Tracking environmental and historical footprints on *Clarence*: Comparative XRD analysis of clay-rich sediment samples from a 19th century wreck site in Port Phillip Bay, Victoria, Australia

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Abstract

Little is known about shipbuilding in Australia in the early to mid 19th century. Under the Australian Research Council (ARC) funded Australian Historic Shipwreck Preservation Project (AHSPP) (www.ahspp.org.au), underwater excavations were carried out in April-May 2012 on the historic trading schooner Clarence, wrecked in shallow waters at Port Phillip Bay, Victoria in September 1850. During excavations, cores collected by lead investigator Peter Veth and principle investigator Vicki Richards were found to contain clay-rich sediment, thought to be ballast. This discovery stimulated investigations of the micro-sedimentary environments and taphonomy associated with the vessel, especially fine-grain sediment supply to the wreck and current and tidal influences on the stability of sediments lodged in and around the site. In order to address these questions, sediment samples were collected from the wreck, seabed and adjacent shorelines. Clay fractions were analysed at the Australian National University (ANU) using X-Ray Diffraction (XRD). Key objectives were to compare mineral signatures in the 'ballast' from clay-rich sources on the seabed and coastal foreshores possibly incorporated as the vessel foundered. Results successfully differentiate a) individual samples by seabed location and b) "ballast" samples in the wreck structure from sampled points around the wreck. The findings suggest that fine sediment within the Clarence shipwreck is likely to be clay ballast, emplaced at some point during the schooner's working life. The results also inform questions regarding the longer-term conservation of Clarence and similar wrecks located in Australian and Southeast Asian shallow-water settings.

Key words: Clarence, Shipwreck, X-ray Diffraction, Sediment, Ballast, Schooner, Conservation.

Introduction

Clarence, one of Australia's best preserved carvel-built, two-masted wooden trading schooners (Gesner, 1985), was constructed on the Williams River in New South Wales by Sydney merchants Gordon Sandernais and Thomas Ayers in 1841 (Coroneos, 1991). At the time, the Williams River shipbuilding industry was in its early stages of growth, producing unique examples of early shipbuilding techniques in Australia (Taylor, 1977). Small wooden coastal trading vessels were integral to the survival and development of Australia in the early to mid-19th century before the adoption of steam transport (Taylor, 1977). For the next nine years, *Clarence* worked as a trade vessel, transporting goods and passengers to and from Victoria, New South Wales and Tasmania. On the 2nd of September 1850, the vessel foundered on a sand bank in Port Phillip Bay while transporting sheep and other goods from Melbourne to Hobart. The vessel remained partially submerged for a number of years until eventually retreating to the sea floor (Gesner, 1985). In the mid-1980s the wreck, now protected under the *Victorian Heritage Act 1995,* was rediscovered and its historical and archaeological significance acknowledged as a result of investigations conducted by the Maritime Archaeology Association of Victoria (MAAV) (Gesner, 1985; Harvey, 1986, 1989). These findings were subsequently reported to the Maritime Archaeology Unit (MAU) of the Victoria Archaeology Survey (VAS). In turn, an environmental monitoring program and report was compiled by Coroneos (1991). Today the wreck is located in shallow waters (4-5 m deep) where natural processes and small vessel anchorage are negatively impacting the vessel's structural preservation. In April-May 2012, in accordance with the United Nations Educational, Scientific and Cultural Organisation (UNESCO) *Convention on the Protection of the Underwater Cultural Heritage*, the first phase of excavation, reburial and *in-situ* preservation of *Clarence* was undertaken by the ARC funded AHSPP. During excavations, it was apparent that there was a shallow layer of loose marine sediment overlying a dense clay-rich unit (appearing to have retained marks possibly produced by bucketing of clay into the hold of the schooner) (Veth et al., 2013) lodged within the vessel structure on top of the schooner's ceiling timbers, between 30-50 cm in depth (Taylor, 2013). A number of finds including cask staves and lids; coir cordage; leather patches and other items were also found within the clay deposit (Veth et al., 2013). Chief investigators Peter Veth, Vicki Richards and project diver Mike Nash, collected cores of the deposit and sediment in and around the wreck. Although clay is proposed to have been a popular form of ballast material in the 19th century (Taylor, 2013), little contemporary or historic material exists regarding its use in Australia. This material makes it difficult to pinpoint particular locations in which clay was collected or how widespread the practice was. If clay-rich sediments are confirmed to be ballast, *Clarence* could be the first example of a clay ballasted vessel to be located and sampled in the country (Veth et al., 2013).

