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BETWEEN METALS AND THREADS: AN ARCHAEOMETRIC APPROACH TO METALLIC ARTEFACTS FROM YAGUACHI CHIEFDOM BURIALS (GUAYAS BASIN, ECUADOR)

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En memoria de mi tía Pau

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ABSTRACT

The Yaguachi chiefdom was one of the largest groups in the region prior and during Spanish contact. The Yaguachi polity was part of the Milagro-Quevedo archaeological culture, who occupied the entire Guayas Basin in the central and southwestern Ecuador between Guayaquil and the Andes during the Integration (800-1400 CE) and early Hispanic Period. This was a highly organized society with a complex political and social organization, evidence of long distance trade, metal jewelry, mound construction and chimney burials. During excavations on the lower Guayas Basin, the 'Vuelta Larga' burial mounds were identified. Some of these burials contained offerings of various types, including local goods like pottery, and foreign ones like metal artefacts, shell beads and obsidian. This research will attempt to determine the metal artefacts' composition, the relation between artefact's composition, color and class, possible connection to other sites or cultures of the Andes and the source of the fibers attached to some of the artefacts, through the use non-destructive analytical techniques: stereomicroscopy, X-ray fluorescence spectrometry, scanning electron microscopy and environmental SEM. From these analyses, connections to other sites might be drawn based on artefacts' metallic composition and manufacturing technique.

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CHAPTER 1: PRE-HISPANIC ANDEAN METALLURGY

PRE-HISPANIC ANDES

Figure 1: Andes mountains. (Image taken from Google Earth).

The Andes is the area in South America that covers the western edge of the continent, and included southern Colombia, Ecuador, Peru, Bolivia, Chile and Argentina. This region was initially settled by hunter-gatherers between 10,000 and 13,000 BCE that migrated from Asia through the Bering Strait (Orlove 1985). Agriculture, domestication of plants and animals, the development of crafts, metallurgy, large scale architecture, and political centralization developed with no connection to the Old World until the Spanish Conquest in 1532 (Orlove 1985).

Time frame	Cultural development
13,000-8,000 BCE	Hunter-gatherers arrival to the Andes
3200 BCE	Start of political centralization in the Andes
3000 BCE	Beginning of domestication of crops and animals
2000 BCE	Beginning of metallurgy
1000 BCE	Spread of metallurgy

Table 1: Time-line of main Andean cultural developments. Based on Orlove 1985; Bray 1971; Easby 1966.

The Prehispanic Andean population domesticated a wide variety of crops and animals throughout its existence. Cotton, beans and gourds cultivation started around 3000 BCE, peanuts around 2500 BCE, potatoes were domesticated by 2000 BCE and other crops, now of worldwide importance, like maize, squashes, avocados, chili pepper, guava and sweet potatoes by 1800 BCE. Animals, like the guinea pig were domesticated by the latter mentioned period (Orlove, 1985). Different types of crafts were also developed in this area; pottery and loom weaving were perfected along with agriculture; monumental architecture began around 2000 BCE and metallurgy was refined and started spreading during the first millennium BCE (Orlove, 1985). These factors were the reason for the growth of these once small societies into the fully organized Inca state as found by the Spanish conquistadors.

ANDEAN METALLURGY

It is said that when the Europeans arrived in the 'West Indies', they were astonished by the metal objects these 'uncivilized' people were able to produce. Many chronicles mention that the gold work found in the area could rival the best in the Old World (Bray 1971; Easby 1966), even though the refined metallurgical tradition had less time to develop and get perfected than had the Old World techniques.

The first evidence of the use of metals and tool creation in the Americas comes from the Great Lakes, in 4000 BCE North America, where copper was hammered in order to create tools (Easby 1966). This technology did not arrive with the Asian migration, but was developed independently from the Old World. In the Andes the oldest metal artefact comes from the Titicaca Basin, in southern Peru, where hammered gold artefacts dating to 2000 BCE have been uncovered (Aldenderfer et al. 2008); the earliest use of copper, for its part, comes from Northeast Argentina and dates to 1400 BCE in the form of a copper mask (Scattolin et al. 2010).

Figure 2: Location of first metallurgical evidence in the Andes.

During the following centuries, and most probably between 1500 and 1000 BCE, diverse social and technological changes lead to new metallurgical techniques were born and spread through the continent, with gold work being the main one due to its raw availability in the area (Easby 1966; Lleras & Ontaneda 2010). There is no unique origin of metallurgy technology, but several all over the Andes with the Paracas, Nazca, Chimu and Moche in Peru, the La Tolita-Tumaco in Ecuador and Colombia, the Muiscas in Colombia and later on, the Incas within the borders of the *Tawantinsuyu,* having the most noteworthy traditions (Bray 1971; Easby 1966; Lechtman 1984a, 1984b; Lleras & Ontaneda 2010). The mining, production and use of metal objects disseminated slowly in the Andes and reached the whole area with the Inca conquest (Lechtman 1985). By the time of the Spanish conquest metallurgy had reached its height, with many chronicles mentioning the quality of the gold work and even describe some techniques the indigenous people used during the metallurgical process (Bray 1971; Easby 1966).

Metal	First evidence	Area	Use
Gold	2000 BCE	South Peru	Jewelry
Copper	1400 BCE	Northern Argentina	Adornments, masks.
Copper-silver	1000 BCE	Northern Peru	Tweezers, axes, needles, agricultural tools and state symbols
Copper-arsenic	850 CE	Bolivia Highlands, southern Peru and northwest Argentina.	Needles, axes, hoes and status objects
Copper-tin	1000 CE	Andes	Adornments, needles, tweezers.

Table 2: Based on information by Hosler 1988, Scanttolin et al. 2011, and Taylor 2013.

In South America two distinct metallurgy traditions (Figure 1) have been identified one that spread from Central America and reached Colombia and the other that developed in the Central Andes, which include Ecuador, Peru and Bolivia, with its heart in northern Peru (Hosler 1988; Taylor 2013). Gold and gold-copper alloys are the most commonly used in lower Central America and Colombia, where this material was shaped into masks, personal adornments and figurines by a variety of casting methods including lost-wax casting (Bray 1971; Hosler 1988). In the Central Andes region, the emphasis was in the color of the final object and copper alloys were used to create tools and objects through sheet-metal techniques (Hosler 1988; Lechtman 1984a; Rehren & Temme 1994).

Figure 3: Map of South America with the main metallurgical traditions. Based on Bray 1971; Hosler 1988; Lechtman 1984a, 1984b; Rehren & Temme 1994)

ANDEAN MINING

The raw materials used for the manufacture of metals in the Andes were usually extracted from nearby deposits, which is the reason for the huge variety of alloys found in the area; each society worked with the material available to them. Gold, for its part, was exploited by most societies starting in around 1000 BCE due to its relative abundance in the area (Bray 1981; Hosler 1988). Arsenic bronze, despite being readily available, can only be found in sites from Ecuador to Chile starting around 1400 CE, mainly near deposits of sulfosalt minerals – including tetrahedrite-tennantite, enargite and arsenopyrite – due to the dissemination of the techniques developed under the Inca rule (Lechtman 1996). Tin bronze, on the other hand, can be mainly found in southern Peru, the Bolivian altiplano near Tiwanaku and in the highlands of northwest Argentina, where deposits of cassiterite are abundant (Lechtman 1996).

ALLOYS

Different alloys, developed in the Andes, dominated Andean metallurgy until the arrival of the Spanish, each one with diverse properties, colors and uses. This widespread use of alloys throughout the Andes was partly due to the lack of purifying techniques for precious metals like silver and gold (Lechtman 1985), but the use of specific alloys for specific uses had more cultural reasons. There is evidence that specific alloys were developed simply due to their final surface color, this being the reason for the development of intensive hammering techniques that resulted with the enrichment of the surface of the metal sheet with the wanted precious metal (Lechtman 1985).

Gold and Tumbaga

The closest equivalent of an Iron Age in South America was what Zevallos Menéndez (2005) denominated Gold Age, because the people of this area of the world used gold not only for jewelry and adornments, but for everyday tools. Gold, silver and copper were often used to produce the same everyday tools, as the value of metals for these groups was not related to the raw material and its purity, but to the final artefact and its symbolism (Zevallos Menéndez 2005). Golden artefacts were produced and used by different South America groups for over 2500 years, until the Spanish conquest, when the conquistadors started a large scale exploitation and exportation of this raw material (Lechtman 1985; Zevallos Menéndez 2005).

According to several chronicles, after Francisco Pizarro took *Atahualpa* prisoner, he requested a room filled with gold and another one with silver as ransom. Pizarro got what he requested, but when the Spanish conquistadors melted down the artefacts, they discovered that despite appearing pure gold or pure silver, the objects were actually impure alloys of those precious metals with copper (Zevallos Menéndez 2005). One of these alloys is commonly referred to as '*tumbaga*', word that comes from the Malay word '*tembaga*'. This alloy is now known to be a copper-gold or copper-silver-gold alloy and was used by the South American societies to manufacture decorative objects of golden color (Blust 1992). Historically, the first mention of this alloy in the Americas comes from Columbus, who encountered it during his travels. However, archaeological evidence shows that it originated in northern South America, most likely modern day Colombia or Venezuela before around 200 CE and spread to Central America and Mexico, the Guianas, Ecuador and northern Peru by the time of the European arrival (Blust 1992; Bray 1971).

Tumbaga was given its characteristic gold surface, highly appreciated by the society, through either hammering, depletion, amalgam or fusion gilding or electrochemical replacement plating (Bray 1971). In this process,

> *"[...] gold and copper are alloyed in varying proportions, and through some simple chemical means the surface copper is removed, thus leaving a gilded exterior that resembles pure gold" (Blust 1992:447).*

This technique gave copper, a base metal – or in some cases silver – the appearance of gold, even when the percentage of gold in the alloy was very low (Bray 1971). While hammering and annealing gave copper-rich *tumbagas* a golden surface; silver-rich *tumbagas* required other chemical treatments to enrich the gold portion of the alloy that resulted in the golden color in the surface (Lechtman 1985). The latter described complex method involved dissolving the silver with natural acidic minerals, technique later adopted and perfected by the Moche and the Chimú (Lechtman 1985).

Copper-silver

Before the development of *tumbaga*, the Andean people developed an alloy of copper and silver, which was highly appreciated and used due to its malleability and toughness (Lechtman 1985). This alloy was used to create adornments through constant hammering and annealing, which gradually formed a copper depleted and silver enriched surface (Lechtman 1984, 1985). The alloy ingot was hammered into thin metal sheets, which were then turned into different objects with greater strength than pure silver or sterling silver (Lechtman 1985;

Taylor 2013). During the hammering and annealing, the copper is lost from the surface through oxidation, resulting in a bright silver surface (Lechtman 1985). This alloy was popular in the coast of Peru and examples of its use can be found in objects found in Moche, Chimú and Chincha sites (Lechtman 1985).

Copper-Arsenic and Copper-Tin

During the Andean Late Intermediate Period (900-1100 CE), one of the most commonly used metallurgical products was the alloy of copper and arsenic, mainly produced in the north coast of modern day Peru (Lechtman 1991). It is believed that the minerals used to produce this alloy were enargite and arsenopyrite, which could be found mainly in the high sierra and in the north coast valleys of the central Andes (Lechtman 1991). Copper-arsenic, was one the most prevalent metal in use in the central Andean region from 900 CE until the European conquest; it was widely produced and used in the Central Andes, specifically in modern-day Ecuador, Peru and Bolivia. Tin-bronze, on the other hand, was also developed during this time but was not commonly used until the Inca conquest and expansion during the 1400s (Lechtman 1991). The production and use of these alloys has been associated with the use of sulfide ores of copper and long-distance exchange (Lechtman 1991).

METALLURGICAL METHODS

The Inca Empire took control of most of the Andes by 1450 CE, taking full advantage of all the extraction, smelting, metalworking, soldering, welding, gilding and silvering techniques that have been developed throughout the *Tawantinsuyu* before their arrival (Rutledge and Gordon 1985). Most of the pre-Inca techniques were still used by the time the Europeans arrived. Pedro Cieza de León describes, in one of his chronicles, a wind furnace known as *huaim* or *huayras* used for smelting and mining by methods similar to placer, shallow shaft and strip mining (Bray 1971; Easby 1966). Other chronicles also mention large-scale metal workshops in the *Chan Chan* area in Peru and the denominated '*patios de indios*' in Colombia and Ecuador (Bray 1971; Easby 1966).

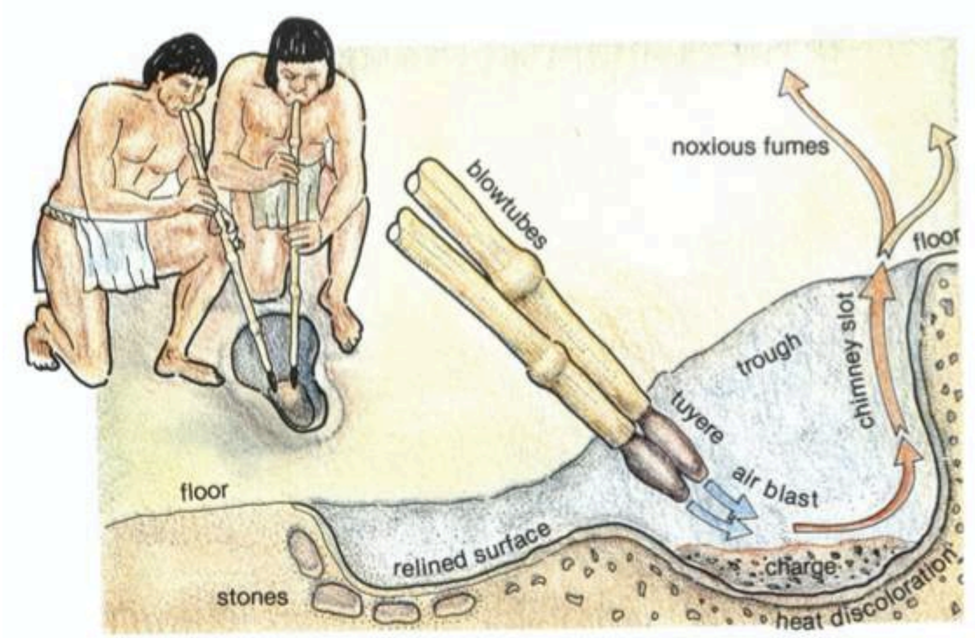

Figure 4: Artists reconstruction of a late arsenical copper smelting furnace. (Shimada el al. 1983).

The most widespread pre-Inca technique for metal production were the aforementioned *huayras*, three feet high terra-cotta cylindrical wind furnaces, used for smelting different ores (Bray 1971; Easby 1966). They could be found usually on hillsides or windy areas in Peru, Bolivia, Argentina and Chile (Bray 1971; Easby 1966). The smelting process was done by placing the crushed ore and charcoal inside the furnace, igniting it and keeping it burning by blowing a current of hot air and carbon dioxide through the holes on the sides of the furnace; the metal eventually settled and was drained from the base of the cylinder (Easby 1966).

Other common technique for artefacts manufacture was the use of sheet metal, where the sheet was placed into concave molds and hammered or pressed into high relief shapes. Decoration was added through techniques that involved engraving, chasing designs, inlaying or filigree (Bray 1971; Schorsch 1998). Sheets of metal were also joined to produce three-dimensional objects through hammering and annealing (Lechtman 1984a). As before mentioned, it was the constant hammering that lead to the color change of the metal's surface (Lechtman 1984a, 1984b).

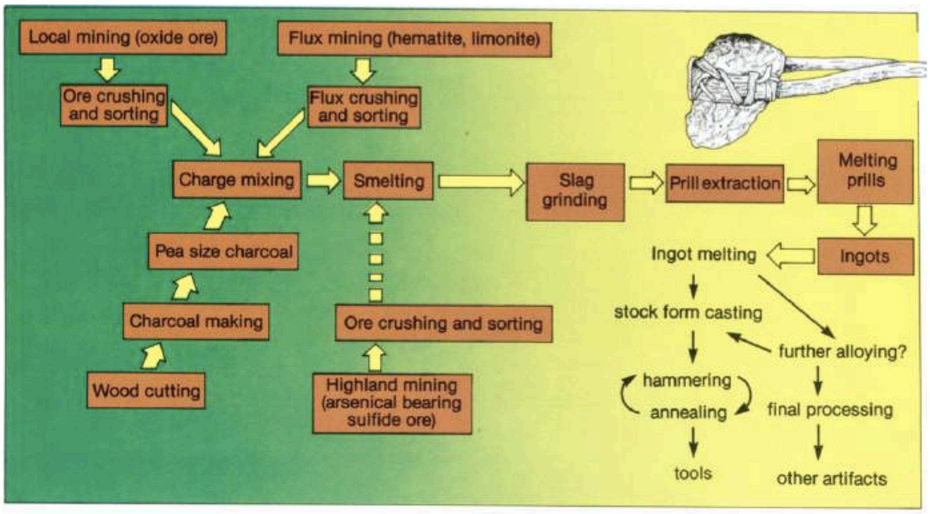

Figure 5: Metallurgical process. (Shimada et al. 1983).

