Materials Properties, Use and Conservation: Construction Materials and Binders

# Analytical techniques for provenance determination of ancient volcanic pozzolans

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# Volcanic pozzolans and geological origin





### **Pozzolanic aggregates**



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### **Pozzolanic reaction**



### **Volcanic pozzolans**

**Volcanic pozzolans** consist of <u>volcanic rocks having a quasi-eminently pyroclastic</u> <u>origin (produced by explosive volcanic activities)</u> and having high concentrations of silica and active aluminia in amorphous or poorly crystalline rocks Therefore they are potentially reactive with lime in mortars: they ecompass many pyroclastic products with abundant volcanic glass, such as <u>pumice, volcanic ash</u>, <u>tephra, perlites and obsidians, poorly lithified tuffs and ignimbrites, and breccias</u>



# **Volcanic pozzolans**

### **Pumice**

Highly vesicular roughtextured volcanic glass



### Scoria

Vesicular, dark-colored volcanic rock usually feebly-to-moderately silica undersaturated.



### Tuffs

Volcanic ashes, scoria, pumices other volcanic products and lithified after sedimentation into a rock





Lithified tuff outcrop (Phlegraean Fields, Naples)

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Unlithified pumice outcrop (Phlegraean Fields, Naples)









### **Volcanic pozzolans**

### **Obsidian and perlites**

<u>Obsidian</u> is a natural glass formed by the rapid cooling of extremely silica-rich magmas (about 65 to 80%), having a very low in water content. <u>Perlite</u> is an amorphous volcanic glass, very rich in  $SiO_{2}$ , having a high-water content, typically formed by the hydration of obsidian.











# **Eploitation of volcanic pozzolans in antiquity**





## Uses of volcanic pozzolans in antiquity





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In the coating mortars of Punic Pantelleria cisterns (4th-3rd cent. B.C.) use of local volcanic pozzolans (Cuddia Rossa) for the production of waterproof and hydraulic mortars.

→ Volcanic pozzolans were not "discovered" by the Romans



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Shön *et alii 2011;* Shön 2012





### Vitruvian volcanic pozzolans

### Harenae fossiciae (Colli Albani)

(Volcanic scoriaceous cinerites)



*In caementiciis autem structuris primum est de harena quaerendum*, ut ea sit idonea ad materiem miscendam neque habeat terram commixtam. genera **autem** *harenae fossiciae sunt haec, nigra, cana, rubra, carbunculus.* 

In buildings of rubble work it is of the first importance that the sand be fit for mixing with the lime, and unalloyed with earth. The different sorts are these; black, white, deep red, and bright red.

Vitr. *De arch*. 2.5.1

recentes autem fossiciae cum in structuris tantas habeant virtutes, eae in tectorio ideo non sunt utiles quod pinguitudine eius calx palea commixta propter vehementiam non potest sine rimis inarescere.

Though pit sand (*harenae fossiciae*) is excellent for mortar, it is unfit for plastering; for being of such a rich quality, when added to the lime and straw, its great strength does not suffer it to dry without cracks.

Vitr. *De arch*. 2.5.3



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Black Pozzolan 407k.a. BP

Pozzolanelle

366k.a. BP

= Vitruvian *nigra* ?



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**Red Pozzolan** 475k.a. BP

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Vitruvian *rubra* + *nigra (?)*According to Jackson 2007



Fig. 3. Stratigraphy and sample locations of Castel di Leva (CDL) section. PR: Pozzolane Rosse; PN: Pozzolane Nere; PL: Pozzolanelle.







The Origins of Concrete Construction in Roman Architecture

> Technology and Society in Republican Italy

> > Marcello Mogetta

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1 mm

Da Jackson et al. 2007 Materials Properties, Use and Conservation: Construction Materials and Binders







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# Hadrian Mausoleum (2nd c. CE), nowadays Castel Sant'Angelo in Rome





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Est etiam genus pulveris quod efficit naturaliter res admirandas. <u>Nascitur in regionibus Baiais in</u> <u>agris municipiorum quae sunt circa Vesuvium</u> montem.

