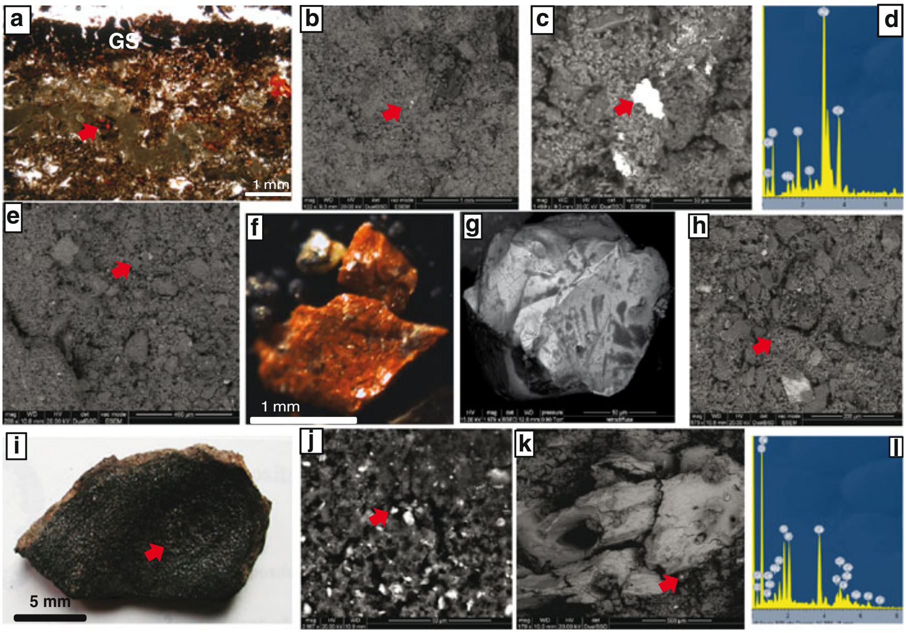


Fig. 6 Selection of diagnostic properties of the polychromic floors and painted walls. **a** View under the microscope of a thin section from the lowermost prepared carbonaceous floor (phase 0), showing the upper graphitic surface (*GS*), a silty-clay dark brown layer with crushed bones, and a calcareous grayish white layer with reddish spots of iron-rich coloring inclusions (*red arrow*). **b** ESEM-BSE view of the graphitic surface from **a** showing the fine mixing of vitreous carbon and silt in an open silicate cement with metal inclusions (*red arrow*). **c** ESEM-BSE detailed view of **b** showing the silver thermal splash, by-product from the thermally processed hydrocarbon fuel. **d** EDS spectra of **c**. **e** ESEM-BSE view of the grayish white layer formed of well-sorted siliceous silt with metal inclusions (*red arrow*), by-product from the thermally processed hydrocarbon fuel. **f** Binocular view of a reddish quartz grain with iron-rich and carbonaceous inclusions, by-product from the thermally processed hydrocarbon fuel. **g** ESEM-BSE view of an ilmenite with carbonaceous impregnation, by-product from the thermally processed hydrocarbon fuel. **h** ESEM-BSE view of the reddish surface formed of well-sorted siliceous silt with high amount of iron-rich components. **i** Fragment of dark brown bone showing a black graphitic crust, formed by contact by-product with the thermally processed hydrocarbon fuel; the inner brown part seems only weakly baked. **j** ESEM-BSE view of the graphitic crust on **i** showing abundant Fe–Ni inclusions in the carbonaceous fine mass. **k** ESEM-BSE view of the silty-clay dark brown layer from **a** with crushed bones in the siliceous matrix with singular inclusions (*red arrow*). **l** EDS spectra of a rare earth phosphate inclusion

oxides—iron-rich reddened quartz or carbon-rich ilmenite (Figs. 6f, g, h)—was found to be specific of the red-colored motifs. The common occurrence of silt-sized barium sulfate, rare earth-phosphate, drop-shaped zircon, and metal inclusions, together with domains of quick-lime and partly heat-decomposed marine microfossils, within a cement formed by the fine mixing of calcium sulfate and calcitic mud, also provided solid evidence for the singular origin of the white materials used for preparing the plastered floor and for the painted walls (Fig. 6d). In comparison to the type 1 natural singular assemblage, the close correlation between the range of specific components and their peculiar aspect attests to a meticulous separation of materials with different colors. In contrast to the double origin of singular materials that were used along to Da'de phase 0 (fresh hydrocarbon fuel and combustion byproducts), the range of carbonaceous polymorphs and associated components show that the mud floors, the plaster, and the paintings of Da'de I construction were predominantly made from thermally processed by-products from the singular fuel (Figs. 6b, c, d). These particular firing activities are well traced by the common occurrence in the filling deposits of heated pebbles with carbon-rich metal films and of graphitized coarse spongy bones that might be fragments of the tools used for processing the hydrocarbon fuel (Fig. 6i).