SIGNIFICANCE OF METALS IN ANDES CULTURE

In spite of having developed diverse metalworking techniques and some particular alloys, the Andes lacks an equivalent to the Bronze and Iron ages of the Old World. According to Letchman (1985), the reason may be related the uses and significance that were given to the precious metals in their daily lives. In the Andes, metals were not used for transportation or for warfare purposes, they served a symbolic function with a social, political and religious purpose. They were meant to display social identities, roles and socio-economic status both in life and death; and as such, were worn by the living or placed on the dead as ear and nose rings, pendants, hair pins, bracelets, breastplates, headdresses and masks, or as adornments in clothes (Bernier 2010; Lechtman 1984a, 1985). As Lechtman (1985:15) said, *"status among Andean peoples, as in many societies, was instantly conveyed by what one wore in life and by what one wore at death".*

Metals and access to them was marked by social status. Artefacts made from copper-arsenic alloys were readily available and were used by all the population; copper-silver, copper-silver-gold and gilt copper artefacts were reserved for the low elites while high-karat gold alloys were only available for the high-ranking elites (Shimada et al. 2004). The metals used to display rank, power or religious force were meant to be of very specific colors, they had to look either gold or silver. In this manner, the Andean experimentation with alloys, leading to the development of binary or ternary alloys of copper, silver and gold probably had an aesthetic reason (Lechtman 1984a, 1985). The golden and silver colors had a special ritual and political meaning in the Andes; the silver color was specially associated with a cult that began around 1000 BCE in the central Andes, lasting until the Inca conquest, when silver became a color used and controlled only by the Inca themselves (Lechtman 1985). Copper and bronze, on the other hand, were also highly valued, but were mostly used for tools and to display lower social ranks; in spite of this, the use of copper was the reason Andean metallurgy developed into a complex and refined technique (Lechtman 1985).

CHAPTER 2: PRE-HISPANIC ECUADOR AND METALLURGY

Ecuador, located in the northern part of the Andes had a long pre-hispanic cultural development that is studied in four chronological Periods: Preceramic (10,000-3,200 BCE), Formative (3,200-300 BCE), Regional Development (300 BCE- 800 CE) and Integration (800-1532 CE) (Meggers 1966; Moreno Yanez and Brochart 1997; Porras 1987; Delgado-Espinoza 2002). Sedentism in the area can be traced back to the early Holocene (Stothert 1988; Raymond 2008), while evidence of the first permanent settlements date back to the late fourth millennium BCE (Marcos 2003, Raymond 1993, Raymond 2008). (Table 2)

PERIOD NAME	TIME RANGE	SIGNIFICANCE
PRE-CERAMIC OR ARCHAIC	10000 BCE – 3200 BCE	• Stone tools • Evidence of burial practices • Nomadic life
FORMATIVE	3200 BCE – 300 BCE	• Start of village life • Pottery (oldest in America) – Valdivia figurines • Maize cultivation • Evidence of long-distance exchange
REGIONAL DEVELOPMENT	300 BCE – 800 CE	• Development of urban centers • Production of metal (gold and silver) artefacts (La Tolita) • Early chiefdoms
INTEGRATION	800 CE – Spanish conquest	• Evidence of use of gold, silver and copper artefacts • Hierarchical system • Social and political complexity

Table 3: Ecuadorian archaeological periods and their main significance. Based on Raymond 2008, Stothert 1995, Zeidler 2008, Pearsall 2003, Meggers 1966, McEwan & Delgado-Espinoza 2008.

PRE-HISPANIC COASTAL ECUADOR

PRE-CERAMIC OR ARCHAIC

The first archaeological period in Ecuador is the Preceramic, which starts with the arrival of nomadic groups in the area at around 10,000 BCE, and last until 3,200 BCE, this period is characterized by nomadic groups, hunting, fishing, stone tools, and early development of burial practices (Raymond 2008; Delgado-Espinoza 2002). The coast of Ecuador seems to have been the center of the first permanent or semi-permanent settlements development, with Las Vegas region in the southwest, near the Santa Elena peninsula, being the earliest and best recorded (Raymond 2008). Las Vegas was occupied from 8500 to 4600 BCE approximately, and evidence of hunting, fishing, cultivation and foraging has been uncovered in the over thirty sites that archaeologists have identified; these people seem to have been semi-sedentary (Stothert 1985; Raymond 2008).

FORMATIVE

There's no evidence of other permanent settlements in the area for about a thousand years, until the denominated Formative Period gets underway (Raymond 2008). The Formative, which spans from 3,200 to 300 BCE, was characterized by the development of ceramic, sedentary settlements in the form of villages, maize cultuvation and early inter-regional contact and exchange (Raymond 2008; Zeidler 2008). The Valdivia, which are the main Early Formative culture in the coast of Ecuador, created larger sites with distinct distribution patterns where plant and animal domestication developed alongside a specialized and distinct pottery and stone industry, with female figurines as the main focus (Hill 1975; Meggers et al. 1965; Pearsall 2003; Raymond 2008; Zeidler 2008). The Machalilla, for its part, the main Middle Formative culture of coastal Ecuador, was spread from the Santa Elena Peninsula to southern Manabí coast, and from northern Manabí to southern Esmeraldas (Villalba et al 2006; Staller 2001; Zeidler 2008). The Machalilla practices a mixed economy based on farming, hunting and fishing; as well as pottery and stone production (Pearsall 2003; Zeidler 2008). The Late Formative Chorrera culture, found originally in the Guayas basin is considered the most geographically widespread of Ecuador's pre-hispanic cultures, is known for the zoomorphic, anthropomorphic, and phytomorphic ceramic vessels and influences the entire coastal lowlands and a part of the Andean highlands (Bushnell 1951; Evans and Meggers 1954; Zeidler 2008).

REGIONAL DEVELOPMENT

The Regional Development in Ecuador's coast, which lasted from 300 BCE to 800 CE was characterized by extensive cultural transformations due to constant contact between groups, which lead to the rise of diverse regional cultural styles, various social and political structures, the production and display of luxury goods made of stone, metal and *spondylus*, organized trade, and the development of larger urban centers that were part of a series of chiefdoms and king groups (Meggers 1966; Masucci 2008). The main regional chiefdoms in the coast of Ecuador were Tolita-Tumaco and Tiaone in Esmeraldas, Jama-Coaque in northern Manabí and Esmeraldas, Bahía in central Manabí, Guangala in souther Manabí and Guayas, Tejar-Daule in the Guayas river basin and Jambelí in El Oro (Masucci 2008).

INTEGRATION

Finally, the Integration Period, which lasted from 800 CE until the Spanish conquest in 1532, saw the emergence of integrated political units consisting of confederations of towns under a single leader, with an established hierarchy and political system (Meggers 1966; McEwan & Delgado-Espinoza 2008). The main policy that emerged in Ecuador's coast was the Milagro-Quevedo phase, found in the upper Guayas Basin, Samborondon, at the mouth of the Babahoyo River and in the adjacent Milagro-Quevedo-Taura area (McEwan & Delgado-Espinoza 2008). The aforementioned sites have diverse structures, but share constructions of mounds or tolas as their political or social center, specialized ceramic production, faunal domestication, common burial practices and access to exotic materials like spondylus beads, metalwork, obsidian and textiles (Estrada 1957; Delgado-Espinoza 2006; McEwan & Delgado 2008). The coastal polities in these area developed diverse technologies in order to take advantage of their environment, these included raised field systems, water capturing structures, terrace systems and deep-sea balsa rafts (McEwan & Delgado-Espinoza 2008). Another important coastal policy was the Manteño, in Guayas and central and southern Manabí, who had an elaborate social hierarchy, and constant interaction between their own sites and as far north as Acapulco and along the Peruvian coast due to the formation of ports and trading towns along the Pacific coast paired with their balsa-rafts (McEwan & Delgado-Espinoza 2008).

Years	Chronology	Cultural Manifestation		
	Colonial Period	Spanish Empire		
1534		Inca Empire		
1500		Jama-Coaque	Milagro-Quevedo	Manteño-Huancavilca
1476	Integration			
1000				
800				
500				
0	Regional Development	La Tolita	Bahia	
300				
500				
830	Late Formative	Chorrera		
1000				
		Machalilla		
1500	Middle Formative			
2000				
	Early Formative	Valdivia		
3200				
10000	Pre-ceramic or Archaic	Las Vegas Culture		

Table 4: Ecuadorian Chronology and main cultural manifestations. Based on Raymond 2008, Stothert 1995, Zeidler 2008, Pearsall 2003, Meggers 1966, McEwan & Delgado-Espinoza 2008.

METALLURGY IN PRE-HISPANIC ECUADOR

Ecuador is located on top of several copper, gold, silver, platinum, arsenic and other poli-metallic deposits, which have been exploited since pre-hispanic times metals both through mining and panning (Lleras & Ontaneda 2010). Generally speaking, Ecuadorian metallurgical traditions is characterized by the use of all available metals like gold, silver and copper and of both naturally occurring alloys like Au-Ag and Au-Pt and intentionally created alloys, like Au-Ag, Au-Ag-Cu, Ag-Cu, Cu-As, Cu-Sn and Cu-Zn for the production of both jewelry and everyday tools, without an apparent differentiation of use (Lleras & Ontaneda 2010). The most widely used metals varied from area to area and depended on availability and access to raw materials; in the coast, gold was mainly obtained through panning, while in the mountainous region, mining was the main extraction method; copper was abundant in the Andes, while platinum and silver were found in association to gold in the coast and to the copper in the Andes (Lleras & Ontaneda 2010).

ORIGIN OF METALURGICAL PRACTICES IN ECUADOR

As mentioned in Chapter 1, archaeological evidence suggests that origin of two distinct metallurgical traditions in South America, one located in Peru, where hammering was the chosen technique although, and the other in Colombia, where a lost-wax method was developed and later perfected; both traditions spread through the continent and were in use and constant evolution until the arrival of the Spanish (Zevallos Menéndez 2005). Ecuador also has early evidence of metallurgical practices: the oldest being Salango in Manabí at around 1500 BCE and Putushio in Loja from about 1460 to 865 BCE, this last one holds evidence of long metallurgical traditions and was possibly a diffusion center (Zevallos Menéndez 2005; Lleras & Ontaneda 2010). Independent metallurgical practices developed both in this aforementioned areas and other sites both in South America until 500 BCE, when metal production and use spreads and stabilizes, allowing for some traditions to gain power (Lleras & Ontaneda 2010).

It was been theorized that metallurgical tradition in Ecuador started during the Middle Formative, with Narrio in the Andes and Chorrera in the Coast, although archaeological evidence of widespread metallurgical practices is scarce until the Late Formative or Early Regional Development Periods when stablished traditions start emerging (Lleras & Ontaneda 2010). Until the Incas start process of technological standardization in the XV century, Ecuador was inhabited by diverse groups, each with their own political, social and technological characteristics and traditions, which have been identified thanks to modern archaeological research (Lleras & Ontaneda 2010).

METALLURGICAL TRADITIONS

Metallic artefacts produced through three main techniques have been uncovered in Ecuador: hammering or sheeting, lost-wax or mold casting and platinum sintering, each technique has been associated with specific traditions inside the area (Lleras & Ontaneda 2010). In the region of modern Ecuador, evidence of three pre-incan traditions have been found: the first, somewhat disconnected from the rest of the country, in the northern coast of the province of Esmeraldas, where the La Tolita-Tumaco culture developed; one in the south Andes, in the Cañar and Azuay provinces, with the Cerro Narrío, the Cañari and the Putushio phases at its heart; and finally the Guayas Basin, the contact point of both traditions, and a center of metallurgical production in its own right (Rehren & Temme 1994; Zevallos Menéndez 2005). Despite the refinement and quality of these techniques, they were slowly lost: first with the arrival of the Incas, when Peruvian techniques were introduced and local traditions were replaced or merged and finally disappeared due to the Europeas, who were more interested in mining, than in manufacturing techniques (Lleras & Ontaneda 2010).

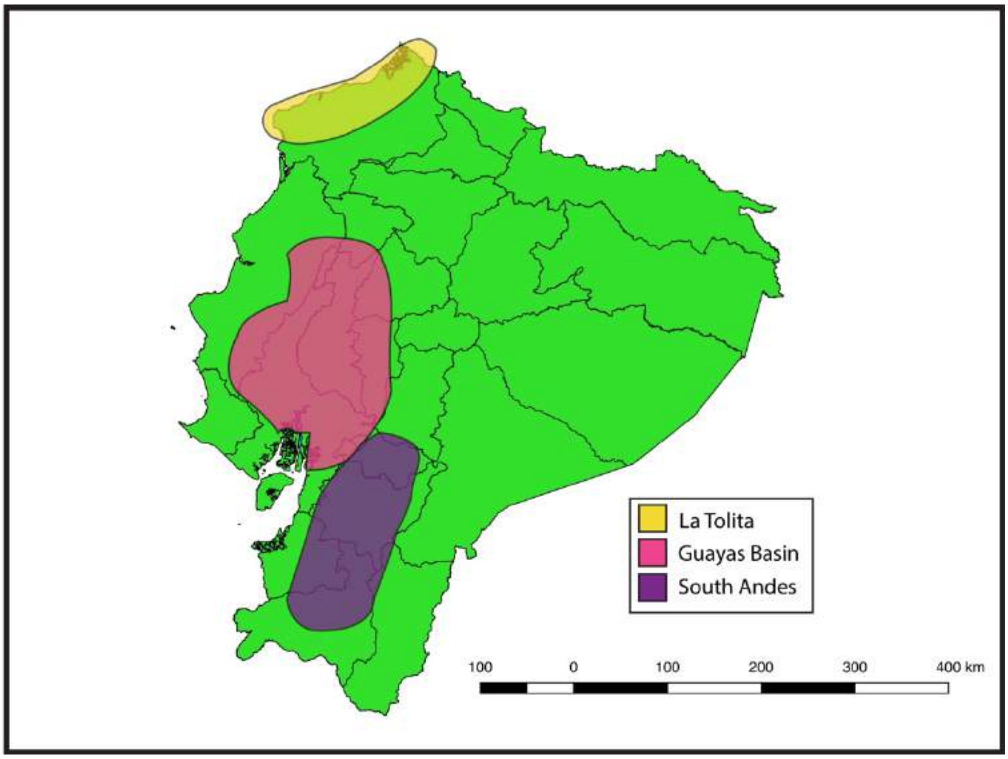

Figure 6: Location of Ecuadorian metallurgical traditions.

La Tolita

The northern coast of Ecuador, in the Esmeralda province is considered one of the main technological development areas from 500 BCE to 200 CE (Lleras & Ontaneda 2010). The material found in La Tolita island includes sophisticated jewelry and unfinished objects, which demonstrates the presence of a workshops in the area (Scott 2011; Lleras & Ontaneda 2010). La Tolita, which earliest occupation dates to 600 BCE, is considered the most important metallurgic site of the region due to the skillfully made artefacts, the unique gold, gold-platinum alloys and copper alloys used in their tradition and the creation of powder metallurgy (Scott & Bray 1994; Lleras & Ontaneda 2010; Scott 2011). This tradition is characterized by the use of pieces of sheet metal, their assembling by means of soldering, welding, granulation or filigree and the incorporation of shell, a second metal and colored stones (Scott & Bray 2011). The Tolita's techniques spread north, reaching Chocó and the Cauca Valley in Colombia, and south covering Manabí's coast and reaching the Guayas Basin, allowing for the appearance of several groups both in Ecuador and Colombia (Lleras & Ontaneda 2010). This metalwork tradition is quite distinct from that of the Andes and Colombia and it spans more than fifteen hundred years, as it survived until the Spanish contact, which is evident in some artefacts that made their way to the Old World few years after conquest (Scott & Bray 1994).