Vitr. 2.6.1

There is also a kind of powder that naturally makes things amazing. It grows in the regions of Baiani and in the fields of the municipalities around Mount Vesuvius.





### Phlegraean fields eruptions (Late Pleistocene 40 k.a. BP – present)

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### Somma-Vesuvius eruptions (Late Pleistocene 40 k.a. BP - present)



#### Somma-Vesuvius



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**Vitruvio**, *De arch.*, V, 12, 2-3. Eae autem structurae, quae in aqua sunt futurae, videntur sic esse faciendae, uti portetur pulvis a regionibus quae sunt a Cumis continuatae ad promontorium Minervae [...]. Deinde tunc in eo loco, qui definitus erit. arcae stipitibus robusteis et catenis inclusae demittendae destinandaeque aquam in firmiter: deinde inter eas ex transtillis inferior pars sub aqua exequenda et purganda, et mixta caementis ex mortario materia (quemadmodum supra scriptum est) ibi congerendum, donicum compleatur structurae spatium, quod fuerit inter arcas.

The structures to be made in the water, it seems to me, should be done in this manner. Let the dust be transported from those reasons which extend from Cumae to the promontory of Minerva [...]. In the place that will be established, let the closed arches be dropped into the water and connected validly with strong poles and chains: moreover inside those by means of rafts purge and level the lower part under the water, and then throw in cement matter mixed with lime (as was said above), until that space of structure that there is between the arches is filled.

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### Thermae of Baia, Gulf of Naples







Analisi: Rispoli et al. 2019 Materials Properties, Use and Conservation: Construction Materials and Binders









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...Commixtum cum calce et caemento non modo ceteris aedificiis praestat firmitatem, sed etiam moles, quae struuntur in mari, sub aqua solidescunt (Vitr. 2.6.1)

The mixture with lime and cement <u>not only guarantees stability</u> to the rest of the buildings, but also solidifies the masses that are built in the sea under water.

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Trajan Age (2<sup>nd</sup> c. CE)







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### Volcanic pozzolans diffusion (Imperial era)



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### Volcanic pozzolans diffusion (Imperial era)



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### **Alternative volcanic pozzolans**

Region	Site	Function	Cronology	Rock type	References
Etruria	Vulci e Vulsinii	structural	Republican Age	Pyroclastic rocks (Vulsinii, Latera)	Marra, D'Ambrosio 2013;D'Ambrosio et al. 2015;
Pantelleria	-	Cistern revetments	4th-3rd c. BC	Volc. Scorias (Cuddia Rossa/Bruciata)	Schön et al. 2012; Schön 2014, 203-212
Pantelleria	Scauri	Plasters	4th c. AD	Volcanic scoria (Cuddia rossa)	Montana et al. 2013
Sardinia	Nora	Structural	1st c. BC	Perlites and obsidians (Mt. Arci)	Columbu et al. 2019
Germany	Koln	Structural	2nd c. AD	Tuff (Rhineland Trass)	Lamprecht 1984, 46-49; Wang, Althaus 1994
Asia Minor	Sagalassos	Structural	Imperial age	Differentiated volcanics (Lake Golcuk)	Callebaut et al. 2000; Degryse et al. 2002
Asia Minor	Nysa and Aigai	Structural	Imperial Age	Differentiated volcanics (Dikili-Çandarlı)	Uğurlu Sağın, Engin Duran, Böke 2021

























# **Euganean Hills**







Trachyte / Latite
breccias



















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# Analytical techniques for provenance determination





### **Optical microscopy**



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### **Optical microscopy**



- **Geochemistry** is the science that uses the <u>tools and principles of chemistry to</u> <u>explain the mechanisms behind major geological systems</u> such as the Earth's crust and its oceans.
- In geology, igneous differentiation, or magmatic differentiation, is an umbrella term for the various processes by which <u>magmas undergo bulk</u> <u>chemical change during the partial melting process</u>, <u>cooling</u>, <u>emplacement</u>, or <u>eruption</u>. The sequence of magmas produced by igneous differentiation is known as a magma series.
- In magmatic differentiation differences in chemical composition are affected by several factors (i.e. Distinct melting events from distinct sources; Crystal fractionation; Mixing of 2 or more magmas; Assimilation/contamination of magmas by crustal rocks
- <u>This results in variation in the concentration of chemical mayor elements (in</u> particular Si), <u>and in the trace element concentrations</u>. Trace elements are elements that occur in low concentrations in rocks, usually less than 0.1 % (usually reported in units of parts per million, ppm).