In addition, the presence of fragmented items (beads, gypsum figurines) made from singular rock types helps tracing by-products that were made from the residues left after the extraction of the coloring materials from the thermally processed hydrocarbon fuel. The great amount of heat-treated carbonaceous polymorphs appears to have played a major role in the remarkable preservation of the painted building. The meticulous maintenance of the successive mud floors, with a scarcity of micro-debris usually left by daily activities, shows that area [B] during the entire Da'de phase I has been continuously maintained, with nearly no debris accumulation, except the ones from occasional refreshing of the mud floors and of the painted walls.

Abruptness of the transition from Da'de phase I to phase II is marked by the rapid intentional filling and partial destruction of the exceptional building, with its distinctive cross-layered stratification, the lack of trampling evidence, and the occurrence of fragment blocks with paintings that collapsed from the elevated constructions.

The Type 2 Singular Signals in the Da'de II to III Occupation Deposits

The transition from Da'de phase I to II is marked by a widely extended well-prepared mud floor that contains only rare singular debris, mostly within brickearth fragments which are recycled from the former construction materials. The occupation deposits from Da'de phase II and III contrast with the previous ones by the erratic occurrence of the singular components, although they are encountered in all kinds of prepared materials (adobe constructions and mud floors), debris accumulation from activity areas and objects. This is particularly well illustrated by a great variability in the amount and the types of the carbonaceous polymorphs that we retrieved from the charred ash filling of contiguous combustion structures in the Da'de II occupation strata from area

[B] (Fig. 7). Thin ash strata and pit hearth ash fill appeared to dominantly contain abundant charcoal with calcitic ash, whereas a great amount of vitreous char with abundant unusual contrasted inclusions and polymer filaments were found in the juxtaposed flat hearths (Figs. 7g, h, i). We are thus facing two distinctive contexts of firing by-products: the charcoal-rich ones that derived from wood combustible and the ones with a high amount of singular components that derived from the use of a particular hydrocarbon fuel with high volatile content. Each category of firing by-products seems to relate to a specific functioning, as suggested by the different morphologies and firing intensities of the combustion structures. A similar use of the two fuel sources is also well established by the joint occurrence of charcoal, vitreous char, and carbonaceous filaments in the ashy matrix of pit hearths, with abundant burnt stones and gravels, which are commonly encountered in the different areas of Da'de II phases (Fig. 7b). The presence of the polymer filaments with native metals and thin coatings of similar metals on the burnt stones showed that these two components are pyrolysis residues from the singular fuel. In addition, the unbaked walls of the pit hearth and the occurrence of highly fired bone fragments with metal coatings suggest fast and rapid heating by flame pyrolysis of a volatile-rich fuel.

The presence of singular organo-components has been also identified in the traces of red ochre found in various contexts, either as fine powder encrusted within ground stones or in the reddish staining of a human skull in the house of death during the Da'de III phase (Figs. 8a, b, c), and as local concentrations on plastered surfaces. In contrast to the presence of the three distinctive pigments in the paintings of Da'de I, the various ochre traces encountered in Da'de II and III consist only of the reddish singular components with high content in iron oxides. In addition, similar types of transformed items made from unusual rock types as the ones encountered in Da'de I contexts were found, either as complete objects, partly processed ones, or fragments (Fig. 8).