South Andes

The metal artefacts found in southern Ecuador are usually related to tombs or burials, are dated to around 400 CE and tend to indicate contact to coastal cultures of northern Peru, like the Chimu and even the Moche (Rehren & Temme 1994; Lleras & Ontaneda 2010). The objects in this area include objects made of gold, silver and copper-alloys extracted from mines in El Oro province, and there's evidence of diverse crafting methods, like hammering, soldering, granulation, wiring and later lost-wax method (Rehren & Temme 1994; Zevallos Menéndes 2005). The origin of this tradition appears to be Putushio between 1460 to 865 BCE, from where it spread to the south, where it merged with northern Peruvian traditions; and north towards the Cañari and the Puruha areas, until it reached the area of Quito during the V century CE and later the Imbabura province (Zevallos Menéndez 2005; Lleras & Ontaneda 2010).

Guayas Basin

The artefacts related to this tradition were made with gold, silver, copper, lead, and in some cases arsenic alloys and have been found in association to mounds known as 'tolas', which is a traditional building practice in the area due to the river growth (Zevallos Menéndez 2005; Romero-Bastidas. et al 2017). The people of Guayas and Daule river basins adopted some of La Tolita and Cañari-Puruha methods and developed new metallurgical techniques which included: plastic deformation, emptying and surface treatment; creating a new tradition with its center in Santa Elena and the Bahía people as its promoters (Stemper 1993; Lleras & Ontaneda 2010; Romero-Bastidas. et al 2017). The Bahía tradition became the base of other late, yet important metallurgical traditions in the Ecuadorian coast like Milagro-Quevedo and Manteño-Huancavilca (Lleras & Ontaneda 2010). Research carried out about the surge of chiefdom in this area (Stemper 1993; Delgado-Espinoza 2002) have discovered evidence of craft specialization, a relationship between metal jewelry and status, the use of metals for tools and as an exchange medium, as well as evidence of contact with other between groups and with groups outside the Guayas Basin

CHAPTER 3: THE SITE

THE MILAGRO-QUEVEDO PHASE

The Integration Period was the climax of regional interaction due to the development of a more complex and effective agricultural system, a progressive expansion and formation of urban centers that was only stopped by the Spanish conquest (Meggers 1966; Marcos 1985; Delgado-Espinoza 2002). One of the main representatives of this period was the Milagro-Quevedo society, which appeared in the lower Guayas Basin, near the end of the Guayas river around 400CE and expanded to occupy the areas surrounding the Daule river, modern day Guayaquil and eventually the entire basin: from Quevedo to the north, to Tenguel in the south and east from the Chongon Hills to the Andes (Marcos 1985; Stemper 1993; Sutliff 1998; Delgado-Espinoza 2002). The Chono nation, as they were known in Spanish chronicles even managed to spread their influence and power to areas up north until Santo Doming de los Tsáchilas (Marcos 1985; Sutliff 1998; Zevallos Menéndez 1995; Delgado-Espinoza 2002; Lleras & Ontaneda 2010).

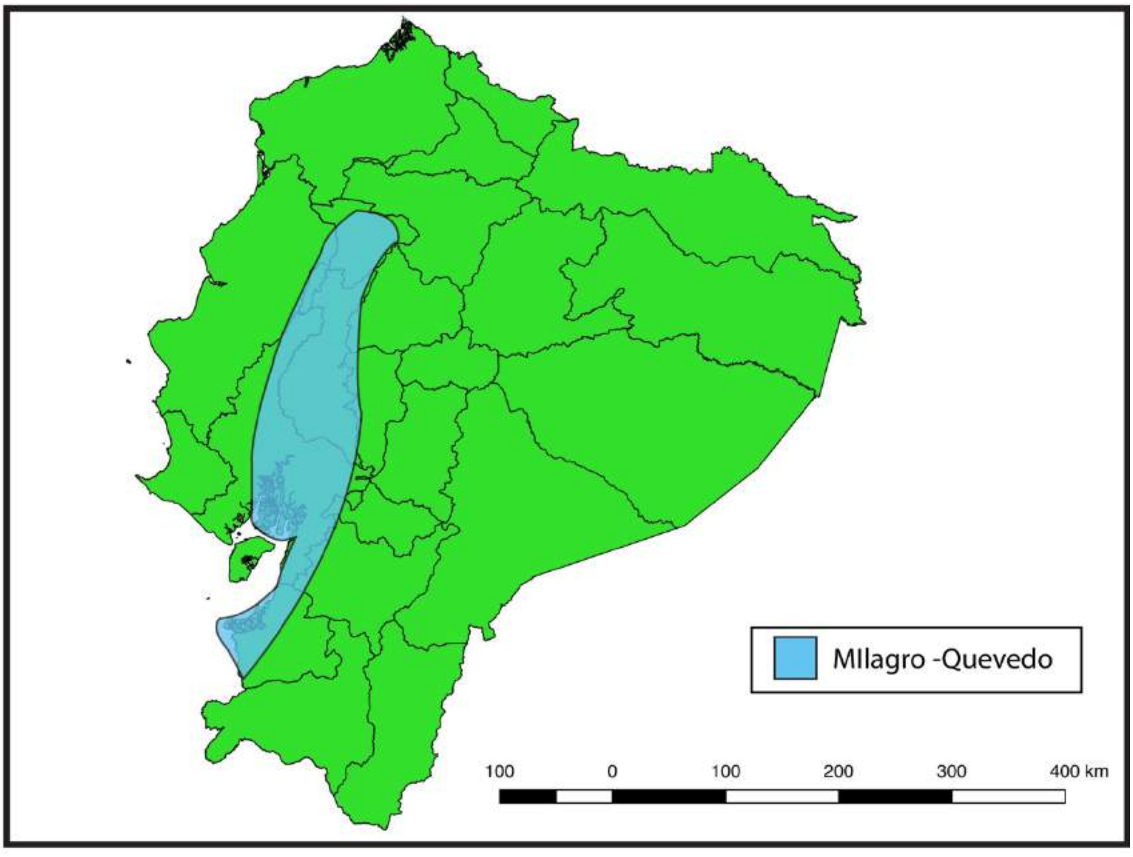

Figure 7: Location of Milagro-Quevedo culture.

This society was characterized by a specialized craft production which include a unique pottery style and production, and metalworking, cotton and wool production, agricultural raised field systems, the construction of houses and urban centers atop mounds, regional and extra-regional trade and chimney burials (Meggers 1966; Marcos 1985; Zevallos Menéndez 1995; Sutliff 1998; Delgado-Espinoza 2002). The Milagro developed an extensive infrastructure and had approximately 50,000 hectares of agricultural 'raised fields' which were subjected to intensive farming by 1200 CE, which were later exploited by the Spanish (Sutliff 1998).

The Milagro-Quevedo established a complex social and political organization, which included many chiefly polities or chiefdoms, with a three-tiered settlement hierarchy, that consisted of regional centers for public and ritual activities, sub-centers for agriculture, trade or craft production and small aldeas or villages (Buys and Muse 1987; Delgado-Espinoza 2002). Their complex social hierarchy is evident in the burial patterns of each settlement and the exotic resources, like metals, precious stones and shells, found in association with them (Estrada 1954; Meggers 1966; Moreno Yanez 1988; Suarez 1991; Sutliff 1998; Delgado-Espinoza 2002). The presence of these exotic artifacts shows the importance in long-distance relationships and suggests a constant interaction between chiefdoms in the area and with groups outside of the Guayas Basin (Sutliff 1998).

THE YAGUACHI CHIEFDOM

The raised fields in Guayas Basin area, were the Milagro-Quevedo formed their chiefdom, were investigated, identified and separated arbitrarily into nine complexes; one of which is the Yaguachi chiefdom area, the focus of this thesis. The Yaguachi polity was part of the Milagro-Quevedo archaeological culture, who occupied the entire Guayas Basin in the central and southwestern Ecuador between Guayaquil and the Andes during the Integration (800-1400 CE) and early Hispanic Periods (Delgado 2002). This was a highly organized society with a three-tiered hierarchy settlement pattern along the rivers, a complex political and social organization, evidence of long distance trade, metal jewelry, mound construction and chimney burials (Delgado 2002).

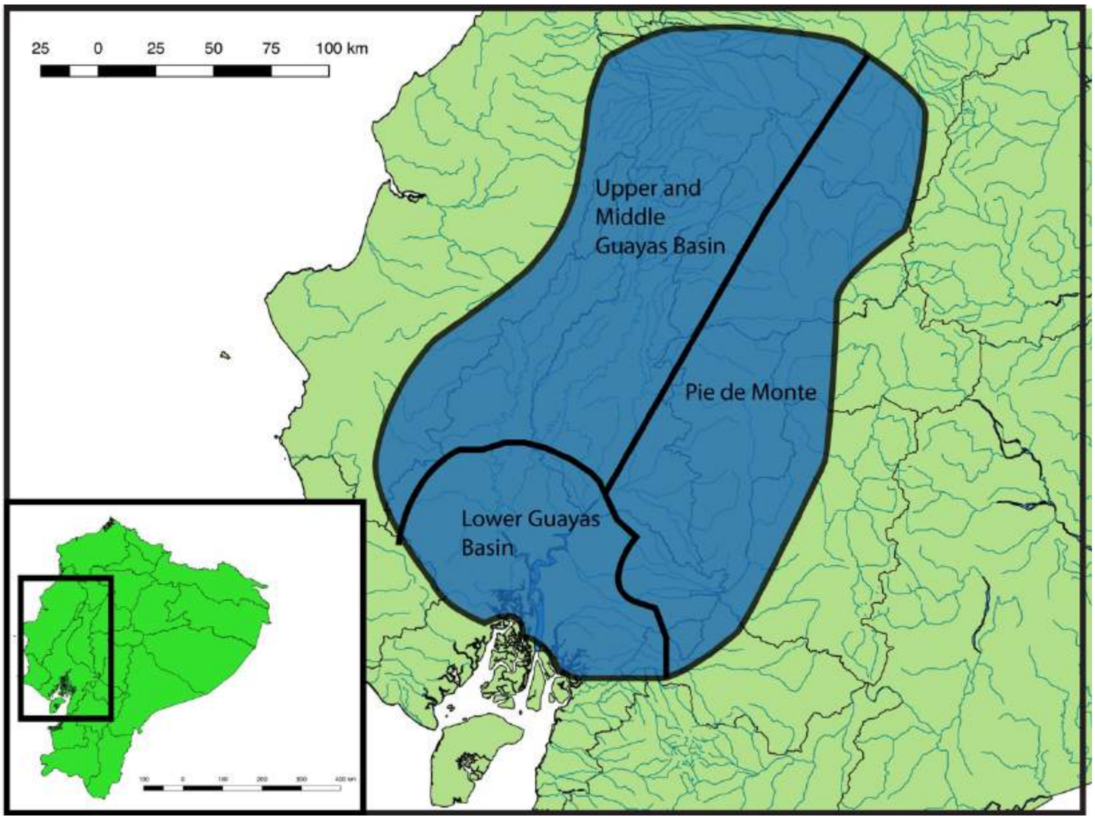

Figure 8: General Map of The Guayas Basin, Coastal Ecuador. (Based on Delgado-Espinoza 2002).

The Yaguachi chiefdom, located between the Guayas river to the west, the Boliche and Taura rivers to the south and southeast and modern road connecting Milagro, Yaguachi and Durán to the northeast and northwest, and consists of two raised field systems: Taura and Durán (Delgado-Espinoza 2002). The settlements in this area date back to 700 CE, and archaeological research show evidence of intensive agriculture and large earth mounds for public, ritual and burial purposes (Delgado-Espinoza 2002). The Yaguachi polity is considered one the the largest regional chiefdoms in the area before and during Spanish contact, according to early chronicles the Yaguachi or Chono people had a well organized sociopolitical system and a clearly stablished site hierarchy (Espinoza Soriano 1988; Muse 1989; Delgado-Espinoza 2002).

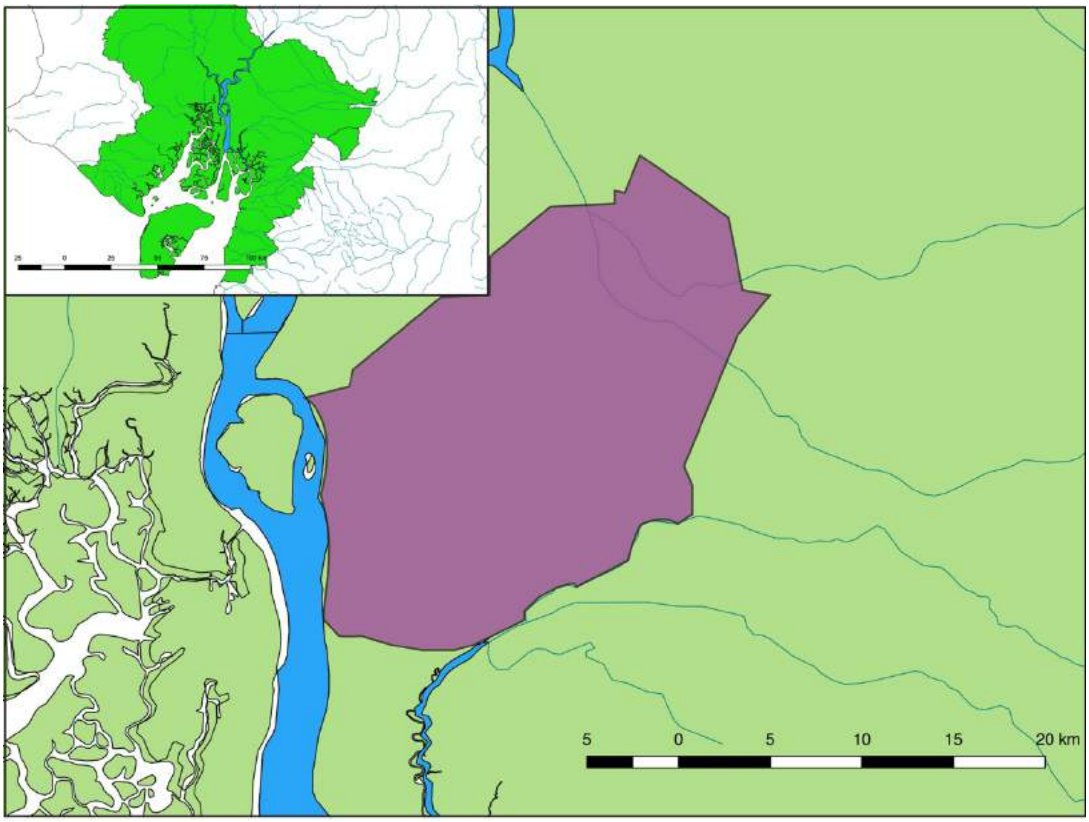

Figure 9: Map of surveyed Area during the 'Yaguachi Project'. (Based on Delgado-Espinoza 2002).

During excavations lead by Florencio Delgado-Espinoza (2002) in the late 1990s an area of 428km^2 in the low Guayas Basin was surveyed; 641 mounds in 16 settlements or sites were identified, these sites seemed to have been organized into the aforementioned three-tiered hierarchy with main regional centers, sub-centers, trading posts, agricultural villages or burial sites and isolated aldeas or rural villages (Delgado-Espinoza 2002). Based on the gathered archaeological evidence the local demography is estimated to have been between 30,000 and 40,000 people and domestic settings were mainly found in the denominated sub-centers and villages. First tier primary centers, the political decision centers, were located close to the main rivers of the area, the Guayas, Boliche and the Taura rivers; second tier centers held diverse functions, but were mainly a link between primary centers and rural villages and had easy access to rivers and *esteros* to allow river-based interregional exchange; while rural villages were found in the most fertile lands, far from the main rivers and populated by the non-elite people (Delgado-Espinoza 2002).

VUELTA LARGA SITE

Vuelta Larga in one of the 16 sites uncovered in the Guayas Basin, it was identified as a secondary site and consists of seven mounds dispersed around the modern village of Vuelta Larga (Delgado-Espinoza 2002). During the aforementioned excavations three of these mounds were tested, VL-T1 was identified as a burial mound and 182 burials were uncovered, 104 of which were primary, and 78 had been identified as secondary (Delgado-Espinoza 2002). The Yaguachi chiefdom had a very complex burial system, which included mass burial, single burial, urn burial, chimney burial and often involved reburial practices, all preformed publicly at one burial mound per site (Delgado-Espinoza 2002).