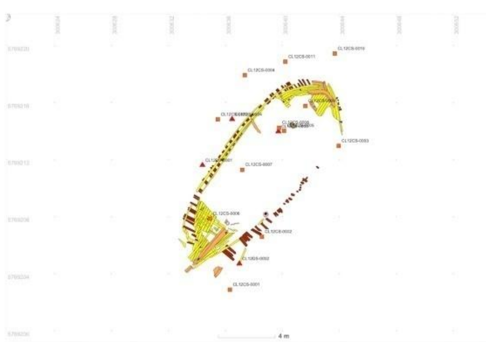

Fig. 1 Short core and grab sample locations in the wreck of the Clarence. (A. Khan)

This discovery and questions surrounding future preservation of the wreck stimulated investigations into micro-sedimentary environments and taphonomy associated with the site and the stability of sediments lodged in and around the wreck. Key objectives were to compare mineral signatures in the 'ballast' from clay-rich sources on the seabed and coastal foreshores to confirm the likelihood of clay-rich sediments being ballast. In order to address these questions, sediment samples were collected from the surrounding onshore landscape and analysed using XRD in March 2013 along with samples collected in April-May 2012 from the wreck and adjacent seabed at the Research School of Earth Sciences (RSES), ANU. This analysis was also undertaken to identify the extent of sediment preservation or damage within the wreck. XRD is considered a cheap, fast and relatively effective method for identifying and analysing clay minerals. Although some criticisms have been made in regards to its overall accuracy, if correct precautions are taken it can be a useful tool in identifying clay mineral variations in sediments (Kahle et al., 2002).

Procedures and Methods

Sample Sets and Sampling and Preparation Techniques.

Initial XRD analysis was carried out on nine sediment samples collected by project divers as part of the *Clarence* excavations.

- **Set A:** CLI1-0006-9. Samples retrieved as either grab samples or undisturbed cores from the wreck site. Locations are shown in (Fig. 1).
- **Set B:** CLI2-0001-4 and CLI2-0010. Samples retrieved as either grab samples or undisturbed cores from the seabed surrounding the wreck. Locations are shown in (Fig. 1).

XRD analysis was then carried out on 15 samples taken from offshore geography directly associated with Port Phillip Bay during a field trip to the area in March 2013. At the *Clarence* site, the major mechanism by which terrestrial-derived fine grain sediments enter the near shore zone will be run-off plumes after heavy rain. Cliff collapse and direct wave erosion of regolith at cliff bases will make minor contributions (Cardno, 2011). The bedrock and saprolites sampled also approximate the range of deposits likely to be forming seabeds around and beneath the locations where *Clarence* foundered. The following samples were collected based on the above assumptions.

- **Set C:** SPA1-15. 50-200g of well sorted deposits were collected from five locations across the headlands of Port Arlington and St Leonards (Fig. 2) and stored in clear zip-lock bags. Samples were retrieved from exposures and sections along low cliffed shorelines and one low elevation intertidal clay-pan. Locations and sampling design aimed to produce representative examples of sediments in the complex weathered geology and regolith of Bellarine shorelines, which are actively eroding and releasing sediments into the near shore zone.
- SPA1-5 and 12-15 were collected from eroding cliff sections.
- SPA 6 and 7 were collected from an intertidal weathered outcrop.
- SPA 8, 9 and 10 were collected from Edwards Point, where a salt-pan and mudflat are situated. This site was chosen and sampled as an example of a sediment sink where fine clay-silts have settled from an intertidal water column

behind a bay bar sand barrier. These provide a first-order proxy for sediments/clay mineralogies likely to deposit out of the Port Phillip Bay water column.

- SPA11 was sampled from St Leonards Beach, seaward of the adjacent mudflat at Edwards Point.

Methods

Analysed sediment from Set A was carefully removed from cores in order to avoid contamination of clay-rich deposits with overlying loose marine sediment. Analysed sediment from Set B was sampled at 2cm intervals in order to collect a representative sample of surface and underlying sediments on the sea floor. Therefore, XRD analysis on all Set B samples (except CLI2-0004 and CLI2-0010) was conducted twice per core and conducted on all aspects of the sample, including miscellaneous marine sediment. Analysed sediment from Set C was prepared in order to produce an accurate representation of coastal landscape formations. All remaining sediment from Sets A, B and C were retained in separate storage containers at the RSES, ANU. All sample sets were prepared and analysed using powder XRD and clay separation methods. Powder XRD was carried out with a SIEMENS D501 Bragg-Brentano diffractometer equipped with a graphite monochromator and scintillation detector, using Cu K-alpha radiation. Bulk samples were milled for 10 min in ethanol with a McCrone Micronizing Mill, and dried at 40°C. Samples were suspended on a side-packed sample holder and analysed from 2 to 70° 2-theta, at a step width of 0.02°