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When considering the rocks in the mantle, trace elements can be divided into *incompatible elements*, those that do not easily fit into the crystal structure of minerals in the mantle, and *compatible elements*, those that do fit easily into the crystal structure of minerals in the mantle.

- <u>Incompatible elements</u> these are elements like K, Rb, Cs, Ta, Nb, U, Th, Y, Hf, Zr, and the *Rare Earth Elements* (REE)- La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, & Lu. Most have a large ionic radius. Mantle minerals like olivine, pyroxene, spinel, & garnet do not have crystallographic sites for large ions.
- <u>Compatible elements</u> these are elements like Ni, Cr, Co, V, and Sc, which have smaller ionic radii and fit more easily into crystallographic sites that normally accommodate Mg, and Fe.

When a mantle rock begins to melt, the incompatible elements will be ejected preferentially from the solid and enter the liquid. This is because if these elements are present in minerals in the rock, they will not be in energetically favorable sites in the crystals. As melting proceeds the concentration of these incompatible elements will decrease because (1) there will be less of them to enter the melt, and (2) their concentrations will become more and more diluted as other elements enter the melt.

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Periodic Table of Elements and Oxides for Petrologists																	
Atomic # Symbol Element						for Av ion (w ) (pj Se	Average composition of oxide (weight percent) or element (ppm) in various rock types . See caption for sources. Average composition of oxide (weight percent) or element (ppm) in various reservoirs . See caption for sources See caption for sources								nce ide nula		
1 H 1.008		Atom	ic Wt.	with or	racter of bond h oxygen Formula												<sup>2</sup> He <sup>4.003</sup>
3	4	4 Be 9.012Links to other Periodic Tables5 B6 C7 N8 O9 F9.012• Royal Society of Chemistry Periodic Table • IUPAC Periodic Table of the Elements and Isotopes10.8112.01114.00715.99918.998														9	10
Li	Be															F	Ne
6.941	9.012															18.998	20.18
11 Na 22.990	<sup>12</sup> Mg <sub>24.305</sub>	• 1	The Earth Michael I	Scientist Dayah Dy	entist's Periodic Table of the Elements and Their Ions h Dynamic Periodic Table											17 Cl 35.45	18 Ar 39.948
19	20	21	22	23	<sup>24</sup>	<sup>25</sup>	26	27	28	29	<sup>30</sup>	<sup>31</sup>	<sup>32</sup>	<sup>33</sup>	<sup>34</sup>	35	36
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
39.098	40.078	44.956	47.987	50.942	<sup>51.996</sup>	54.938	55.845	58.933	58.693	63.546	65.38	69.723	72.630	74.922	<sub>78.971</sub>	79.904	83.798
37	<sup>38</sup>	<sup>39</sup>	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54
<b>Rb</b>	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
85.468	87.62	88.906	91.224	92.906	95.95	98	101.070	102.906	106.42	107.868	112.414	114.818	118.710	121.760	127.60	126.904	131.293
55	56	57-71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86
Cs	Ba		Hf	Ta	W	<b>Re</b>	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
132.905	137.327		178.49	180.948	183.84	186.207	190.23	192.217	195.084	196.967	200.592	204.38	207.2	208.980	209	210	222
87 Fr 223	88 Ra 226	89-103	104 Rf 267	105 Db 268	106 Sg 269	107 Bh 270	108 Hs 269	109 Mt 278	110 Ds 281	${\mathop{\rm Rg}\limits_{_{280}}}$	<sup>112</sup> Cn <sub>285</sub>	113 Nh 286	114 Fl 289	<sup>115</sup> Mc <sup>289</sup>	116 Lv 293	117 Ts 294	118 Og 294
	RB Value	es	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71
	.1-100 wt <sup>6</sup>	%	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
	0-1000 pp	m	138.905	140. 116	140.908	144.242	145	150.36	151.964	157.25	158.925	162.500	164.930	167.259	168.934	173.045	174.967
0	.1-10 ppm	L	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103
<	0.1 ppm		Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr
1	.a.		227	232.038	231.036	238.029	237	244	243	247	247	251	252	257	258	259	262