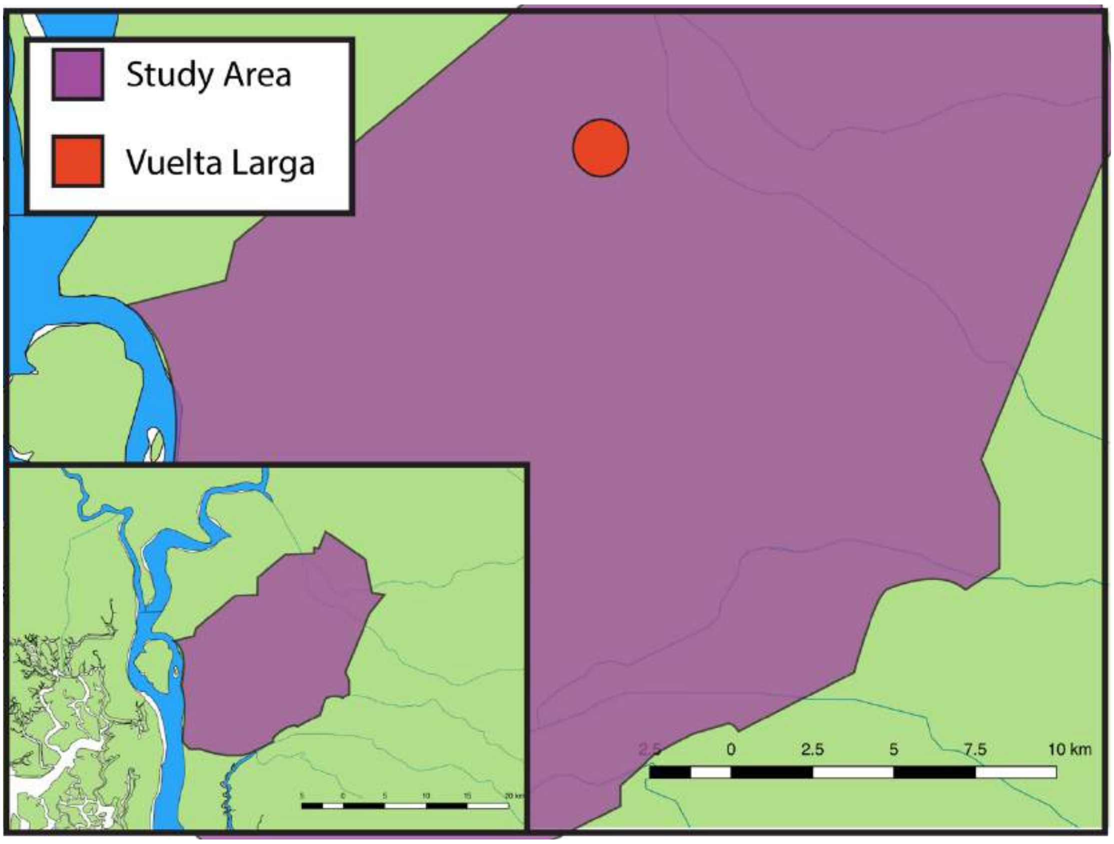

Figure 10: Study area and Vuleta Larga site. (Based on Delgado-Espinoza 2002).

Burials and Offerings at Vuelta Larga

In Vuelta Larga, the primary burials consisted of 104 individuals: 10 children 10 sub-adults, 38 adults and 46 unidentified due to preservation; physical analysis also helped determine the presence of 25 males and 26 females, the remaining 53 individuals' sex was not identifiable (Delgado-Espinoza 2002). The secondary burials, on the other hand, consisted of dismembered individuals, therefore counting, sexing and ageing was almost impossible; these burials, however, contain offerings of various types, including local pottery, and an assortment of apparently foreign goods consisting of metal artifacts, shell beads, quartz beads and one obsidian blade (Delgado-Espinoza 2002).

Figure 11: General view of excavation is Vuelta Larga - Mound 1 (VL-T1). (Delgado-Espinoza 2002).

CHAPTER 4: RESEARCH PROPOSAL

Ecuadorian metallurgy of the Guayas Basin, with the exception of some recent analyses carried out in the INPC, lead by Romero-Bastidas, who studied metallic artefacts from the Manteño-Huancavilca (Romero-Bastidas et al. 2017) and is currently leading other metallurgical projects; has not gone through chemical and physical analyses which can further the knowledge of this tradition. Sutliff (1989 and 1998) discussed the evidence of metallic domestic production and use in the Milagro society, but little was discussed about composition and connections to other sites. Because of this lack of research, little is known about the alloy composition of the Guayas Basin metallic artefacts, their possible origin and connection to other groups, both in Ecuador and in northern Peru; which could lead to hypothesis regarding short and long distance trading.

During excavations preformed by Florencio Delgado, PhD., in the lower Guayas Basin area, 16 sites were identified with 641 mounds for public and domestic functions; the settlement pattern include centers, subcenters and rural vilagges, most of them close to main rivers, which would have aided in exchange relations (Delgado 2002). One of the studied mounds 'Vuelta Larga Mound 1' served funerary purposes and held 182 individuals, in both primary and secondary burials, some of which contained offerings of various types, including local goods like pottery, and foreign ones like metal artefacts, shell beads and obsidian (Delgado 2002). The metallic artefacts, identified visually as copper based alloys, include ear and nose rings, tweezers, jingle bells, beads, needles and plate pieces with fibers attached. These artefacts or their pre-forms, have been hypothesized to come from the closest production site: Sicán on the north coast of Perú, as there is no evidence of local metal production or raw material in the area (Shimada 1985; Delgado 2002).

By means of a collaboration with the CIS-USFQ (Center for Socio-Cultural Research at Universidad San Francisco de Quito) and permits obtained from the INPC (National Institute for Cultural Heritage) in Ecuador, 22 metallic artefacts were obtained for this research, 5 of which had partly mineralized fibers of unknown origin attached. These 22 samples, selected based on macroscopic analysis of conservation status, will be analyzed through the use non-destructive analytical techniques: Energy Dispersive X-Ray Fluorescence Analysis Spectroscopy (EDXRF), Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM/EDS) and in the case of the samples with fibers: Environmental Scanning

Electron Microscopy (ESEM), in an attempt to determine the artefacts' metallic composition and possible connection of this metallurgical tradition to other traditions in the area.

AIMS

GENERAL AIMS

By the use of different analytical non-destructive techniques and minimally invasive techniques in the 22 metallic artefacts obtained from archaeological excavations in the Guayas Basin, it is expected to obtain data that will allow to characterize the objects and contribute to a better understanding of Ecuadorian pre-Hispanic metallurgical traditions.

SPECIFIC AIMS

This research aims to:

- Characterize of the Yaguachi metallic samples.
- Compare these results to other analysis of metallic artefacts in the area and determine the existence of connections in alloy composition.
- Determine the possible use of the different alloys.
- Determine if the Yaguachi chiefdom was in contact and traded with other chiefdoms or groups in the area by comparing these results to research of the results from analyzes of Manteño-Huancavilca and northern Peruvian metals.
- Characterize of the fibers attached to some artefacts.
- Determine possible sources of clothing and knitting techniques.
- Promote the scientific study of South American cultural heritage objects.

RELEVANCE

This research attempts to use analytical methods, usually used in Material Sciences, and collect data that can assist in answering archaeological queries and contribute to the development of the field in Ecuador. The study of pre-Hispanic metallurgical techniques and archaeological metals in Ecuador has been closely linked to object shape, hierarchy markers, the use of color and the development of chiefdoms. Next to no research has involved chemical and analytical techniques to answer archaeological questions until recently. Most studies classify metals and alloys based on macroscopic methods of identification, shape, color, metal oxidation, artefact use or recovery location. By using non-destructive analytical methods to study archaeological materials, we attempt to prove that archaeological research can benefit from the use of these techniques without damaging the artefacts and gaining information that can elucidate different aspects of pre-Hispanic societies.

CHAPTER 5: MATERIALS

ANALYZED MATERIALS

From the metallic artefacts recovered from 'Vuelta Larga Mound 1', 22 were chosen based on state of conservation and shape after a macroscopic examination preformed in Quito by the author of this research and with supervision of Florencio Delgado-Espinoza, PhD., archaeologist who recovered this material in 1999. The selected artefacts and fragments were approved by Florencio Delgado-Espinoza, and later taken to the INPC (National Institute for Cultural Heritage) to obtain the required permits to extract them from Ecuador and analyze them in Europe.

The 22 artefacts, previously identified as copper alloys through visual examination (Delgado 2002), come from secondary burials found in the same mound in the Vuelta Larga site. During the visual examination, fibers were found in 4 of the artefacts. All artefacts and fragments were weighted, labeled and packed in small plastic bags:

ID code	Sample	Weight (g)
VL 009	Ring	2
VL 010	2 beads with fiber	1
VL 013	Tweezers	2
VL 036	Bracelet fragment	1
VL 036	Pectoral fragment	3
VL 036	Not identified fragment	2
VL 106	Beads with fiber	>1
VL 114	Tweezers fragment	>1
VL 1000	Ring	2
VL 1006	Nose ring	2
VL 1017	Ring fragments	>1
VL 1023	Bell	1g
VL 1033	Nose ring	2
VL 1051	Ring fragment	>1
VL 2000.1	Hook	1
VL 2000.2	Undetermined	2
VL 2005	Ring	>1
VL 2005	Bell	>1
VL 3003	Bead	>1
VL 3009	Ring and ring fragment	1
VL 3021	Needle fragment	1
VL 3027	2 axe fragments	1

Table 5: List of samples and weight.

The Vuelta Larga site, as mentioned before was part of the Yaguachi chiefdom, which is known to have been part of the cultural group known as Milagro-Quevedo were known for their metallurgy.

Figure 12: Some metal artefacts found in VL1. (a-VL009, b-VL036.2, c-VL1033, d-VL3021.

METALS IN ANCIENT HUMAN SOCIETIES

Metals are mineral monoatomic solids with a polycristalline structure and high melting points due to strong atomic bonds (Mercier et al. 2002). Human societies have been known to use metals due to their aesthetically appealing appearance, their acquired social significance and their versatility since the third millennium BCE (Varella 2013). Metals, mainly iron, aluminum, copper, silver and gold and its alloys, have been exploited and used by artisans for artefact creation all over the world, in different times and for an endless number or reasons (Mercier et al. 2002; Varella 2013).

The choice of which metal to use depended both in its metal properties and in their significance for the society: malleability, toughness, ductility, strength, hardness and color were some of the properties ancient artisans considered when choosing a pure metal or an alloy (Mercier et al. 2002; Notis 2014). For most of human history, metals were used in alloy form, combinations of two or more metals, either due to the impure nature of their sources, or due to the artisans' choice (Mercier et al. 2002). Alloys, were more commonly used by ancient societies due to their natural availability, their enhanced properties, versatility and their lower melting points (Mercier et al. 2002; Callister 2007).

Copper and Its Alloys

Pure copper is readily found in mines around the world and has a melting point of 1084°C, which means it can be easily melted and shaped into long wired or thing sheets through casting and hammering; it can also be easily mixed with other metals or non-metals like tin, zinc, gold, silver, lead and carbon, to create different alloys, each with a new set of properties, different melting points and colors (Bayley et al. 2001; Callister 2007). Copper-based alloys are known to be tough, hard, resistant to corrosion, have a lower melting point than pure copper, which facilitates casting, and can be strengthened by heat-treatments and cold-working (Callister 2007).

Milagro-Quevedo Metallurgy

The metallurgical tradition of the MIlagro-Quevedo people was developed some time between 400 and 1500 CE (Sutliff 1998; Lleras & Ontaneda 2010). Metal production was of great importance, as it was present in the lives of all members of society, who either participated in the production process or used the final artifact as a social identity or status sign (Sutliff 1998). The grave goods uncovered by archaeological excavations show a wide range of social ranks and spheres, evident in the raw material, the type and the quantity of metallic artifacts found in each burial (Sutliff 1998).

The main features of Milagro-Quevedo metallurgy are the shaping done through cold hammering and annealing, which happened in either small workshops inside homes for personal use or in bigger specialized workshops, and the copper based raw materials used in metal artifact production (Hosler 1998; Sutliff 1998 Lleras & Ontaneda 2010). On one hand, copper and copper-arsenic alloys were used for small domestic objects like needles and hooks, adornments like tweezers, bells, ear and nose rings, giant axes, coin-axes, and large command staffs appear to have been massively produced and widely distributed to both elite and non-elite members of society (Sutliff 1998; Lleras & Ontaneda 2010). Copper-silver alloys, on the other hand, were used selectively due to its scarcity, and were used to produce ritual and ornamental items for the most elites like spiral noserings, rings, bells and tweezers (Sutliff 1998). Finally, gold was the most selective and had restricted circulation; gold, tumbaga, silver and golden copper smaller artifacts like bowls, collars, bead necklaces and nose and ear rings, were produced in smaller quantities and with more specialized techniques like repousée, filigree and granulation for the highest ranking elite (Sutliff 1998; Lleras & Ontaneda 2010).

The difference in importance of the aforementioned raw materials, their availability to the population and the variability in composition had a direct correlation to the access to the raw material source, which many researchers agree was not in the area (Sutliff 1998).

As mentioned before, most of the Milagro-Quevedo metallurgical tradition focused on the fabrication of personal ornamentation, which is evident both in the ceramic iconography, but specially in the offerings found in tombs; using complex and thoughtfully worked metallic nose rings, simple copper earrings, necklaces made with metal, shell and precious stones beads and pectorals made of different copper alloys, gold and silver, depending on the person's status (Zevallos Menéndez 2005; Lleras & Ontaneda 2010). Besides personal ornaments, burial offerings also include high quantities of tools like tweezers, hooks and needles, and dozens of bundles of coin-axes tied together with tread (Lleras & Ontaneda 2010).

Metal Objects as Offerings

The metal objects recovered from burial Mound 1 included earrings, nose rings, tweezers, needles, bells, beads, bracelets and breastplates, some were found with preserved pieces of fabric or thread attached to them. The metals were preliminarily visually identified as copper alloys, mainly copper-gold and copper-arsenic (Delgado-Espinoza 2002). Previous researchers suggested some preforms were brought to the site from the Sicán polity in northern Peru, the closest metal production site, as there is no evidence of local production in the southwestern coast of Ecuador and the raw material is not present in the area (Shimada 1985; Muse 1991; Delgado-Espinoza 2002).

Figure 13: Cranium with metal offerings. VL-T1. (Delgado-Espinoza 2002).

ANCIENT ANDEAN TEXTILES

The societies than inhabited the Andes before Spanish conquest produced an endless number of crafts, including metal-work, wood-carving, stone-work, ceramic and textiles (Ainsworth 1925). Examples of most of these crafts are readily available for study, unfortunately mainly due to hot and moist climate, few archaeological textiles remain (Federman et al. 2006). The available examples of Andean textiles show an impressive, complex, high quality and detailed industry, that produced items of high value inside their societies (Ferman et al. 2006; Bernardino et al. 2015). The oldest textile remains in the Andes comes from the Atacama Desert in Chile, and dates back to around 3000 years ago; with other examples of ancient textiles found in Peru, Argentina, Bolivia and Ecuador, all these textiles appear to have been made with camelid wool (Wheeler et al.1995).

The Andean textile industry depended on two main fibers: camelid wool and Peruvian Full Rough cotton, with the occasional use of strange materials like rabbit hair, bat wings, bird feathers, human hair and shell beads (Ainsworth 1925; Lange et al. 1987). The fiber was either used in its natural colors or dyed blue, red, yellow or brown before being hand twisted into yarn (Ainsworth 1925; D'Harcourt 2002). The wool seems have been the most commonly used fiber and it proceeded from four animals, native to the Andes: llama, alpaca, guanaco and vicuña, these four animals with different colors, including brown, black and white; and textures, coarse, hairy or fine, gave the textile manufacturers some great raw materials to work with and a great range of end products with different added value and importance (Ainsworth 1925; Lange et al. 1987; Wheeler et al. 1995). Finished textiles were worn both in life and death and were used as ritual offerings and household ornamentation (Lange et al. 1987).

CHAPTER 6: METHODS

Metals in Ecuadorian contexts are rarely found, due to the big amount of tomb looters that tend to find sites before archaeologist do. Ecuadorian museums are filled with artifacts bought to these looters, and as such, rarely come with trustworthy context information, so analyzing any artifact found in an archaeological site represents an important opportunity for archaeologist and museums.