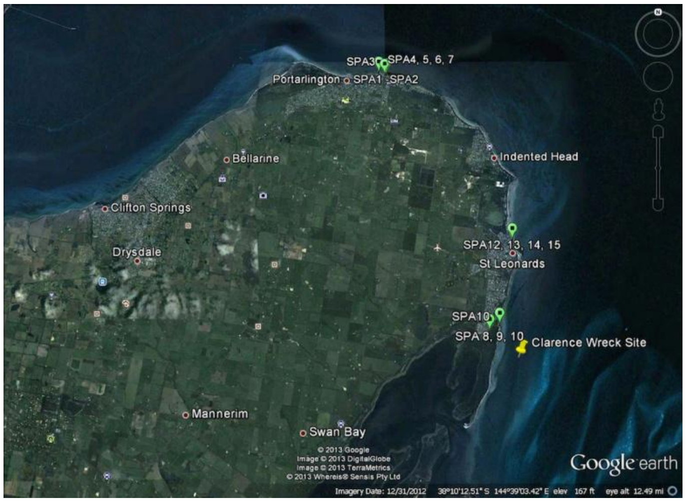

Fig. 2 Locations of SPA1-15 sediment samples surrounding Port Phillip Bay. (Google Earth, 2013)

and a scan speed of 1° per minute. Clay separation was performed by the settling method and samples prepared according to the Millipore Filter Transfer Method (Moore and Reynolds, 1997). Clay samples were analysed after Mg-saturation (scan range 2-42° 2theta, step width 0.02°, scan speed 1°/min), saturation with ethylene glycol (2-32°, 0.02°, 1°/min), and heating to 350°C (2-28°, 0.02°, 1°/min) and to 550°C (2-28°, 0.02°, 1°/min). Results were interpreted using the Bruker AXS software package Diffrac*plus* Eva 10 (2003) for identification, and Siroquant V3 for quantification (using the bulk scan).

Results

Results are shown in Tables 1, 2 and 3 and Fig. 3. Table 1 represents results from Set A, collected from within the wreck. Table 2 represents results from Set B, collected from the sea floor surrounding the wreck site. Table 3 represents results from Set C, collected from surrounding coastal geologies in Port Phillip Bay. Fig. 3 illustrates variations in mineralogical compositions in Sets A and B, specifically with regards to kaolinite, plagioclase and clinopyroxene quantities. Small quantities of vermiculite were present throughout all sample sets: Set A contained an average of 0.50 ± 0.03%, one sample in Set B contained 0.4% vermiculite and three samples in Set C contained traces of the mineral (SPA5, 0.1%, SPA7, 0.4% and SPA 14, 0.6%). Calcite and aragonite were not well represented in Set A, however CLI-0009, contained 5.1% calcite and 6.2% aragonite. Quartz, likely to be associated with loose sand particles from the surrounding seabed, was abundant in Sets A (Mean (M) = 29.9 ± 6.0%) and B (M = 27.5 ± 1.0%). Sets A and B contained halite that can be attributed to trace levels of salt water retained in samples during preparation. Small quantities of illite/smectite were present in the

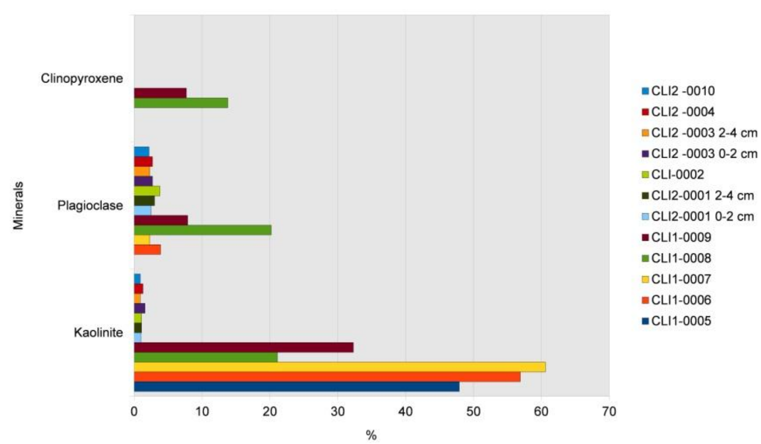

Fig. 3 Mineralogical percentages of kaolinite, plagioclase and clinopyroxene in Sets A and B. (A. Zubrzycka)