The Type 2 Singular Signals in the Da'de IV Occupation Deposits

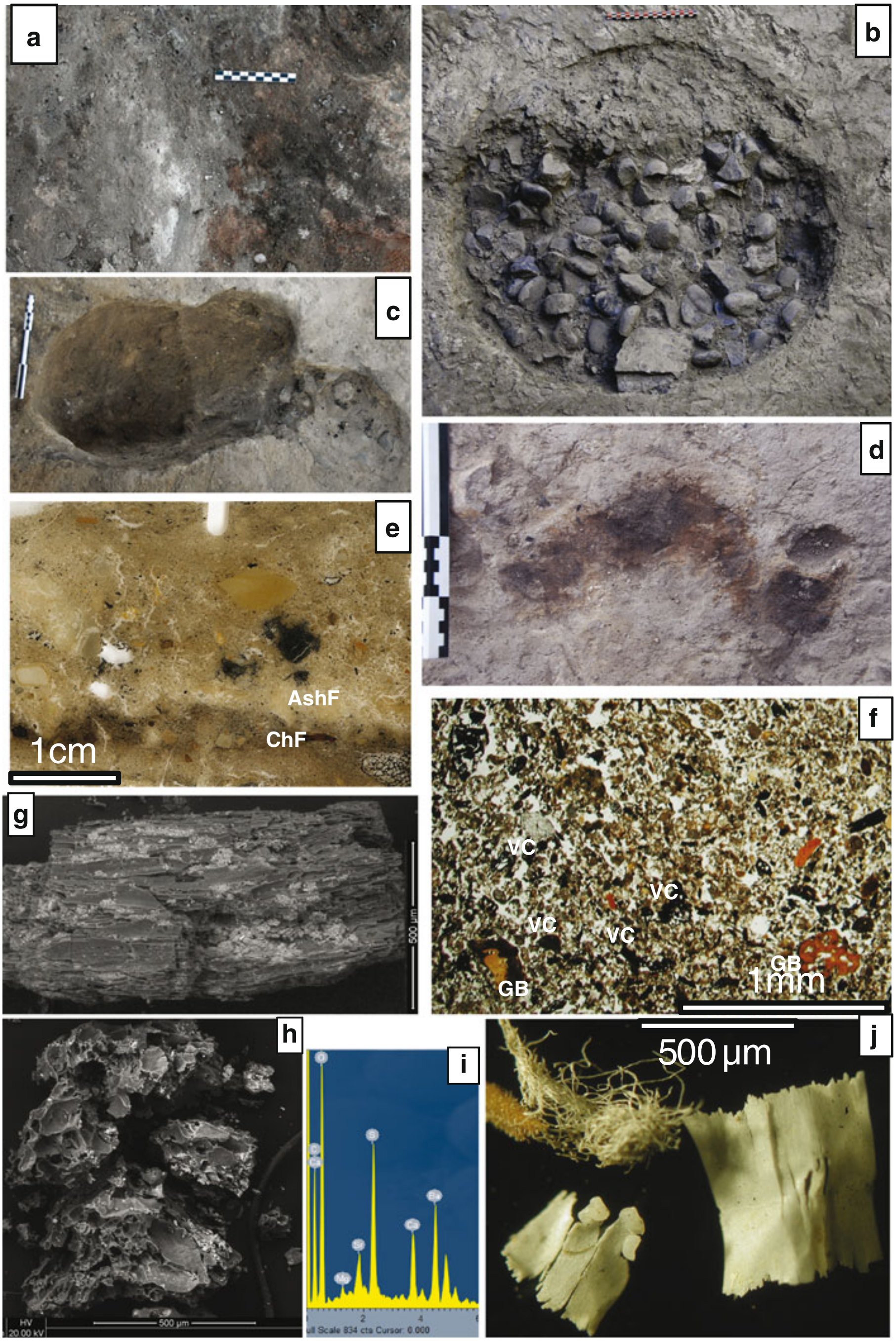
The lack of degradation by natural agents or intentional destruction shows that the site has remained nearly intact for ca. 1,000 years, apparently in the absence of human presence. The Early Neolithic occupation of the Da'de IV phase shows the occasional occurrence of the singular components within various materials linked to human activities, either the earthen architecture or the pre-Halaf ceramic assemblage, or raw and polished stones (Fig. 9). The very fine integration of the unusual carbonaceous polytypes to the calcitic fine mass of the earth materials, with their distinctive mineral tracers, indicates an exploitation of raw sources that were originally rich in fine-sized singular components. These characteristics would correspond to depositional contexts which preferentially trapped aerosols during periods marked by a series of exceptional cosmic events, thus explaining their high content in singular components. The common occurrence of distinctive grains formed of black vitreous carbon, carbonaceous agglutinates (Fig. 9b), or singular rock types in the finely aggregated anthropic microfacies, which are interstratified with the finely prepared mud floor, provides complementary evidence for the use of the singular raw materials during the

Fig. 7 Combustion structures, facies, and residues from Da'de II phase area F, sequence F, structure 736 (546 cm depth). **a** Compound ashy/charred combustion micro-facies forming a 5-cm-thick accumulation, with diffuse contours, and local evidence of reddish baked sediments; joint use of biomass fuel and hydrocarbon ones. **b** Well-defined pit hearth showing a compound ashy/charred combustion micro-facies with abundant burnt stones and gravels, and unbaked walls; dominant use of hydrocarbon fuel. **c** Dark brown charred combustion microfacies filling a small pit hearth with unbaked walls; dominant use of hydrocarbon fuel. **d** Reddish brown charred combustion microfacies filling a flat hearth with unbaked walls; dominant use of hydrocarbon fuel. **e** View in thin section of the charred/ashy micro-facies (*ChF*) with abundant vitreous carbon from **a** overlaid by a loose packing of pise fragments, wood charcoal, and disaggregated mud floor in a ashy matrix (*AshF*); abundant firing residues of hydrocarbon fuel in the two combustion facies. **f** View in thin section of the loose ashy combustion microfacies from the pit hearth in **b**: note the fine sorting of the ashy components, the abundance of graphitized bones (*GB*) and of vitreous carbon (*VC*) suggesting that disaggregation from hydrocarbon-rich, well-prepared materials, possibly earthen walls of the hearth. **g** ESEM-BSE view of a plant charcoal embedded in calcitic ash; **h** ESEM-BSE view of a vitreous carbon grain from the pit hearth in **b** with contrasted inclusions and carbonaceous filaments, produced by cracking of the hydrocarbon fuel. **i** EDS spectra of **h** showing inclusions of barium sulfate and Mg-rich carbonate. **j** Binocular view of fibrous and ribbon-shaped polymer filaments from the pit hearth in **b**, produced by cracking of the hydrocarbon fuel