ANALYTICAL TECHNIQUES

PRELIMINARY ANALYSIS

Optical Microscopy

Optical microscopy is considered the essential precursor in most analyses or investigations and can be used to inspect the surface microstructure of most materials by producing a magnified image and detecting details invisible to the human eye and decide how to proceed (Pavlidou 2013). In the case of metals, the best option is reflected light microscopy, which can help determine the conservation status, corrosion presence and characterize the studied artefact (Pavlidou 2013; Bayley et al 2008). To avoid using destructive techniques, like the ones needed for metallurgical analysis, a low magnification microscope can be used directly on artefacts before moving on to chemical analysis and scanning electron microscopy.

ANALYTICAL TECHNIQUES FOR METALLURGY

The study of archaeological metals is of great significance in cultural heritage studies, specially in countries where analytical methods have not been used until recently. Chemical and physical analyses can assist archaeologist in answering endless questions, which can only be approached when the artifact's context is known, obtain information about artifact's conservation environment and climate change, compare results with other areas in order to map out trade and interaction routes and can additionally confirm information gathered before by museums, help link their pieces to the correct sites or cultures based on similarities with archaeological artifacts (Bayley et al. 2008).

Each archaeological query can be approached in a number of different ways, based on the nature of the material studied, time and funding constraints, the samples available and the invasiveness allowed (Bayley et al. 2001). Analyses can be qualitative or quantitative, and attempt to determine chemical or mineralogical composition of a material and their origin or source, manufacturing techniques and conservation status (Bayley et al. 2001; 2008). When analyzing metallic objects, their conservation status must be taken into account; metals may be preserved in most environments, but tend to show chemical of physical ageing and might eventually loose their alloy nucleus if exposed to unsuitable and unstable soils, temperatures and environments (Mercier et al. 2002). Ageing in archaeological metals can be found in the form of surface corrosion or in some cases the return of the metal to their original state as a sulphide or oxide (Mercier et al. 2002). Some artifacts can be susceptible to environment changes and prone to rapid oxidization once removed from the soil (Callister 2007; Mercier et al. 2002). In order to preserve the metal's properties and shape, minimally invasive methods are recommended when they undergo chemical and physical analyses.

XRF – X-Ray Fluorescence

When analyzing important, invaluable and unique archaeological artefacts, invasive methods are not advisable, researchers sometimes turn to non destructive chemical analysis in order to solve their questions about their materials. The most common chemical analyses involve the use of X-rays, which thanks to their very short wavelength, can penetrate most materials and assist in material characterization and are versatile enough for the creation of mobile systems (Gigante & Ridolfi 2013).

X-ray fluorescence is one of the non-destructive techniques that uses x-rays to characterize materials. XRF uses a primary x-ray to radiate a sample and induces a periodic displacement in it, which generates secondary x-rays of a characteristic energy to be emitted from it and detected by the equipment (Mercier et al. 2002; Bayley et al. 2008; Pollard & Bray 2014). This secondary energy generates a spectrum, which contains peaks for each element present, a sort of finger-print, each peak's energy and counts is measured through specialized computer software and allows to identify which atom or element it came from and its quantity present in the sample (Bayley et al. 2001; Mercier et al. 2002; Gontrani et al. 2013; Pollard & Bray 2014). X-ray fluorescence can give both qualitative and quantitave information regarding the composition of the analyzed samples (Bayley et al. 2008).

X-ray fluorescence is preformed with a spectrometer, which involves an x-ray source, a detector, which either works using a wavelength dispersive (WD) or an energy dispersive (ED) detector, and a multichannel analyzer (Gigante & Ridolfi 2013). EDXRF is the most commonly used for 'in situ' analysis thanks to the low-power 35-50 kV dedicated x-ray tube and the cooled semiconductor detectors; and can determine element concentration in a solid, powder, liquid or thin film with no sample preparation (Gigante & Ridolfi 2013). This technique allows the detection of the whole x-ray spectrum of either whole artefacts of smaller, prepared samples (Bayley et al. 2001). EDXRF analyzes the surface of metals and is able to quantify the composition of gold, silver and copper alloys as well as determine trace elements when present at a level of 0.1% or more (Gigante & Ridolfi 2013; Cesareo et al. 2016).

XRF have quickly become one of the most widely used technique in archaeology and cultural heritage due to its simplicity, versatility, non-destructive capabilities, multielemental analysis and rapid results (Gigante & Ridolfi 2013; Pollard & Bray 2014). XRF is especially useful for antique alloy identification and quantification due to the heavy metals ancient societies used (Pollard & Bray 2014).

SEM – Scanning Electron Microscopy

Scanning electron microscopy is a chemical analysis dependent upon a beam of electrons and the detection of X-rays, it provides with both a high resolution two-dimensional image and a micro-point chemical information (Pollard & Bray 2014). SEM can be used for characterization and high magnification imaging, due to the use of a wide magnification, which spans from 109 to 1,000,009 and an x-ray detector (Pavlidou 2013). The most commonly setting used for cultural heritage and archaeology is the one where the SEM is coupled with an energy dispersive X-ray microanalysis (SEM/EDS), due to the precision of the characterization result of the use of an electron primary beam, which is easily steerable and focusable (Pavlidou 2013; Pollard & Bray 2014). SEM-EDS takes place in a high-vacuum chamber, where the electrons strike the sample with a micro-beam and produce characteristic X-rays, which are detected and permit the identification of the chemical composition of the sample (Pollard & Bray 2014).

The SEM technique requires an electron source, a series of lenses, apertures for the beam to pass through, controls to position the specimen, an area of beam/specimen interaction that can generate, detect and process different types of signals, a high level vacuum specimen chamber and an electrically conductive specimen to analyze (Bayley et al. 2001; Pavlidou 2013). SEM involves an electron beam being focused through the lenses and scanned over the sample, which causes a series of signals (secondary electrons, backscatter electrons and x-rays) to be emitted from the specimen, each signal gives different information and helps produce images of selected areas and determine chemical composition of specific points (Pavlidou 2013). These signals permit multiple element analysis of single spots or larger predetermined areas, allowing the creation of line scans or maps to show distribution of elements in heterogeneous materials, like metallic alloys (Bayley et al. 2001).

FIBER IDENTIFICATION TECHNIQUES

Environmental SEM

Environmental SEM is a rather new equipment that does not work under vacuum, but is able to create images by generating and manipulating a primary electron beam into striking a specimen, which produces secondary and backscatter electrons (Pavlidou 2013). These secondary electrons strike gas molecules before reaching the detector, which results in a cascading effect that increases the amplitude of the signal; this unique characteristic allows non-conductive specimens to be analyzed (Pavlidou 2013).

EQUIPMENT CHARACTERISTICS

The following diagram shows the analytical techniques used for the purpose of these research and the amount of samples analyzed through each method:

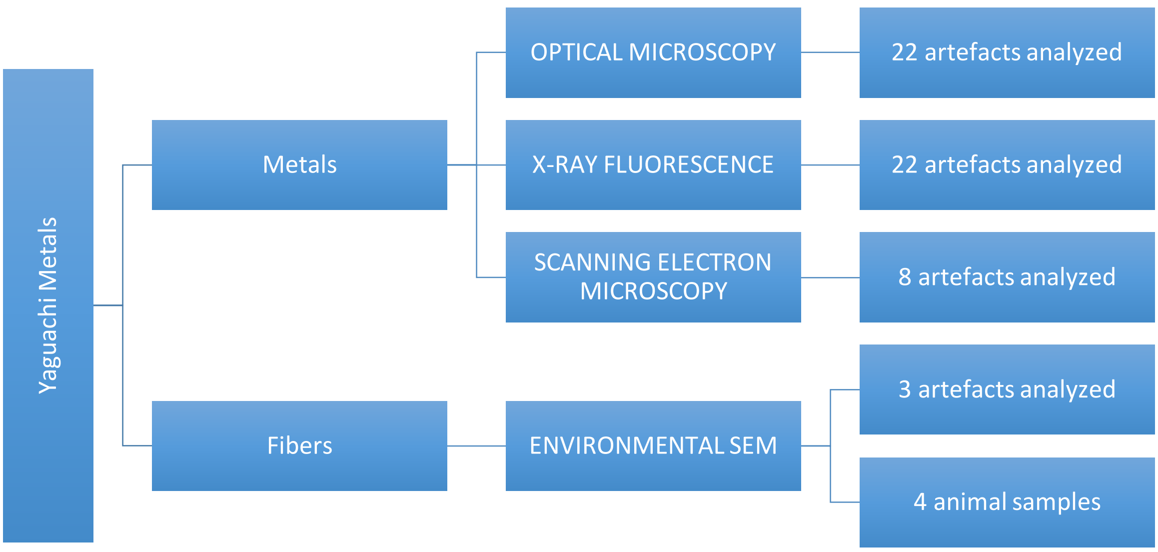

Figure 14: Analysis process of the Yaguachi metals.

PRELIMINARY ANALYSIS

Optical Microscopy

Each of the samples was observed under a Wild M5A stereomicroscope coupled with an eyepiece (10x), a camera adapter, a tube (1.25x) and a set of LED lamps. Details of each of the artefacts were digitally photographed with a high resolution ZEIZZ Axiocam ERc 5s camera with a color CMOS sensor, which provides with 5-megapixel color images. The photographed areas show corrosion, polished regions, details and fibers.

Figure 15: WILD M5A stereo microscope, coupled with led lights and ZEIZZ Axiocam ERc 5s camera.

METAL ANALYSIS

EDXRF

The analysis was carried out with the assistance of Roberto Cesareo, with his own portable equipment. The EDXRF equipment used was composed of a AMPTEK mini X-ray tube with a Ag-anode, and 40kV and 200uA maximum voltage and current; and a 123-Si-drift thermoelectrically cooled detector with an efficiency of 97%, 39% and 14% at 10, 20 and 30 keV, respectively. The equipment irradiated and analyzed an area of approximately 20mm^2 of each artefact, with each sample being placed at a distance between 1.5 and 3 centimeters away from both the X-ray tube and the detector.

Figure 16: EDXRF equipment in use.

The measuring time ranges shifted from 50 to 200 seconds, based on the composition and size of the artefact analyzed. Each artefact had one small area analyzed before and after being polished. Standard copper and arsenic alloys were employed for calibration and quantitative determination of alloy composition.

SEM-EDS

Both SEM and EDS characterization were carried out in the CNR-ISMN laboratory, by using a Cambridge 360 scanning electron microscope equipped with a LaB6 filament and with an energy-dispersive X-ray spectrometer (EDS) INCA 250 and with a four sectors back scattered electron detector. SEM images were recorded at an acceleration voltage of 20 kV.

Figure 17: SEM-EDS system. (www.phy.uniri.hr)

Based on the XRF, 6 artefacts were chosen for SEM-EDS analysis and imaging.

ID code	Sample
VL 036	Bracelet fragment
VL 1000	Ring
VL 1006	Nose ring
VL 2000.1	Hook
VL 3003	Bead
VL 3009	Ring

Table 6: Artefacts analyzed through SEM.

FIBER ANALYSIS

One of the main methods of fiber analysis consists of comparison, which means observing any correlation between fibers from an unidentified source and fibers from a known source in order to determine the possible origin of the first kind (Houck 2009). When considering ancient fibers, researchers encounter either vegetable or animal fibers. Vegetable fibers come from the seed, steam or leaf of plants, like cotton, flax, jute, hemp or sisal; and can be identified by visually examining their internal structure, surface, cell walls, size, shape and thickness or through chemical testing for lignin (Houck 2009). On the other hand, animal fibers, which include wool, angora, cashmere and silk originate from a wide range of animals, including sheep, camelids, rodents, spiders and even worms; and come from their whiskers, guard or fur (Tridico 2009; Houck 2009). Animal fibers can be identified through microscopic visual analysis, where identification of surface characteristics, like scale presence and smoothness, is key, or through chemical analysis, which can uncover the presence of amino acid chains known as protein like keratin or fibroin (Tridico 2009).

Environmental SEM

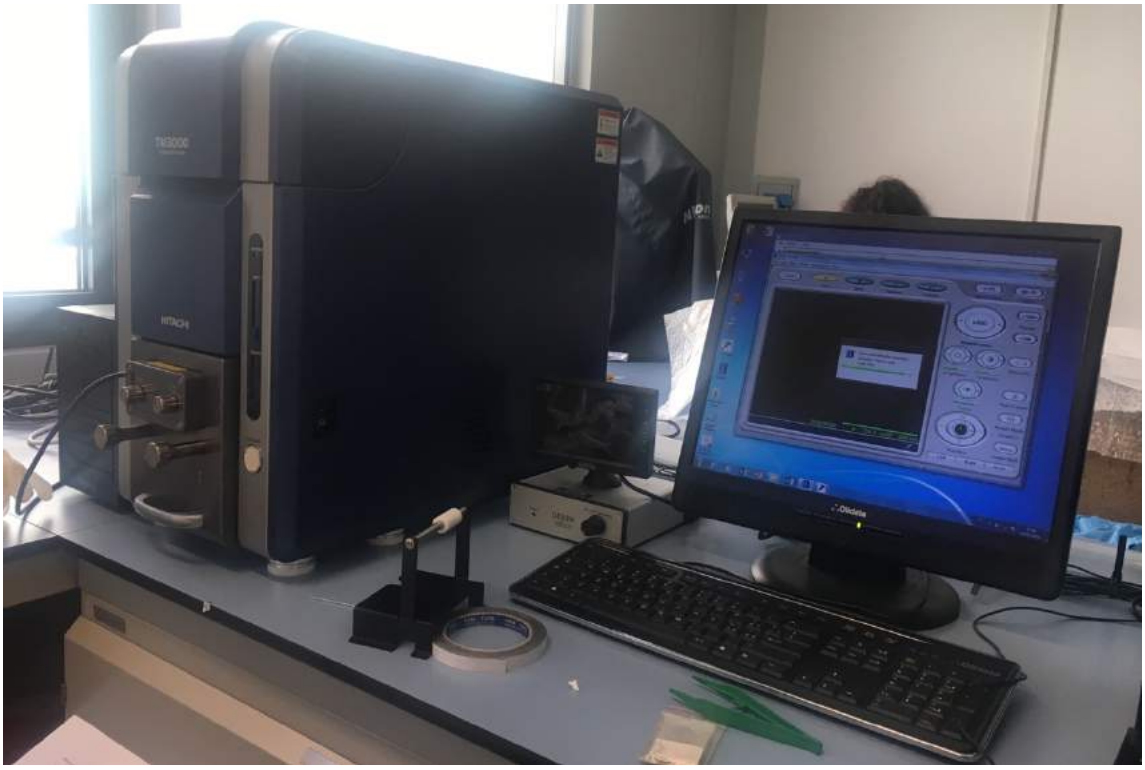

Figure 18: Tabletop Microscope TM3000. Enviromental SEM equipment.

The environmental SEM analysis was carried out with a fully automatic Hitachi Tabletop Microscope TM3000, with a maximum magnification of 30,000x, connected to a PC, where focus, brightness and contrast can be managed. This equipment is used for organic samples, as coating is not necessary and the analysis can be preformed faster than in a normal SEM. Environmental SEM is usually used for imaging purposes.

Three artefacts with fibers were analyzed, and for comparison purposes, 4 raw hairs were also studied under SEM, these hairs came from Ecuadorian llamas and alpacas.

ID code	Sample
VL 010	2 beads with fiber
VL 036.1	Bracelet fragment
VL 036.2	Pectoral fragment

Table 7: List of samples with fibers analyzed.

Animal source	Color
Alpaca	White
Llama	White
Llama	Brown
Llama	Black

Table 8: List of animal hairs analyzed.

CHAPTER 7: RESULTS

The artefacts analyzed, part of burial offerings, were recovered during excavations in the late 1990s and stored wrapped in paper and plastic bags for almost two decades. The first visual examination was done with the help of simple magnifying lenses and allowed the researcher to pick the 22 artefacts analyzed for this thesis. The 22 artefacts, as mentioned before, included both fragments and whole artefacts.

OPTICAL MICROSCOPY

The use of the stereomicroscope showed the presence of a green thin later of corrosion in all of the artefacts, and confirmed the existence on an alloy nucleus. These analyzes also lead to the categorization of the artefacts based on manufacturing technique, apparent core color and presence of fibers

ARTEFACT SHAPE

The categorizing based on artefact main shape lead into three groups, based on technique used for shaping. All of these categories show the Milagro's strong preference for shaping objects by hammering and annealing, which follow the Central Andean metalworking tradition mentioned in chapters 1 and 2 (Sutliff 1998).