Abundance of the elements in various average volcanic rocks is shown as weight percent (wt.%=g per 100 g of rock) of the oxide for SiO2, TiO2, Al2O3, FeO, MnO, MgO, CaO, Na2O, K2O, P2O5 or as parts per million (ppm=micrograms per g) for all other elements.

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**TAS - Total Alkalis vs. Silica Variation Diagram**. The TAS Diagram for volcanic rocks is used to classify igneous rocks using whole rock chemical data as adopted by the International Union of Geological Sciences (IUGS).

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The variation diagram scatter plot of weight percent Na2O + K2O weight percent VS. SiO2 is divided into regions based on the whole rock chemistry rocks with of petrographicallydefine names (see the d QAPF diagram). The TAS diagram enables classification of glassy volcanic (i.e. pozzolans) and other volcanic rocks without, support or in to, petrographic data.

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The analysis of discriminant trace elements (or sometimes of ratio among discriminant trace elements) can help in the discrimination of volcanic domains. i.e. in this case, to distinguish Latian from Campania magmatic districs, based on the concentrations of Nb(Niobium)/Y(Yttrium) vs Zr(Zirconium)/Y(Yttrium)



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### **Analytical techniques**

Discriminant diagrams can report multiple traces (or ratios among traces) useful for discrimination, in this case a further discriminant diagram for distinguiscing Campanian Volcanoes from Latial ones is the Nb (*Niobium*)/Zr(*Zirconium*) vs Th (Thorium)/Ta(Tantalum) scatterplot



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Based on around 950 geochemical fingerprint of pyroclastic rocks from magmatic units of the Gulf of Naples  $\rightarrow$  in-detail intra-regional provenance Yb(Ytterbium) vs Th (Thorium) scatterplot



Based on around 950 geochemical fingerprint of pyroclastic rocks from magmatic units of the Gulf of Naples  $\rightarrow$  in-detail intra-regional provenance definition (Neodimium Vs Rubidium) 1



Eruptiv e event	unit	sample	type	Zr	Nb	Th	Rb	Sr	Y	Ва	La	Ce	Nd	U	Zn	Pr	Sm	Eu	Gd	Dy	Er	Yb	Lu	Та	V	referen ce
Post- NYT	fondo riccio	CF- FR-C2	bomb	229.2	33.2	22.8	309.6	919.8	33.4	1712.1	63.7	123.8	56.8	6.1	73	13.74	10.5	2.5	7.78	5.97	2.95	2.6	0.42	1.8	209	Cannat telli et al 2007
Post- NYT	fondo riccio	CF- FR-C1	scoria	233.2	35	22.1	274.8	903	32.5	1719.2	64.1	123.6	53.9	6.8	62	13.4	10.1	2.37	7.66	5.89	2.89	2.5	0.41	1.8	193	Cannat telli et al 2007
Post- NYT	Minop oli	CF- MI1-C1	scoria	182	26.7	18.1	238.5	946	28.7	1715.2	56	109.7	49.2	5.2	44	12.37	9.7	2.24	6.99	5.47	2.68	2.4	0.36	1.4	208	Cannat telli et al 2007
Post- NYT	Minop oli	CF- MI1-C2	scoria	267.9	42.1	29.7	305.1	724.7	32.1	1048.9	74.3	139.6	57	9	9	14.82	9.9	2.27	7.14	5.65	2.95	2.7	0.41	2.3	110	Cannat telli et al 2007
Post- NYT	Fondi di Baia		tephra	794.0	108.0	75.0	408.0	25.0	61.0	3.0	148.0	247.0	95.0	21.0		27.0	16.4	1.7	13.4	10.6	6.2	6.3	0.9	5.7		Smith et al 2011
Post- NYT	P.S. Nicola		tephra	504.0	66.0	46.0	328.0	331.0	41.0	221.0	103.0	171.0	70.0	12.1		20.0	12.7	2.0	10.4	7.5	4.3	4.6	0.7	3.6		Smith et al 2011
Post- NYT	Pisani 1		tephra	280.0	37.0	25.0	257.0	935.0	34.0	1625.0	68.0	122.0	55.0	7.8		15.0	10.9	2.2	9.0	6.8	3.4	3.2	0.5	2.1		Smith et al 2011
Post- NYT	Pisani 2		tephra	256.0	36.0	21.0	249.0	1007.0	30.0	1517.0	60.0	106.0	47.0	6.3		13.0	9.4	1.9	7.6	5.6	2.8	2.8	0.4	1.9		Smith et al 2011
Post- NYT	Pomici Princip ali		tephra	312.0	45.0	30.0	308.0	919.0	31.0	1779.0	72.0	124.0	52.0	9.1		15.0	9.9	2.1	8.1	6.0	2.8	2.9	0.4	2.3		Smith et al 2011
Post- NYT	S. Martin o	CFA81 a		465.0	54.0		309.0	437.0	22.0	326.0	76.0	165.0														Civetta et al 1991