pre-Halaf occupation. Similarly to the PPN sequence, the meticulous maintenance of the mud floors (cf. Fig. 9a) suggests that the occupation context under study was devoted to the use of transformed by-products, whereas activity areas where the valuable materials would have been processed have been so far not found at the site.

The Type 2 Singular Signals in Da'de V (EB III/IV Bronze Age Cemetery)

With the ultimate evidence of ancient human presence at the site, the well-preserved carbonaceous surface, with its distinctive type 1 natural singular signal, has already shown a strict contemporaneity between the occurrence of a cosmic event and the establishment of the EB III/IV Bronze Age cemetery. The correlation is fully confirmed by the occurrence of singular debris within the loose fine-earth filling of the associated funerary ceramics (Fig. 10). The possibility that the fine earth would have been introduced long after the Bronze Age period, while the burials remained exposed close to the surface, can easily be refuted by examining the greatest concentration of singular components in the ceramic 3 that was sealed at its top by another ceramic. Moreover, the fresh aspect of the components due to the lack of fine calcitic concretions and the slight dissolution of the fine mass contrast with the filling of the other whole ceramics that suffered calcitisation due to invasive colonization by fine roots and incorporation of carbonate-rich dust from the local surroundings. The abundance of the fibrous carbonaceous polymorphs and highly fired, finely fragmented bones in the ceramic fill suggests that the pot initially contained perishable components mixed with by-products derived from the singular raw materials and with calcined bones (Fig. 10). The fine encrustation of the latter by native metals that are not encountered in wood combustion residues indicates that the bones were fired by using volatile-rich fuel with high metal content (Figs. 10d, g, h, i). With regard to the funerary context, these characteristics encourage us to consider the associated ceramics as intentional offerings, possibly goods for the dead. The lack of additional traces of EB III/IV Bronze



Age artifacts in the other excavated areas suggests that this ultimate human presence would be restricted to the cemetery. In the absence of activity areas so far identified, the singular materials are suggested to have been introduced as finished by-products.

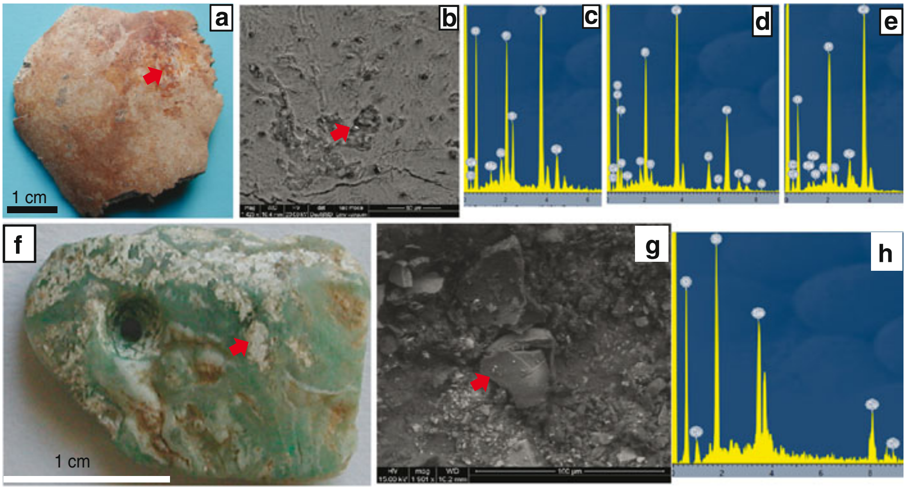


Fig. 8 **a** Ochred fragment of skull, area C, F9a X4, burial CLXXIX showing reddish staining (*red arrow*). **b** ESEM-BSE view showing anfractuosities within the reddish domains with siliceous carbonaceous agglutinates and singular inclusions (*red arrow*) that indicate that the coloring material was prepared from hydrocarbon fuel. **c**–**e** EDS spectra of inclusions from the reddish domains: barium sulfate, Fe–Cr–Ni, silver. **f** Partially polished green bead formed from a singular rock type consisting of carbon-rich hydroxylapatite and woodhouseite : $CaAl_3(PO_4)(SO_4)(OH)_6$ (based on XRD); woodhouseite is a very unusual type of phosphate encountered in hydrothermal ore deposits; the presence of metal and carbon within the phosphate minerals (*red arrow*) suggests here a formation from the airburst debris; the link with the singular assemblage is consolidated by micro-fragments of similar unusual phosphatic minerals in type 2 singular assemblage. **g** ESEM-BSE view showing anfractuosities with siliceous carbonaceous agglutinates with metal inclusions and fragments of marine diatoms (*red arrow*). **h** EDS spectra of Cu–Sn metal inclusions