Category	Number of artefacts
Wire	11
Thin Hammered Sheets	8
Hollow Beads	3

Table 9: Categories of analyzed artefacts based on manufacturing method.

ARTEFACT COLOR

At first sight all of the artefacts appear green with brown patches, due to the oxidation process they went through for being buried for so long and the remains of soil still attached to them. For the purpose of this research, an area of less than 0.5cm^2 of each artefacts was polished. This polishing revealed the alloy core, underneath the oxidized surface and permitted the identification of the surface color of the artefacts.

Color	ID code	Artefact
Bronze	VL 009	Ring
	VL 013	Tweezers
	VL 114	Tweezers fragment
	VL 1000	Ring
	VL 1017	Rings fragment
	VL 1033	Nose ring

	VL 3021	Needle fragment
	VL 3027	2 axe fragments
Golden Bronze	VL 106	Beads with fiber
	VL 1023	Bell
	VL 1051	Ring fragment
	VL 2000.2	Undetermined shape
	VL 2005.2	Bell
	VL 3009	Ring and ring fragment
Dark Bronze	VL 010	2 bead with fiber
	VL 036.1	Bracelet fragment
	VL 036.3	Not identified fragment
	VL 1006	Nose ring
	VL 2005.1	Ring
Silver	VL 3003	Bead
Undetermined	VL 036.2	Pectoral fragment
	VL 2000.1	Hook

Table 10.: Metal colors and artefacts of each color.

In the case of some artefacts, the oxidation and soil made it impossible to determine their surface color.

Figure 19: Examples of artefacts by colors. Bronze: a-VL100, Gold Bronze: b-VL1023, Dark Bronze: c-1006, Silver: d-3003.

ARTEFACTS WITH FIBERS

The analysis through optical microscopy allowed for the identification of several artifacts with fibers. The fibers were all highly mineralized, which made OM identification impossible. The fibers were either coiled into cords for necklaces or used to create textiles pieces of metals were attached to.

ID code	Artefact
VL 010	4 Necklace beads with fiber
VL 036.1	Bracelet fragment
VL 036.2	Pectoral fragment
VL 036.3	Not identified fragment
VL 106	2 Necklace beads with fiber

Table 11: Artefacts with fibers attached.

The fiber cords were created by hand threating fibers into wool and then coiling several threads together, before placing the beads through them.

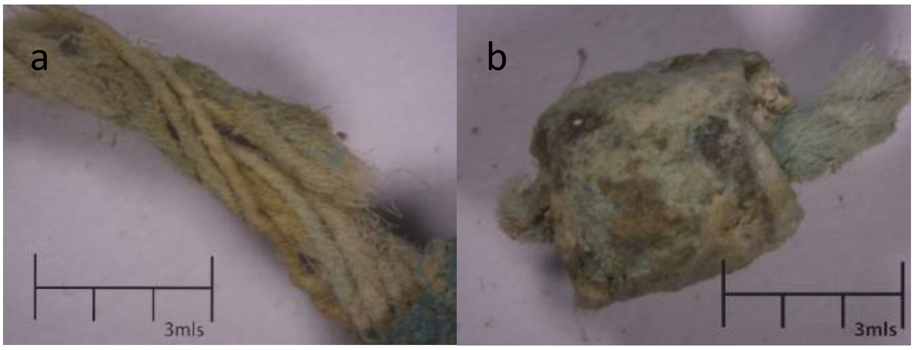

Figure 20: Beads with cords. a-VL010, b-VL106.

The microscopic analysis showed two distinct textile patterns, which can be explained based on the area of the body the artefacts were found on. It seems like weaving patterns were different based on the purpose of the textile, so clothes were made with a specific weaving technique, and the jewelry lining with another. In both cases, one thread was wrapped vertically and another horizontally, but in a matter that created two different weaving patterns. One weaving pattern shows signs of an alternate-warp float weave or a braiding pattern, while the other was a balanced plain weave (Vreeland 1997; Rief 1992; Lopez 2002; Federman et al. 2006).

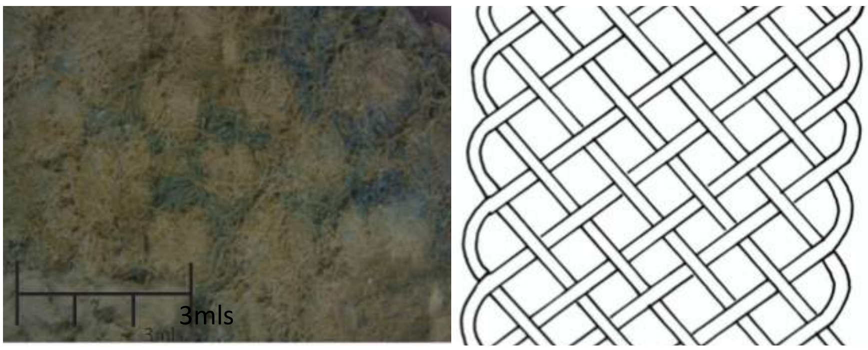

Figure 21: VL036.1 textile pattern and Float Weave pattern (Vreeland 1977).

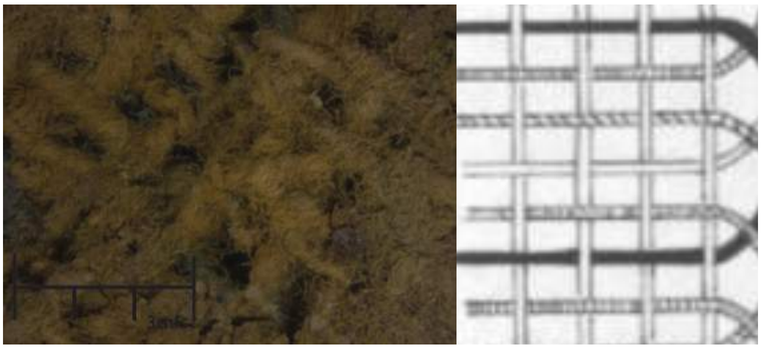

Figure 22: : VL036.2 textile pattern and Plain Weave pattern (Lopez 2002).

X-RAY FLOURESCENCE

The XRF analysis were carried out both before and after polishing, and revealed the presence of several elements in the artefacts, including Fe, Cu, As, Ag, Pb and Br. The iron detected in most samples is related to the soil the artefacts were found on, as its concentration diminished considerably after carrying on a second analysis on a small polished and cleaned area.

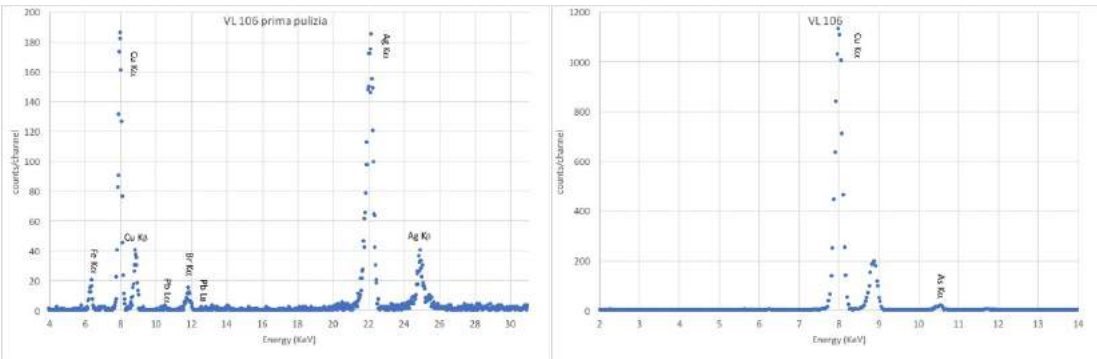

Figure 23: VL 106 spectra before and after polishing.

The results of the analysis done after polishing determine the existence of five different alloys and one pure metal used in the artefacts of this site. All artefacts show high concentrations of copper, which confirms the ongoing believe that copper was the main metal used in the central Andes (Lechtman 1996; Romero-Bastidas et al. 2017), as it was used both for utilitarian artifacts and adornments; analyzing the alloy mixtures and relating them to the objects might help us determine the use of each alloy and their importance in pre-Hispanic societies.

The 6 aforementioned groups were categorized into 3 alloy groups and one pure metal: copper-arsenic alloys, copper-silver alloys, copper-lead alloy and pure copper. Some of these alloys might seem unique, but have also been found in relation to Manteño-Guancavilca contexts (Romero-Bastidas et al. 2017), which, considering there are no mines in the area, might suggest all these alloys and metals came into the Guayas Basin area from the same mine or from the same source through short or long-distance trade.

COPPER ARSENIC ALLOYS

In both the Andean region and Mesoamerica, arsenic-bearing minerals are abundant, and were mined and smelted in high quantities during pre-Hispanic times; in South America this alloy first appeared around 800 CE and kept on being produced throughout the Incan empire until about 1500 CE (Lechtman 1996). Copper-arsenic alloys have been found over a vast area, from Chile to Mexico and is related to either the large number of metals that contain arsenic in their geological occurrences, mainly in large deposits of sulfosalt minerals and partly thanks to the smelting of arsenopyrite with copper ores (Lechtman 1996).

Arsenic fixes itself into copper, and once alloyed together, it is very difficult to separate, creating an arsenic-copper alloy (Lechtman 1996). It is believed that arsenic copper was used for the fabrication of certain type of objects due to this alloy's mechanical properties (Lechtman 1996). Arsenic gives copper increased hardness, ductility, resistance, malleability and higher workability (Lechtman 1996; Romero-Bastidas et al. 2017). Based on the results presented below, we can confirm that certain artefacts were only manufactured with arsenic copper, in this case tweezers, axes, bells, needles and simple thin rings. Some of these objects were mainly used for decoration, while others had a utilitarian and domestic purpose, like weaving and hair holding.

Copper-arsenic alloys were imported in big quantities from mines not yet identified and Milagro-Quevedo metal smiths were able to create a high volume of goods in a production process that involved casting in massive open molds, melting, hammering and annealing (Sutliff 1998).

Group 1: Copper+Arsenic

These artefacts all show a high presence of copper and a presence of arsenic of 1% or less. They are all either utilitarian objects or small adornments, in the case of the tweezers and the axe fragments, they probably had a double function of utilitarian and burial offerings. As they were all burial goods, it is possible the Cu-As alloy might also be related to the social or political standing of the deceased, as these alloy is more common than others found, with 8 out of 22 artefacts belonging to this group.

ID code	Artefact	Cu	As
VL 013	Tweezers	99.70%	0.30%
VL 114	Tweezers fragment	99%	1%
VL 1006	Nose ring	99.50%	0.40%
VL 1017	Ring fragments	99.50%	0.40%
VL 1033	Nose ring	99.90%	0.10%
VL 2005.1	Ring	99.40%	0.60%
VL 2005.2	Bell	99.60%	0.40%
VL 3027	2 axe fragments	99.20%	0.80%

Table 12: Artefacts made from Cu-As alloy.

Group 2: Copper+Arsenic+Lead

These artefacts show a copper base, between 1,5 and 0,7% of arsenic and up to 0.4% of lead. The lead is a strange mineral to add to arsenical copper, but its presence might be explained when the sources are analyzed, as lead can be found in small quantities in copper mines. Considering the low lead presence, this group might be considered to be part of group 1, as for alloy purpose, significance and use.

ID code	Artefact	Cu	As	Pb
VL 1000	Ring	98%	1.50%	0.40%
VL 1023	Bell	99%	0.70%	0.20%
VL 3021	Needle fragment	99%	0.75%	0.40%

Table 13: Artefacts made Cu-As-Pb alloy.

COPPER-ARSENIC- SILVER ALLOYS

Copper-silver alloys are also widely found and used by most pre-Hispanic societies; the Moche, the Chavin, the Sicán and the Chimù in northern Peru were known to use this alloy intentionally for its final color and malleability from as far back as 1200 BCE and created complex and beautiful metal artefacts (Cesareo 2010; Cesareo et al. 2013). The low carat silver, which was made with a copper base, was developed in several societies, including the Moche, this mixture which usually had its surface enriched through depletion gliding was important due to its final color (Cesareo 2010). Silver was found in both small and high amounts in 9 of the analyzed artefacts, which might depend on whether or not its presence was intentional, and what the purpose of the final artefacts was. The source of this alloy is still unknown, but it is believed that it might be found in the southern highlands of Ecuador (Hosler 1994 in Sutliff 1998).

Group 3: Copper+Arsenic+Traces of Silver

These two artefacts, golden bronze in color, show traces of silver as part of their alloy mixture. Both arsenic and silver were found in such small quantities, that they might not be intentional, but associated with the geological source the raw material was extracted from.

ID code	Artefact	Cu	As	Ag
VL 1051	Ring fragment	99.60%	0.20%	0.15%
VL 3009	Ring and ring fragment	99.40%	0.30%	0.30%

Table 14: Artefacts made of Cu-As-Ag alloy.

Group 4: Copper+High Quantities of Silver

These copper based artefacts show a presence of less than 2% of arsenic, a high presence of silver, which is bound to be intentional, in the case of two artefacts, a small quantity of brome and in other two artefacts, a small quantity of lead, these trace quantities were probably related to the geological source of the copper. The amount of silver in the alloy, allow for a wide range of colors, that in this case include: one bronze colored artefact, one bronze colored, two dark bronze, one silver and two undetermined.

ID code	Artefact	Cu	As	Pb	Ag	Notes
VL 009	Ring	74%	0.75%	0.30%	25%	
VL 010	2 beads with fiber	73%	1%	-	26%	
VL 036.2	Pectoral fragment	86%	1.70%	-	12%	
VL 036.3	Not identified	95.50%	-	-	4.50%	Small quantity of Br
VL 106	Beads with fiber	65%	0.50%	-	34%	Small quantity of Br
VL 2000.2	Undetermined	80%	2%	0.50%	17.50%	
VL 3003	Bead	66%	-	-	34%	

Table 15: Artefacts made from Cu-Ag alloy.

The final color of each artefact depends on both the amount of silver in each mixture and in the depletion gliding process, which involves cold hammering and constant reheating, until the desired color is obtained. The constant hammering and in some cases the use of natural acids allows for the copper to be removed from the surface and in this case for the silver to come out and leave a thin film atop (Cesareo 2010). The length of the process and the amount of silver result in different surface colors. The different colors, brightness and shades might have a relation with hierarchical status of the wearer or the purpose of the object, usually ritual or ornamental (Sutliff 1998).

OTHER ALLOYS

Group 5: Copper+Lead

Only one of the analyzed artefacts was an alloy without arsenic. This artefact, dark bronze in color has 0,8% of lead, which might be related to the geological source the raw material came from.

ID code	Artefact	Cu	Pb
VL 036.1	Bracelet fragment	99.20%	0.80%

Table 16: Cu-Pb alloy artefact.

PURE METALS

Group 6: Pure Copper

Out of the 22 analyzed artefacts, only one was found to be a pure metal, in this case pure copper. The artefact is a small wire hook of unknown use. The pureness of the copper is strange and could have been intentional, as most copper geological sources tend to have, as mentioned before, small traces of other elements in them.

ID code	Artefact	Cu
VL 2000.1	Hook	100%

Table 17: Pure copper artefact.

SCANNING ELECTRON MICROSCOPE – SEM-EDS

The SEM analysis was carried out in 6 artefacts, one of each group, chosen based the composition determined by the XRF analysis.

ID code	Artefact	XRF Grouping
VL 036.1	Bracelet fragment	5
VL 1000	Ring	2
VL 1006	Nose ring	1
VL 2000.1	Hook	6
VL 3003	Bead	4
VL 3009	Ring	3

Table 18: List of artefacts analyzed through SEM.

The photographed surfaces of the artefacts show that the metals that formed part of each alloy had different cooling temperatures, as there are areas that were crystallized first, as the metal started cooling down. The images also show hammering marks, that happened during the manufacture of the artefacts.

GROUP 1: VL 1006

The artefact VL 1006 had two areas analyzed. From the XRF, this artefact contains copper and arsenic. Each presented different structures and various degrees of homogeneity. None of the analyzed areas show presence of arsenic, as seen in the XRF, which leads us to believe the arsenic wasn't evenly distributed in the artefact and was probably a trace element from the mines the copper was extracted from.