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# **Geochemical techniques**

Main techniques used to determine the geochemical profile of volcanic rocks

- SEM-EDS (Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy): semi-quantitative major chemical element profiling (expeditious but not resolving for provenance determination)
- XRF (X-ray fluorescence): exact quantification in terms of percentages of major and trace chemical elements, useful for provenance determination but need enough sample quantity (at least 2 grams)
- LA-ICP-MS (laser ablation-inductively coupled plasma-mass spectrometry): extremely useful for very high precision quantification of major and trace chemical elements in volcanic rocks. It can be done punctually to map even extremely minute areals of material, even on individual minerals.



# **Case Studies**

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# The Punic-Roman town of Nora (Sardinia)



- Established by Phoenicians in the 8<sup>th</sup> c. BC become a Punic settlement during the 5<sup>th</sup> c. BC;
  - After the Roman conquest of Sardinia, in the 3<sup>rd</sup> c. AD Nora (and Sardinia) was involved in a flourishing period of renovation, with the construction of new temples, baths and an aqueduct;
  - The University of Padova (DBC) has ongoing excavation activities since 1990 (urban infrastructures, necropolis, private and public buildings)





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# **Analytical method**: XRF (coarse clasts, more than 2 grams of material from each sample available for analysis)

	T.ROM_1	T.ROM_2	T.ROM_3	T.ROM_4	T.ROM_5	T.ROM_6	T.ROM_7	T.ROM_11	T.ROM_20	
%Ox	tuff	pumice	tuff							
SiO <sub>2</sub>	59.31	60.29	60.89	60.82	60.53	61.71	60.5	60.07	62.07	
TiO,	0.48	0.47	0.49	0.53	0.46	0.44	0.47	0.45	0.44	
Al,0,	18.29	18.55	18.45	18.30	18.38	18.26	18.89	18.41	17.75	
Fe <sub>1</sub> O <sub>1</sub>	3.88	3.67	3.51	3.59	3.44	3.18	3.71	3.47	3.17	
MnO	0.15	0.15	0.17	0.17	0.15	0.18	0.13	0.14	0.16	
MgO	1.27	0.39	0.14	0.24	0.33	0.09	0.29	0.27	0.92	
CaO	1.94	2.61	2.17	2.88	2.41	1.99	2.55	2.67	2.97	
Na <sub>2</sub> O	4.11	4.91	5.42	5.68	5.26	5.94	4.63	4.94	2.95	
K,0	9.30	8.00	7.63	7.41	8.01	7.15	8.52	8.23	9.25	
P.O.	0.27	0.08	0.05	0.05	0.07	0.03	0.09	0.07	0.08	
Tot	99.00	99.12	98.92	99.67	99.04	98.97	99.78	98.72	99.76	
L.O.I.	14.97	4.96	3.55	3.76	3.48	2.34	3.38	3.27	11.71	
ppm										
S	169	137	38	126	76	63	56	95	145	
Sc	3	<3	3	13	11	<3	3	<3	3	
v	62	50	26	32	38	18	49	45	38	
Cr	6	<6	4	<6	7	<6	5	<6	12	
Со	3	9	<3	6	6	<3	<3	6	<3	
Ni	5	<3	<3	<3	<3	<3	<3	<3	<3	
Cu	33	9	17	217	143	24	24	31	10	
Zn	103	102	100	106	126	109	89	411	81	
Ga	12	18	13	14	14	17	10	12	13	
Rb	362	390	436	440	411	478	381	392	366	
Sr	223	203	127	81	110	45	278	152	196	
Y	30	45	63	65	49	73	37	43	50	
Zr	386	550	763	779	610	956	517	528	660	
Nb	52	67	94	101	77	113	65	64	86	
Ва	766	104	122	35	46	21	182	73	231	
La	81	102	122	132	108	161	97	93	118	
Ce	161	204	252	267	220	323	196	193	238	
Na	60	/3	103	105	82	110	/2	/1	8/	
PD	/5	/1	62	62	55	50	62	53	49	
1 <b>n</b>	43	49	/2	/4	50	89	50	52	6/	