Discussion

The study performed at Tell Da'de illustrates how we have been able to investigate possible causative linkages between the occurrence of exceptional geogenic events, i.e., cosmic airblasts, and distinctive tempo of cultural changes, assumed to represent social discontinuities (Fig. 11). We have identified three exceptional situations that are marked by sudden debris fall linked to a cosmic event, defined here by a type 1 natural singular signal, i.e., an unusual carbonaceous surface with a particular assemblage of organo-mineral components. The high-resolution microstratigraphic analysis has helped us to establish a strict contemporaneity between each of these exceptional geogenic events and a remarkable moment in the site history: (1) the initial settlement at Da'de phase 0; (2) the establishment of a magnificent communal building at Da'de phase I; and (3) after a long abandonment, the Da'de V occupation with the Bronze Age III/IV cemetery. For each situation, sealing of each nearly intact type 1 carbonaceous surface by cultural deposits has provided solid evidence to suggest that the population at Da'de, and most likely as well in the surroundings, experienced a suite of unusual events. This most probably started by shooting stars, explosive fire balls, and debris pulverization at the ground, with surface propagation of violent airblast, and then, possibly, heavy precipitation from the dense clouds that the sudden aerosol increase would have nucleated, depending upon the amount of carbonaceous dust in the atmosphere. The latter effects seem to have left significant traces in the host soil that is contemporaneous with Da'de phase 0, marked by a

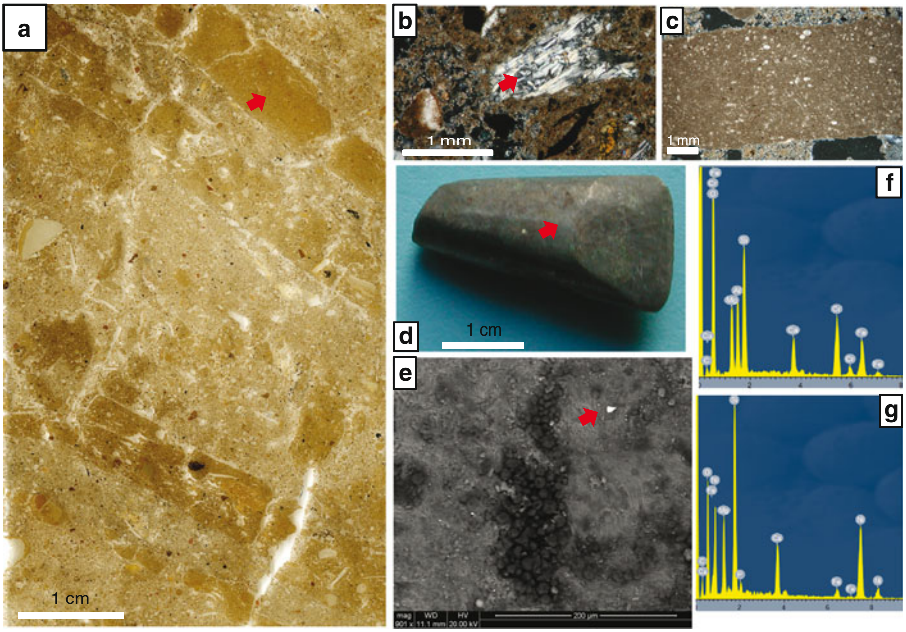


Fig. 9 Pre-Halaf occupation layers. **a** View in thin section of the microstratified sequence showing the alternation of finely prepared clay floor (*red arrow*) and finely aggregated sandy silt with common carbonaceous domains. **b** View under crossed polarizers of a singular carbonaceous agglutinate formed of densely packed anisotropic filaments (*red arrow*), suggesting mixing of by-products from the hydrocarbon fuel and of carefully prepared local clay. **c** View under crossed polarizers of a pre-Halaf ceramic consisting of very fine calcitic mud. **d** View of a small polished ax made of carbonaceous, phosphore-rich enstatite (magnesium pyroxene) with metal deposits (*red arrow*); this unusual composition for an igneous rock type suggests a formation from the airburst debris. **e** ESEM-BSE view showing anfractuosities with carbonaceous agglutinates with metal inclusions (*red arrow*) that might be traces of the material used for polishing. **f**, **g** EDS spectra of Cr and Ni metal inclusions