Area A

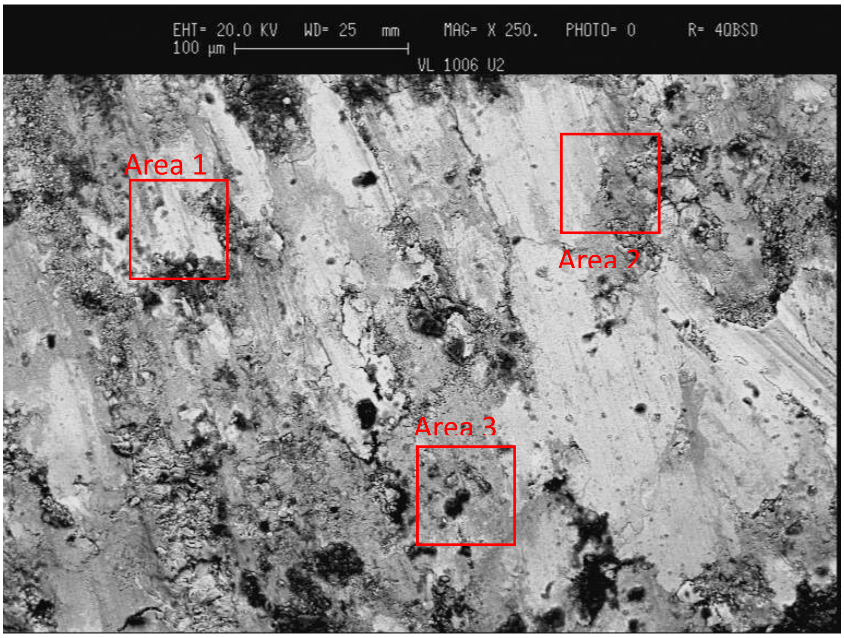

Figure 24: VL 1006, Area A, SEM image.

Area A had 3 smaller areas analyzed, showing presence of C, Si, Cl, Cu and O. The arsenic presence is not registered in this case, probably due to the area selected.

Spectrum	**C**	**Si**	**Cl**	**Cu**	**O**
Area 1	2.69%	0.95%	4.59%	66.73%	25.04%
Area 2	3.17%	2.77%	2.52%	63.86%	27.67%
Area 3	2.94%	2.28%	1.12%	66.48%	27.18%
Area A	2.93%	2.00%	2.74%	65.69%	26.63%

Table 19: Results of EDS in Area A of VL 1006.

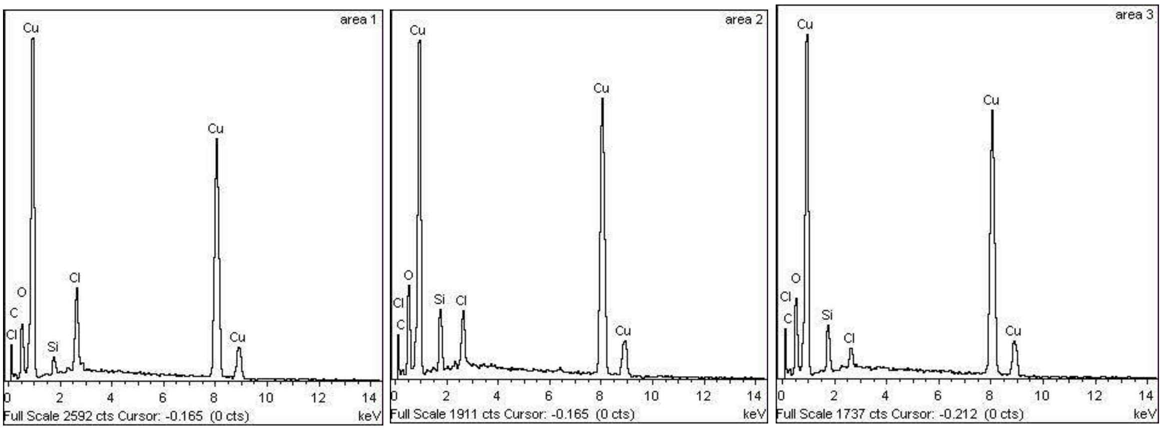

Figure 25: VL 1006, Area A, EDS spectrums

Area B

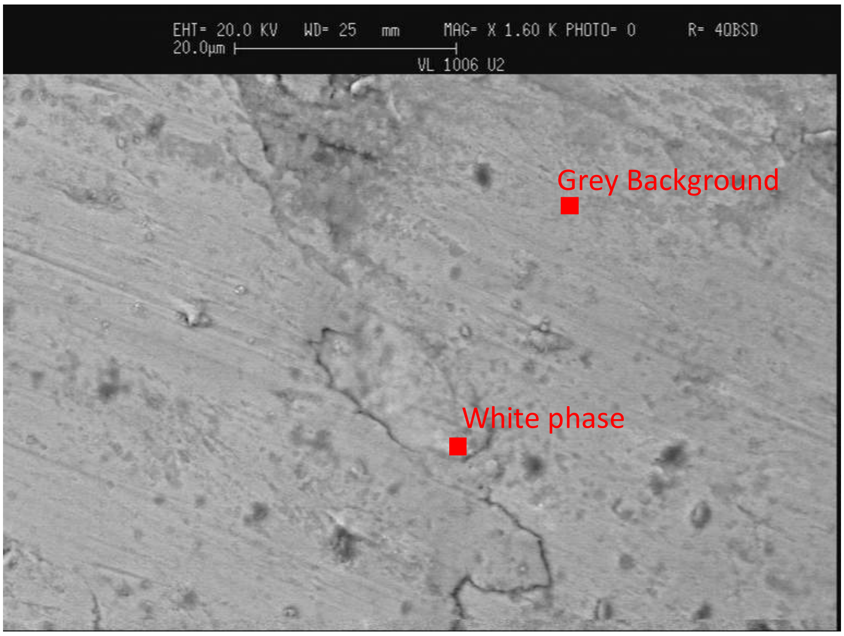

Figure 26: VL 1006, Area B, SEM image.

Area B had two different colors, that were analyzed through point EDS analysis. These analyses revealed the presence of C, S, Cl, Cu, Pb and O. The gray background lacks lead and chlorite, while the white phase show presence of all elements. The white phase contains lead, which leads to the white color and faster crystallization of the phase.

Spectrum	**C**	**S**	**Cl**	**Cu**	**Pb**	**O**
Grey Background	1.37%	0.34%		75.21%		23.08%
White phase		0.41%	1.02%	75.95%	2.66%	19.95%

Table 20: Results of EDS in Area B of VL 1006.

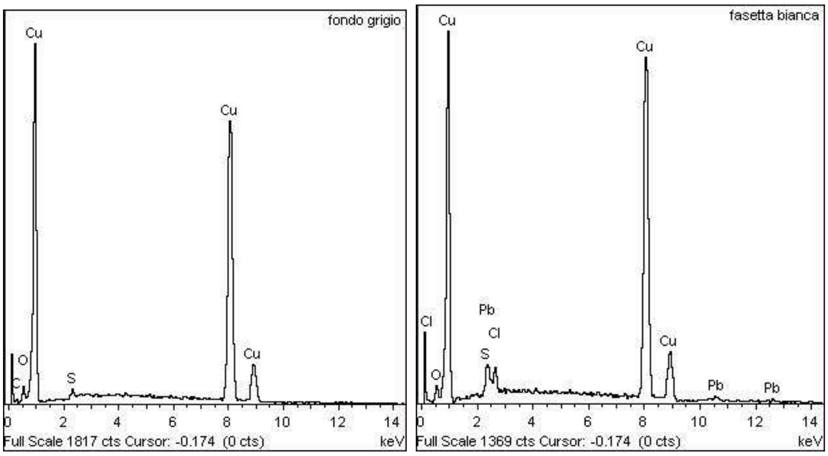

Figure 27: VL 1006, Area B, EDS spectrums.

GROUP 2: VL 1000

The artefact analyzed from group 2 was VL 1000, which had 4 areas selected for further studies. The XRF group this artefact belongs to shows copper, arsenic and lead. The areas analyzed show high quantities of copper, and traces of carbon, chlorite, arsenic, oxygen and in some cases lead. The C, Cl and O present in the areas analyzed are probably signals from the environment and the space between the detector and the artefact or signals from the soil still attached to the surface of the artefact; so we can affirm that VL 1000 contains copper, arsenic and lead, confirming the findings of the XRF.

Area A

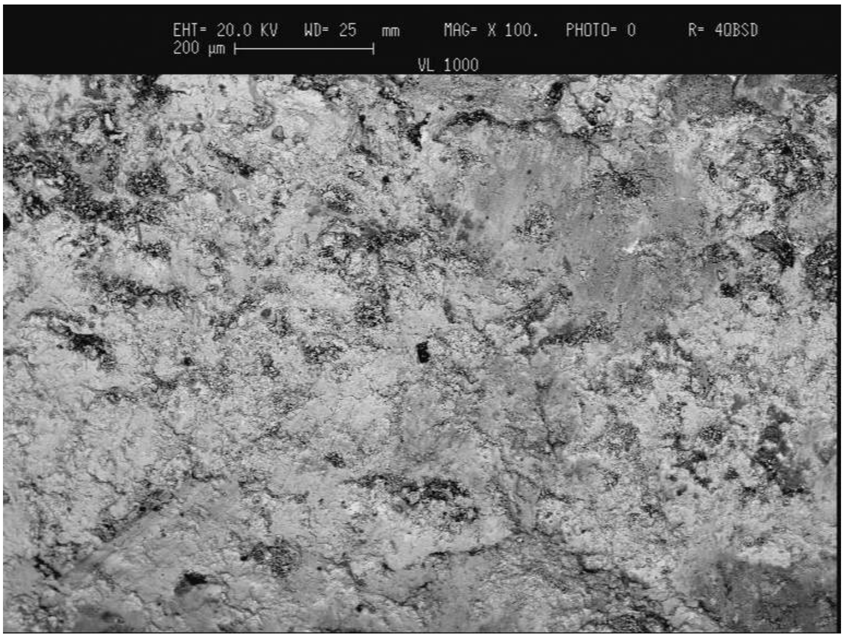

Figure 28: VL 1000, Area A, SEM image.

Area A showed presence of C, Cl, Cu, As, Pb and O. As mentioned before, the C, Cl and O are not to be taken into account as part of the alloy composition, but this is one of the areas that show traces of arsenic and lead, as well as high amounts of copper, which confirms the XRF groupings.

Spectrum	C	Cl	Cu	As	Pb	O
Area A	3.96%	0.56%	63.05%	3.55%	1.22%	27.65%

Table 21: Results of EDS in Area A of VL 1000.

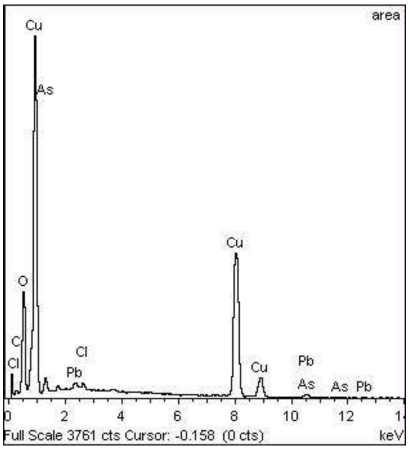

Figure 29: VL 1000, Area A, EDS spectrum.

Area B

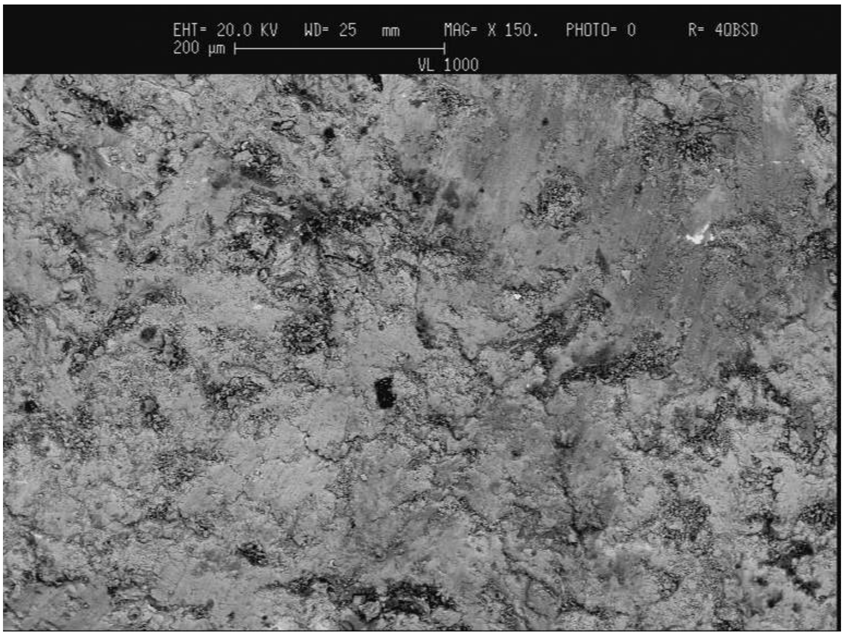

Figure 30: VL 1000, Area B, SEM image.

Area B shows presence of C, Cl, Cu, As and O. As with the previous area, C, Cl and O come from the space between the artefact and the detector and the soil still attached to the surface of the artefact. This area shows no presence of lead, which makes us consider the fact that the Pb presence is not homogeneous in the whole artefact.

Spectrum	C	Cl	Cu	As	O
Area B	4.70%	0.37%	63.36%	2.33%	29.23%

Table 22: Results of EDS in Area B of VL 1000.

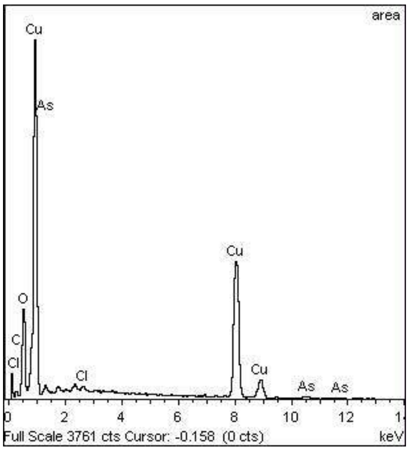

Figure 31: VL 1000, Area B, EDS spectrum.

Area C

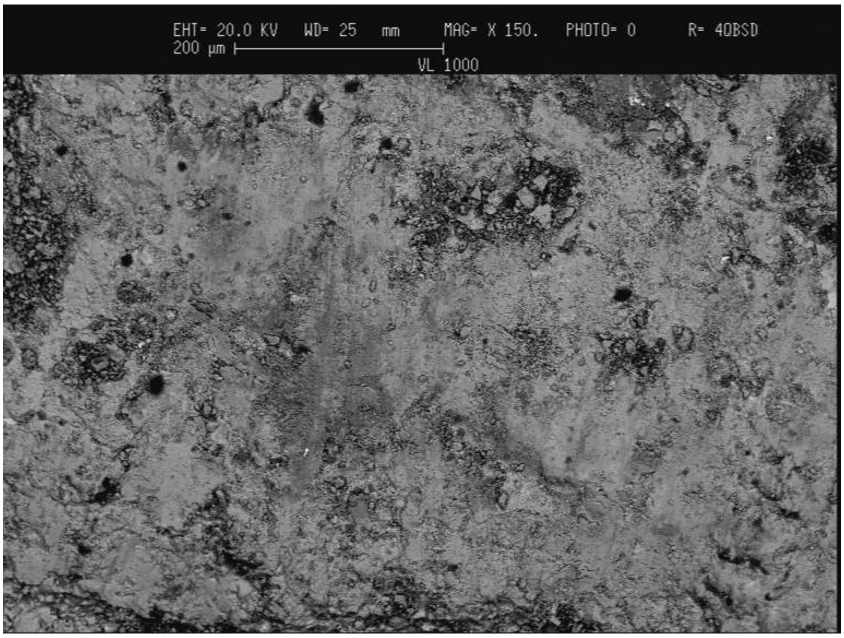

Figure 32: VL 1000, Area C, SEM image.

Area C shows C, Cl, Cu, As, Pb and O. This area shows both arsenic and lead in small quantities mixed in with the main metal, that is the copper. As with the two previous areas areas analyzed, the presence of C, Cl and O have a relationship with the environment the artefact was analyzed inside and the soil attached to the surface.

Spectrum	**C**	**Cl**	**Cu**	**As**	**Pb**	**O**
Area C	3.82%	0.63%	65.02%	2.39%	0.75%	27.38%

Table 23: Results of EDS in Area C of VL 1000.