majority of Set B samples (M = 0.4 ± 0.3%) while only one sample in Set A, CLI-0005, contained the clay mineral (5.6%). The presence of clinopyroxene was unique to Set A samples CI10008 (13.8%) and CLI10009 (7.7%), which were collected in direct association with clay-rich deposits in the wreck (Fig. 3). Large quantities of kaolinite were present in Set A (M = 43.7 ± 16.7%). In comparison, Set B samples were clay poor, containing only trace levels of kaolinite (M = 1.1 ± 0.2%) (Fig. 3). Plagioclase was present in both Set A and B with Set A containing an average of 6.8 ± 7.9% and Set B an average of 2.7 ± 0.5% (Fig. 3). Calcite and aragonite were found in abundance in Set B (M = 51.2 ± 2.5% and 13.5 ± 0.8%, respectively).

All Set C samples, except SPA11 (beach sand), were clay rich and contained little to no traces of feldspars, such as plagioclase (0%) and K-feldspar, which was only present in SPA3 (1.8%). In comparison, Set A contained an average of 3.3 ± 2% K-feldspar. Only one sample in Set C, SPA10, contained aragonite (15.3%) probably associated with a midden deposit or activity associated with higher sea levels or storm surges. Clay fractions from SPA1 and 8 were dominated with illite/muscovite (27.4% and 32.9%, respectively). Clay fractions from SPA4 (74.3%), SPA5 (89.3%), SPA6 (85.6%) and SPA15 (87.8%) were dominated by kaolinite. SPA7 contained the largest quantity of kaolinite at 96.3%. Overall, the mean quantity of kaolinite within Set C was 40.1 ± 37.8% (as opposed to M = 43.7 ± 16.7% in Set A and 1.1 ± 0.2% in Set B). SPA2 was dominated by goethite (52.6%), which was not present in Set A or B. Set C contained a mean average of 24.8 ± 25.3% quartz. This average was reached by excluding SPA11, a sample of beach sand containing 97.7% quartz. Average quartz quantities in Set C including SPA11 is 29.7 ± 30.8%. Anatase was present in SPA4 (3.9%), SPA5 (4.6%), SPA6 (2.9%) and SPA 7 (2.7%), all collected from low lying cliff sections. Because a number of samples, especially those within data sets A and B contained halite and/or calcite inaccurate results relating to quantification of minerals and clays in the final stages of data analysis were present. In order to account for these irregularities total clays with and without these minerals have been included in all tables.

Discussion

The hypothesis that clay deposits accumulated in *Clarence* after becoming submerged in its present position as a result of slack water “fines settling” within the vessel is inconsistent with data accumulated from the XRD analysis of all samples. This can be observed by comparing mineralogical variability and clay quantities from all sample sets, especially those in Set A and B. Some of the most significant results came from samples CLI1-0008 and CLI1-0009, collected from clay-rich deposits thought to be ballast. These were the only collected and analysed samples to contain clinopyroxene. All samples in Set A contained high percentages of kaolinite, however, CLI1-0008 and 0009 contained significantly less kaolinite (21.1% in CLI1-0008 and 32.3% in CLI1-0009) than analysed samples collected from the wreck, which contained an average of 55.1 ± 6.5% kaolinite (Fig. 3). CLI1-0008 and 0009 also contained higher quantities of plagioclase and K-feldspar than other samples (see Table 1 and Fig. 3). Thus, clay-rich sediment, thought to be ballast, found near the schooner's ceiling planking contains a unique mineralogical composition compared to other sampled sediments within the wreck structure. The hypothesis that clay-rich sediments may have accumulated on the wreck over time, taking on the appearance of clay ballast, is also unlikely based on results from Set B. If clay rich sediments derived from the surrounding landscape had settled on the wreck via storm surges or tidal and current activity, it is likely that similar clay minerals would be present on the adjacent seabed. However, low quantities of kaolinite and a complete lack of clinopyroxene in Set B suggest that this is not the case (Fig. 3).