rainfall increase which suggests persistence of a dust veil for years to decades. In contrast, the aeolian dust accumulation after the cosmic events that are associated to Da'de phase I and V would more indicate sudden fall of debris formed by airblast, followed by an increased dryness. The soil records for the three situations encountered at Da'de clearly show that the cosmic events did not induce catastrophic damage of landscape properties or life, but more likely marked subtle changes in the seasonality and weather pattern for a short time period. It seems more likely that in each case, the population was rather prepared, or even more was expecting the debris fall to occur in order to benefit from the renewal of the singular fuel resources, possibly after a long period of shortage. Each of these particular situations appears to represent a discontinuous signal which at some points of time has simultaneously induced sudden changes, both in the environmental settings, particularly with regard to soil quality, weather pattern, or landscape features, and in cultural behavior due to the direct consequences on the quality and availability of valuable fuel resources. Each moment should better be viewed as a critical period with variable effects at local to regional scales, rather than a severe dual crisis that would have formed a sharp stratigraphic discontinuity throughout all kinds of sedimentary archives or cultural deposits. In agreement to our former study in other contexts, the data from Da'de

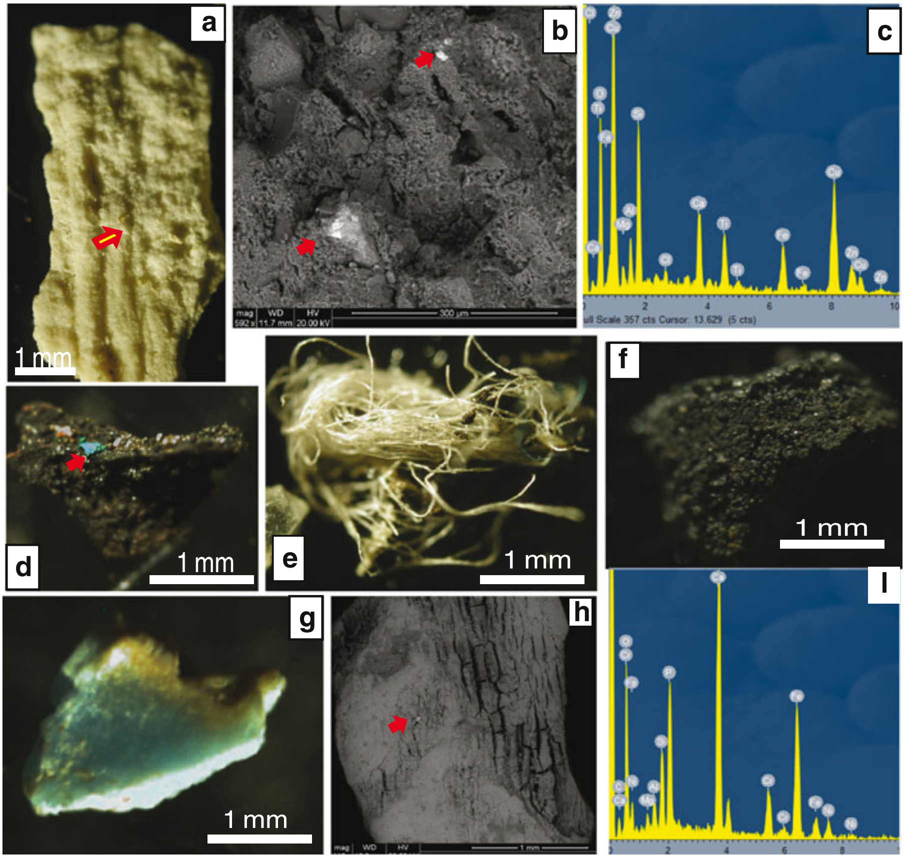


Fig. 10 Bronze Age III/IV burial, filling of an intact ceramic. **a** Binocular view of a clast of siliceous sandstone in which the presence of a translucent carbonaceous film with metal inclusions (*red arrow*) shows its unusual character, most likely by-products from the hydrocarbon fuel. **b** ESEM-BSE view of **a** showing metal particles in the siliceous cement and metal thermal-splash deposits on the angular quartz grains (*red arrow*). c EDS spectra of **b** Cu–Zn metal splash. **d** Binocular view of a bone fragment embedded in black graphitic carbon and with a blue carbonaceous polymer, by-products from the thermally processed hydrocarbon fuel. **e** Binocular view of carbonaceous filaments consisting of aliphatic polymer, by-products from the thermally processed hydrocarbon fuel. **f** Binocular view of a char fragment formed of finely cracked graphitic carbon, by-products from the thermally processed hydrocarbon fuel. **g** Binocular view of a highly heated green bone fragment. **h** ESEM-BSE view of **g** showing the cracked surface with siliceous and metal inclusions that are traces of the volatile-rich fuel used. **i** EDS spectra of a Fe–Cr–Ni metal inclusion

consolidate the physical reality of a cosmic-linked discontinuity with its singular carbonaceous surface.