U	× ×	16	22	20	18	26	14	15	16	

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Analytical method: XRF (coarse clasts, more than 2 grams of material from each sample available for analysis)

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dei Leganti Idraulici

DI GEOSCIENZE

Materials Properties, Use and Conservation:

**Construction Materials and Binders** 



Full compatibility with pyroclastic products of the Gulf of Naples, in particular with the late eruptions of Phlegraean Fields (post-NYT, along the coastline of the Gulf of Naples)

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terials Properties, Use and Conservation:

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### Aquileia (Friuli Venezia Giulia)





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- Established as colonia latina in 181 bC
- During the Imperial age was enriched by construction of theatre, amphitheater, circus
- In 4<sup>th</sup>-c. AD was celebrated as one of the most prestigious centers in Roman world
- In 5<sup>th</sup>-c AD falled against Attila invasion
- Reconquered by Byzantium in 6<sup>th</sup>-c. AD
- Progressively abandoned since the 7<sup>th</sup>-c. AD

### Materials Properties, Use and Conservation: Construction Materials and Binders





**dBC** 

# Aquileia – Theatre (beginning 1st c AD)



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### Aquileia – Theatre (beginning 1st c AD)



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## Aquileia – Theatre (beginning 1st c AD)



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Analytical method (trace elements): LA-ICP-MS Materials Properties, Use and Conservation: Construction Materials and Binders

# Lagoon of Venice – TSF (1st c AD)

Roman water-tank (weel cistern type) buried in underwater environment in the lagoon of Venice, dated to the Roman Imperial Age (1st – 2nd c. CE)





### Lagoon of Venice – TSF (1st c AD)



### **Conclusive remarks**



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i Materiali Cementizi dei Leganti Idraulici

DI GEOSCIENZE

**DI PADOVA** 

**Construction Materials and Binders** 

### **Conclusive remarks**

- These **provenance studies** are extremely helpful in tracking the trading of materials, in this case particular building materials, in antiquity.
- Obviously, this is only the first step: the archaeometrical analysis makes it possible to verify the provenance in many cases (or to narrow the field of possible provenances) of pozzolanic material, but from this point it is important to contextualize the data in relation to a precise historical, historical-economic, historical-cultural framework: this is the archeological component, which must explain and motivate certain commercial choices, with dynamics of shifting workers, technological choices required by clients
- $\rightarrow$  Transition to the realm of pure archeology.





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### **Materials Properties, Use and Conservation: Construction Materials and Binders**

# THANK YOU FOR YOUR **ATTENTION!**