XRD results from Set C also indicate that there is no mineralogical relationship between sediments collected from surrounding coastal geologies and clay-rich sediments found within the wreck. In regards to the stability of sediments within the wreck, comparing mineralogical data from sample Sets A and B indicates that bioturbation and damaging ocean currents have had minimal effect on what may be *in situ* clay sediments directly associated with the wreck. This can be observed via the presence of calcite in Set A where the mineral is only present in CLI-0009 (5.1%). However, biased sample preparation in the lab may be associated with this result, i.e. shell fragments and other material being removed from Set A and B samples prior to milling.

Conclusion

The wreck of the *Clarence* can provide researchers with valuable information on the history of shipbuilding and ballast use in Australia during the early to mid-19th Century. The location of the wreck also carries the potential to inform researchers of the detrimental effect environmental and anthropogenic activity may have on the survival of the *Clarence* and other wrecks in similar locations. The discovery of clay-rich sediment during the 2012 excavation gave researchers a unique opportunity to investigate both of these questions using identical methodologies. Results from XRD analysis successfully differentiated samples collected from the seabed adjacent to the wreck, clay-rich sediments collected within the wreck and sediment collected from the surrounding coastline. This suggests that clay-rich sediments are unlikely to have been transported to the wreck via factors such as storm surges or ocean currents carrying fine grained sediments from the adjacent coastline. Findings suggest that clay sediment within the *Clarence* shipwreck is likely to be clay ballast or another form of clay material, emplaced in the vessel at some point during its working life. Results were further emphasised due to the presence of clinopyroxene, plagioclase and kaolinite in samples directly associated with clay-rich deposits found near the hull of the vessel. The survival of clay-rich sediment within the wreck suggests that although natural and anthropogenic activity can have a detrimental effect on the preservation of underwater cultural heritage, it is possible for archaeologically significant material to be well preserved within these contexts. In order to reach a more definitive conclusion on the likelihood that clay-rich deposits are indeed ballast, more data from Australian built wrecks and archival material is required. It is also suggested that further comparative analysis of clay-rich sediments found on the wreck and the surrounding landscape be undertaken using alternative analytical methods.

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Biography

Adele Zubrzycka *is a Masters of Archaeological Science candidate at the Australian National University. Her Masters thesis examined built structures and material culture associated with the first Presbyterian mission station established in Vanuatu (1848). Her research interests lie in historical archaeology with an emphasis on the colonial period in South Asia, specifically the production and trade of indigo in India.*

Anthony Barham *is a geoarchaeologist by training with a specific interest in landscape stratigraphy and archaeological site environments. His research spans Regolith and Soil Science, Quaternary Science and Archaeology. He has a long-standing interest in the Holocene geomorphological development and human occupation record of Australian tropical coastlines, especially regional syntheses of archaeological and palaeoenvironmental records in Torres Strait, and recently on the Kimberley coast of Western Australia (WA). Current research includes evaluating impacts of past tsunami events (and cyclone storm surges) in WA using archaeological data sets.*

Dr Ulrike Troitzsch *is the X-ray Diffraction Laboratory Manager at the Australian National University, Canberra. Her thesis was based on the crystal structure and thermodynamic properties of titanite solid solution* $Ca(Ti,Al)(O,F)SiO_4$. *Her research interests lie in experimental and metamorphic petrology, mineralogy, crystal chemistry, solid solutions, ceramics granites and migmatites crystal structure determination and refinement.*

	CLI1-0005	CLI1-0006	CLI1-0007	CLI1-0008	CLI1-0009
Location	Close to ceiling planking near foremast stump	Near keelson, stern end	Centre of wreck	Close to ceiling planking near foremast stump	Near ceiling planking, bow end
Goodness of fit X^2	3.16	3.80	3.31	4.09	3.55
Minerals					
Quartz	34.9	22.5	25.2	36.6	30.4
Plagioclase	-	3.9	2.3	20.2	7.9
K-feldspar	-	3.6	3.2	4.8	5.0
Halite	1.2	5.9	2.5	0.5	0.6
Calcite	-	-	-	-	5.1
Illite/smectite	5.6	-	-	-	-
Vermiculite	-	0.2	0.7	0.6	0.8
Aragonite	-	-	-	-	6.2
Gypsum	-	-	-	-	2.1
Illite/muscovite	10.4	7.0	5.5	2.4	1.9
Kaolinite	47.9	56.9	60.6	21.1	32.3
Clinopyroxene	-	-	-	**13.8**	**7.7**
Total	100	100	100	100	100
Total clay	**63.9**	**64.1**	**66.8**	**24.1**	**35.0**
Total clay w/out carbonates or halite	**64.7**	**68.1**	**68.5**	**24.2**	**39.7**