The first cosmic event that we have so far identified strictly correlates with the initial PPNA settlement (Da'de 0) when the occupation was possibly seasonal, consisting of light habitations, i.e., tents and/or wood-made constructions (area [SB]) and around a small-sized exceptional structure (area [B]). At present, the fragmentary data obtained from the two test pits suggest that the settlement was created in a previously non-occupied area and that the lowermost exceptional structure was dug and sculpted into the virgin soil. In addition, the data collected so far from the two tested areas seem to have provided evidence that indicate a suite of

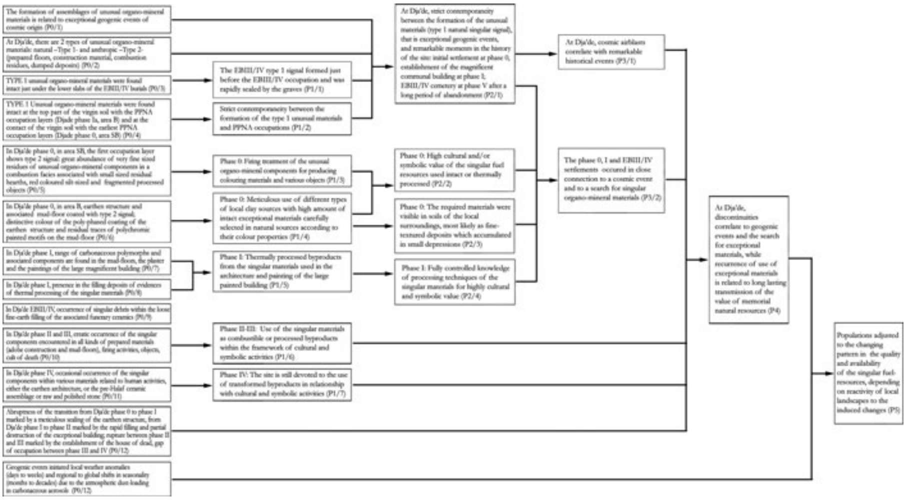


Fig. 11 Synoptic view of the scientific construct ("logicist diagram")

technical activities with the singular fuel resources which were just provided by the cosmic event. Obviously, the use of raw clay with high content of singular components in the fine mass implies that the required materials were visible for a short time in soils of the local surroundings, most likely as fine-textured deposits which accumulated in small depressions just after the cosmic airbursts, in relation to debris-linked heavy rainy events. These exceptional fine clay deposits are expected to have rapidly degraded under runoff and flooding, when reworked with local soils and sediments, and to not have accumulated any more, as soon as the amount of carbonaceous aerosols in the atmosphere dropped below a certain threshold. The availability of the singular clay sources in the surroundings only for a short time implies that they would have been meticulously collected in the days just following episodes of debris fall from a cosmic airburst. Thus, from a strictly soil/sedimentary viewpoint, the initial settlement at Da'de 0, with its earthen structure, can be inferred to have occurred in close connection to a cosmic event, i.e., more or less at the same time, and because of the high cultural and/or symbolic value of the hydrocarbon fuel resources. This hypothesis is well confirmed by the evidence for thermal processing of the singular fuel encountered both in areas [SB] and [B]. Along to phase 0, the close timing noticed between the periodical use of the singular fuel resources, both as raw clay materials and thermally processed by-products, and the recurrent degradation of earthen structure by heavy rainfall suggests that a series of cosmic events would have occurred during a rather short time period, possibly decades.

The Da'de I phase appears as the second major discontinuity in the cultural sequence which is marked by a new architectural design in area [B] and a general shift to earthen habitation that is traced in the three excavated areas. This seems to have occurred soon after a cosmic event, although at present, the restricted access in area [B] to the contemporary virgin soil and the early Da'de I occupation layers (Ia) only provides a fragmentary view on possible changes of soil landscapes and weather seasonality at the transition from Da'de 0 to I phases, during a few decades. The new planning was aimed to construct a long-lasting building as clearly expressed by the solid foundations, the choice of materials, the preparation techniques, and the internal design down to the smallest detail. The successive plastering phases reveal continuous maintenance in a habitat where all the daily actions of domestic life were excluded. In addition, the relict films of native metals of the lowermost mud floors suggest that mechanical actions were carefully avoided in order not to degrade the meticulously painted surface. This evidence for a particular use of the magnificent construction at Da'de would fully match the ongoing explanation from an archaeological perspective of collective buildings that would have been devoted to ritual practices (Stordeur 2006).

The third major discontinuity is represented by the careful sealing of the exceptional building which marks the transition between Da'de phases I and II. The stratigraphic record indicates a well-controlled planning with meticulous deposition of valuable items. The abundance of fragmented blocks with paintings at the bottom of the filling suggests that the sealing was preceded by an intentional destruction of the highest part. The three successive layers of paintings encountered for the entire painted walls clearly attest to occasional reshaping. The evidence for an increased fragility through time of the paintings might express a subtle change in seasonality with drier conditions and, as a consequence, a possible modification in the availability and quality of the singular fuel resources. At present, all the evidence converge to view the sealing as the intention to have preserved intact and forever the magnificent building with its associated items, although it was no longer visible, and highly degraded. The Da'de II phase appears as a new conceptual design of the special habitat, with marked change in the processing and use of the exceptional fuel resources. Further exploration is required to elucidate the exact function of the combustion structures showing variable proportion of combustion residues from the exceptional fuel resources and the range of activities that were performed in the associated habitations. In contrast to Da'de I phase, the constant reshaping of the earthen architecture suggests a possible change in the symbolic value of construction materials which seem to have been commonly recycled. Alternatively, the evidence for seasonal maintenance of the habitation and their more frequent reshaping could also express a decreased availability in the surrounding landscapes of the raw clay materials with high content in singular fine components, known for long as materials of good structural stability, thus well suited to long-lasting constructions.

The establishment of the house of dead expresses another social discontinuity with the Da'de III phase. The red ochre pigments on a skull seem to reflect some kind of continuity in the use of the singular fuel resources, which is also well expressed by their common traces in the abundant combustion structures all along the Da'de III phase, particularly the polymer filaments and the metal coatings on the burnt stones of the firing pits.

The Da'de IV phase that occurred 500 years after a gap of occupation has provided solid evidence to recognize the exploitation of fresh singular hydrocarbon fuel resources, their use in carefully prepared construction materials, and the production of transformed objects. In the absence of a virgin soil that would correlate with the pre-Halaf period, the hypothesis that a cosmic event would have occurred at that time yet remains to be attested by further investigations in sites and landscapes of the surrounding regions.

In contrast, the sudden return of human presence at Da’de with the establishment of the EB III/IV cemetery during phase V, after more than 4,000 years of abandonment, is clearly attested to have occurred just after the ca. 4-kyr BP cosmic event. Similarly to what has been noticed during previous phases of occupation, the use of newly delivered singular fuel resources in the funerary ceramics suggests that these exceptional materials were at that time of high symbolic value with regard to the cult of death.

Conclusion

The identification of a distinctive assemblage of singular components in natural and anthropic contexts at Da’de enabled us to focus on a particular range of unusual geogenic events, i.e., cosmic airbursts. Their originality lies in their ability to have recurrently delivered at the earth surface composite materials that formed from terrestrial aerosols due to exceptional thermal and high-pressure conditions of airbursts. The occurrence of highly resistant carbonaceous polymorphs, volatile-rich hydrocarbon fuel, carbon-associated native metals, and colored compounds has allowed to explain why these materials have been of particular interest for ancient humans.

At present, all the evidence so far collected at Da’de shows a close timing between the episodes of human presence and the periods when freshly delivered singular raw materials were available. This puzzling correlation requires to be further elucidated by similar investigation on sites of the surrounding regions for the occupation periods that are not represented at Da’de. The fine match between shifts in the use of the singular by-products for construction, habitation design, firing, and cult of death, and direct effects of environmental factors on the singular hydrocarbon fuel resources provides an original perspective on the link between cosmic events and cultural discontinuities. Rather than catastrophic manifestation which would have led to social crisis marked by desertion and collapse due to severe damage to life and properties, we propose to consider cosmic events as a cascade of dual signals with historical dimension. On one hand, the instantaneous airblast, which might have occurred several times over critical time periods, initiated the sudden (seconds to days) delivery of the singular hydrocarbon fuel resources and, as direct consequences, local weather anomalies (days to weeks) and regional to global shifts in seasonality (months to decades) due to the atmospheric dust loading in carbonaceous aerosols. On the other hand, populations adjusted exploitation strategies and transformation techniques to the changing pattern in the quality and availability of the singular hydrocarbon fuel resources, at first rapidly (days to weeks) following the sudden delivery, then more slowly (years to generations) depending on reactivity of local landscapes to the induced environmental changes. The long-term Da’de record, particularly the PPN sequence, illustrates how these constant, although discontinuous, adjustment might have been reflected in modifications of occupation pattern, habitation lifestyles, technical and/or symbolic activities at different timing (days to generations).

The evidence at Da’de for the recurrent use of the singular fuel over the long term appears to reveal a long-lasting transmission on the value of memorial natural resources which were only available from time to time in the surroundings. The occurrence of similar singular organo-mineral components in much older contexts from different cultural entities, i.e., in the Neanderthal occupation contexts at Abric Romani (Courty *et al.* 2012b), opens a challenging perspective on the possibility to trace ancestral traditions of universal dimension.

At last, the cultural situations encountered at Da'de suggest that continuity to have used the singular hydrocarbon fuel resources in traditions linked to the cult of death reveals an ancestral knowledge on the long-lasting resistance of these exceptional carbonaceous by-products.

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