Table 1 CLI1 samples collected from *the Clarence* wreck site, April/May 2012. (A. Zubrzycka)

	CLI2-0001 0-2 cm	**CLI2-0001 2-4 cm**	**CLI-0002**	**CLI2-0003 0-2 cm**	**CLI2-0003 2-4 cm**	**CLI2-0004**	**CLI2-0010**
Location	Stern end, starboard side.	Stern end, starboard side.	Stern end, starboard side.	Bow end, starboard side.	Bow end, starboard side.	Bow end, port side.	Bow end, port side.
Goodness of fit X^2	2.76	2.55	2.52	2.43	2.47	2.29	2.38
Minerals							
Quartz	28.5	30.6	28.4	28.7	25.4	24.8	26.1
Plagioclase	2.5	3.0	3.8	2.7	2.3	2.7	2.2
Halite	1.3	0.6	1.2	1.0	1.6	0.9	1.1
Calcite	51.3	48.7	48.2	49.3	53.6	54.7	52.5
Dolomite	1.6	1.1	1.1	1.2	1.0	1.0	1.2
Aragonite	12.5	12.5	14.2	14.7	13.6	13.3	13.9
Vermiculite	-	0.4	-	-	-	-	-
Illite/muscovite	1.3	1.0	1.5	0.5	1.2	1.0	1.8
Illite/smectite	-	1.0	0.5	0.3	0.4	0.3	0.3
Kaolinite	1.0	1.1	1.1	1.6	0.9	1.3	0.9
Total	100	100	100	100	100	100	100
Total Clay	**2.3**	**3.5**	**3.1**	**2.4**	**2.5**	**2.6**	**3.0**
Total clay w/out carbonates and halite	**6.9**	**9.4**	**8.8**	**7.1**	**8.3**	**8.6**	**9.6**

Table 2 CLI2 samples collected from adjacent sea floor associated with *the Clarence* wreck site, April/May 2012. (A. Zubrzycka)

	SPA1	SPA2	SPA3	SPA4	SPA5	SPA6	SPA7	SPA8	SPA9	SPA10	SPA11	SPA12	SPA13	SPA14	SPA15
Goodness of fit X^2	2.97	2.55	3.39	3.57	4.85	4.51	5.15	4.32	4.07	3.42	3.04	3.89	3.99	4.59	4.51
Minerals															
Quartz	44.1	28.1	64.9	-	0.5	0.3	-	14.9	13.9	25.9	97.7	74.4	55.4	17.6	8.4
K-feldspar	-	-	1.8	-	-	-	-	-	-	-	-	-	-	-	-
Goethite	10.7	52.6	-	1.6	3.4	7.4	-	-	-	-	-	-	10.9	14.9	-
Hematite	-	-	-	14.8		3.1	-	-	-	-	2.3	2.8	-	-	-
Halite	-	-	-	5.4	1.8	0.7	0.6	14.7	42.8	9.4	-	-	-	-	1.9
Calcite	14.7	4.4	6.5	-	-	-	-	1.8	-	11.0	-	-	-	-	-
Aragonite	-	-	-	-	-	-	-	-	-	15.8	-	-	-	-	-
Gypsum	-	-	1.6	-	-	-	-	1.3	3.7	1.5	-	1.1	0.7	-	-
Smectite	-	9.6	-	-	-	-	-	-	-	-	-	-	-	-	-
Vermiculite	-	-	-	-	0.1	-	0.4	-	-	-	-	-	-	0.6	-
Illite/ muscovite	27.4	3	20.8	-	0.30	-	-	32.9	16.8	18.8	-	-	-	1.9	1.9
Illite/ smectite	-	-	-	-	-	-	-	17.5	10.6	7.5	-	-	-	-	-
Kaolinite	3.1	2.3	4.4	74.3	89.3	85.6	96.3	16.9	12.2	10.1	-	21.7	33.0	65.0	87.8
Anatase	-	-	-	3.9	4.6	2.9	2.7	-	-	-	-	-	-	-	-
Total	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Total Clay	30.5	14.9	25.2	74.3	89.7	85.6	96.7	67.3	39.6	36.4	0.0	21.7	33.0	67.5	89.7
Total clay w/out carbonates and halite	35.8	15.6	27	78.5	91.3	86.2	97.3	80.6	69.2	57.1	0.0	22.3	33.0	67.5	91.4

Table 3 SPA samples collected from geology surrounding Port Phillip Bay, March 2013. (A. Zubrzycka)