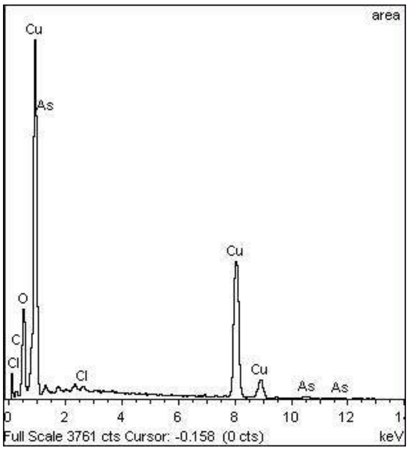

Figure 33: VL 1000, Area C, EDS spectrum.

Area D

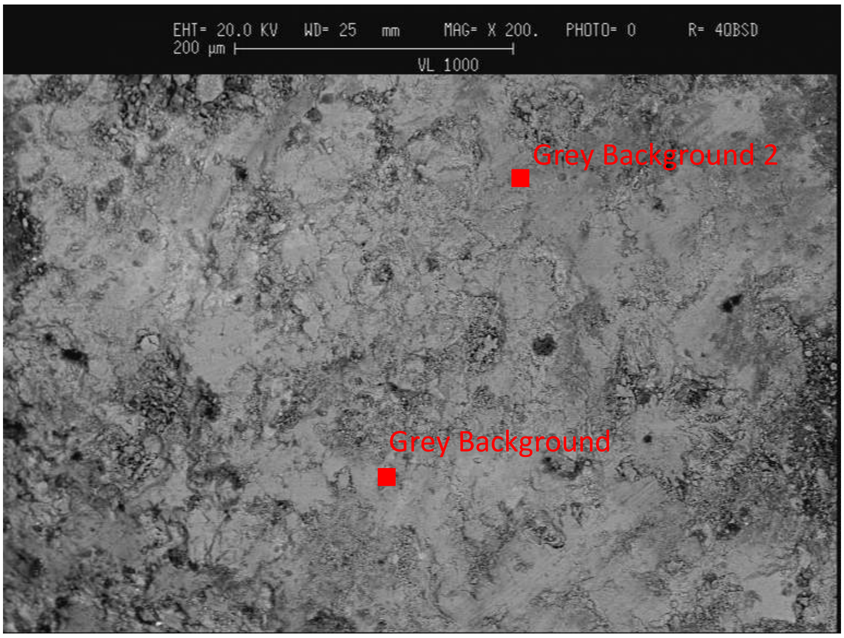

Figure 34: VL 1000, Area D, SEM image.

Area D was both analyzed as an area, and had two specific points analyzed. In all these analyses, the only metal detected was copper, with some presence of C, Cl and O, which is related to the analysis environment and the soil in the surface of the artefact.

Spectrum	**C**	**Cl**	**Cu**	**O**
Grey Background	3.88%	0.60%	68.06%	27.46%
Grey Background 2	2.31%	0.48%	72.73%	24.48%
Area D	3.09%	0.54%	70.39%	25.97%

Table 24: Results of EDS in Area D of VL 1000.

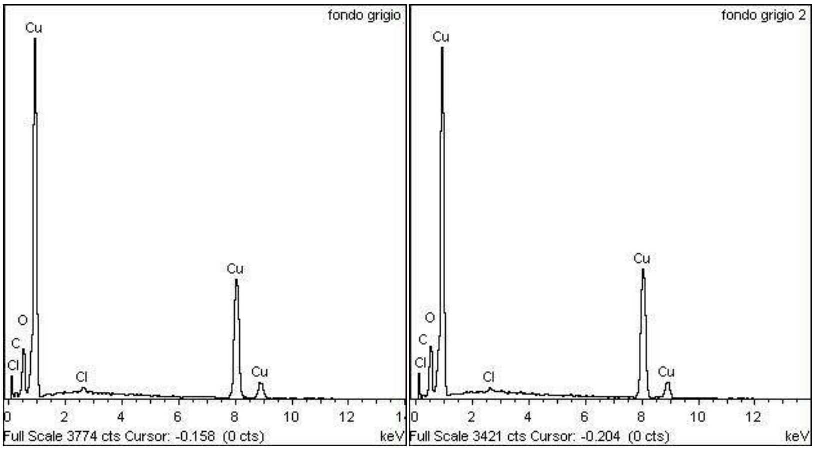

Figure 35: VL 1000, Area D, EDS spectrums.

GROUP 3: VL 3009

The artefact VL 3009 was chosen from group 3 to have two areas analyzed. From the XRF, this artefact showed copper, arsenic and traces of silver. The EDS shows presence of C, Fe, Si, P, Cl and O, which can be related to the environment and the soil; high quantities of Cu, as well as small quantities of As and Ag, as seen in the XRF; and even Au, which was not detected in the previous analysis.

Area A

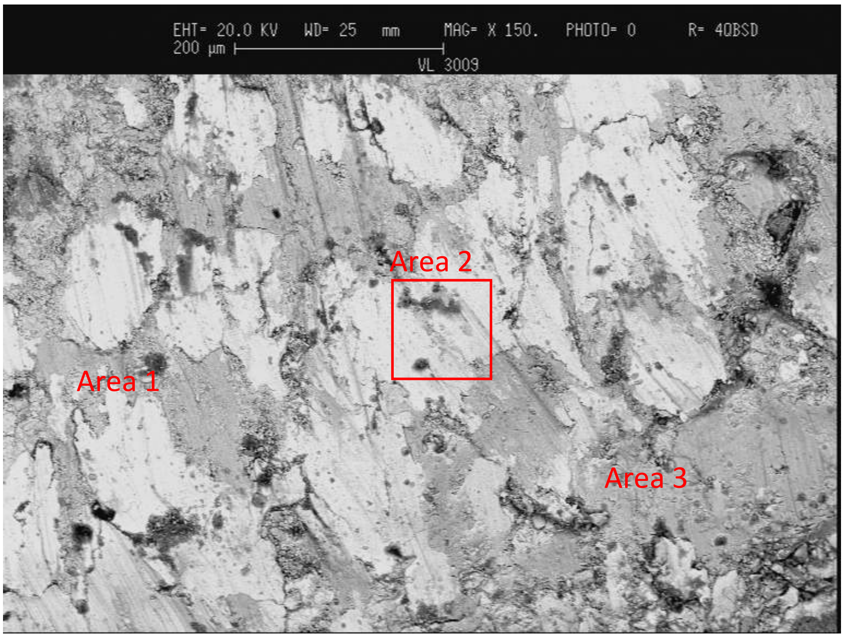

Figure 36: VL 3009, Area A, SEM image.

Area A was analyzed as a whole, and also had 3 smaller areas analyzed. Area A and all the smaller analyzed areas show presence of C, Si, P, Cl, Cu and O. The C, Si, P, Cl and O are signals related to the soil the artefact was buried in, the environment inside the equipment and some trace elements in the alloy.

Spectrum	C	Si	P	Cl	Cu	O
Area 1	3.28	0.44	0.27	7.20	63.29	25.52
Area 2	3.67	0.43	0.37	7.29	61.91	26.33
Area 3	4.07	0.76	0.51	7.59	59.68	27.39
Area A	3.67	0.54	0.38	7.36	61.63	26.41

Table 25: Results of EDS in Area A of VL 3009.

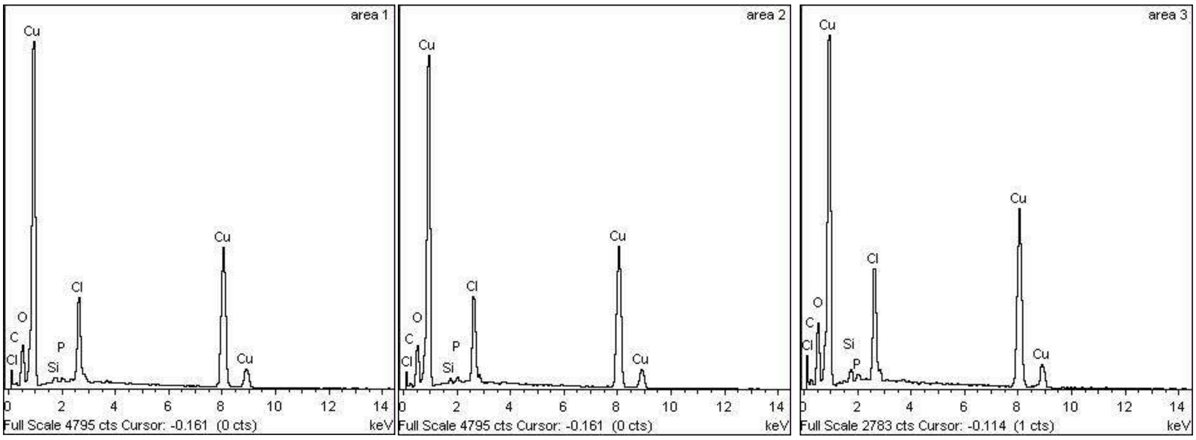

Figure 37: VL 3009, Area A, EDS spectrums.

Area B

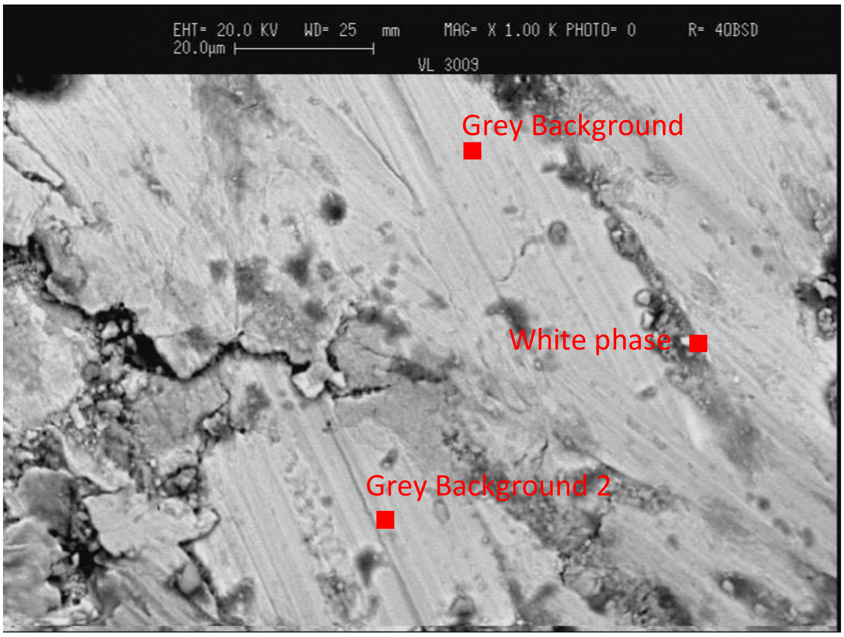

Figure 38: VL 3009, Area B, SEM image.

Area B, on the other hand, had 3 specific points analyzed, two from the background, and one crystallized point. It was in these analyses that the presence of arsenic, gold and silver was detected. The two grey backgrounds show copper and arsenic; while the white phase shows copper, silver and gold. The quantities of both silver and gold are quite small compared to the copper, and can only be found the small white crystallized points, so their presence can be considered unintentional.

Spectrum	C	Cl	Fe	Cu	As	Ag	Au	O
White phase	3.23%	0.81%	0.99%	28.58%		27.86%	18.15%	20.37%
Grey Background	2.06%			73.07%	0.76%			24.12%
Grey Background 2	1.73%			74.01%	0.75%			23.50%

Table 26: Results of EDS in Area B of VL 3009.

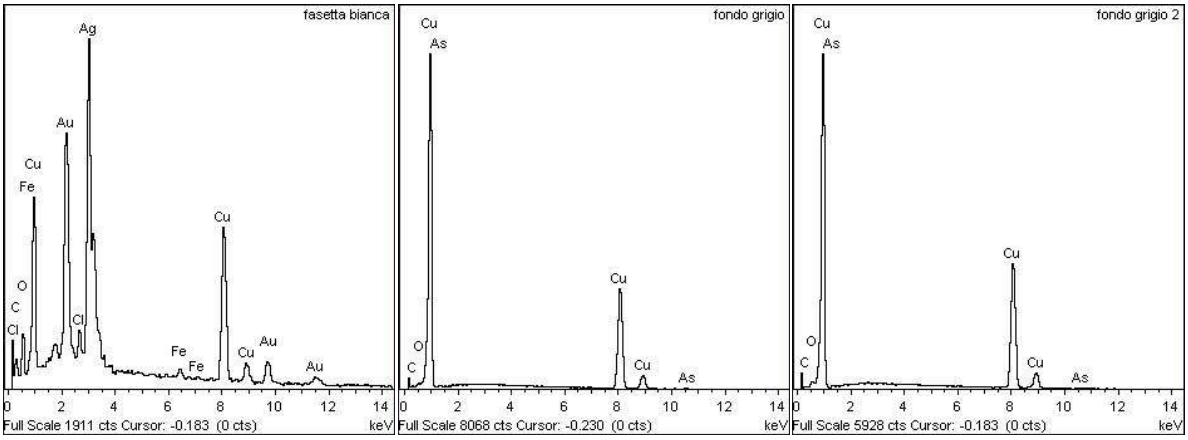

Figure 39: VL 3009, Area B, EDS spectrums.

GROUP 4: VL 3003

Artefact VL 3003 belongs to XRF group 4, which contains copper and high quantities of silver was analyzed through SEM-EDS in two areas. These analyses determined presence of C, Al, Si, Cl and O; as well as Cu, Ag and Au. As mentioned before, C, Cl and O are most likely related to the soil in the surface, and the equipment environment; Al and Si might be trace elements found in connection to the geological sources.

Area A

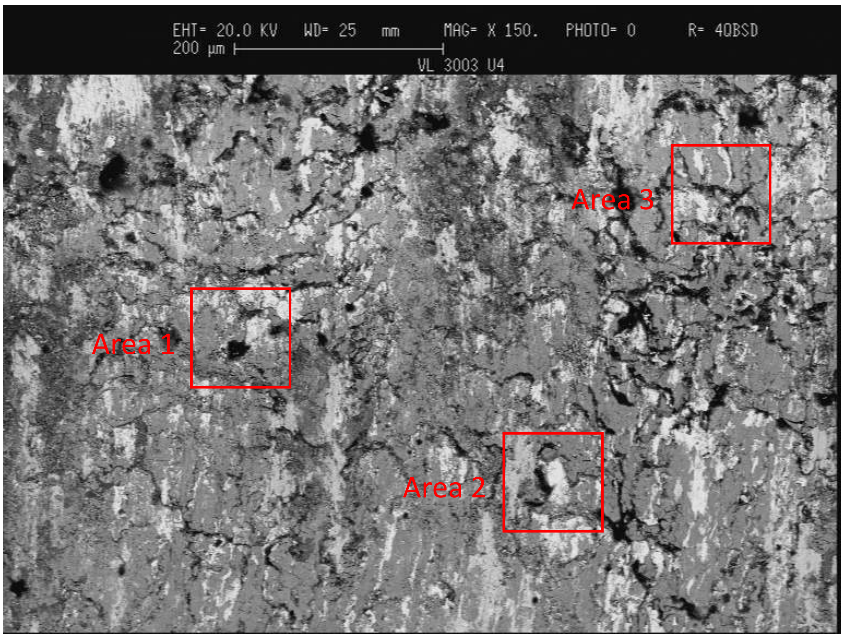

Figure 40: VL 3003, Area A, SEM image.

Area A, has three smaller areas analyzed, each of which showed presence of C, Al, Si, Cl, Cu, Ag and O. Silver was found in all of the EDS results, which suggest that its presence was intentional and make this a Cu-Ag alloy. All three areas show above 16% presence of silver and high quantities of copper. The Al, Si and C are trace elements that relate to the geological sources the silver or the copper came from.

Spectrum	**C**	**Al**	**Si**	**Cl**	**Cu**	**Ag**	**O**
Area 1	8.08%	0.19%	0.40%	3.27%	36.82%	18.44%	32.80%
Area 2	8.01%	0.29%	0.44%	4.03%	38.29%	16.03%	32.91%
Area 3	7.14%	1.48%	1.86%	6.35%	33.46%	17.51%	32.19%
Area A	7.74%	0.65%	0.90%	4.55%	36.19%	17.33%	32.64%

Table 27: Results of EDS in Area A of VL 3003.

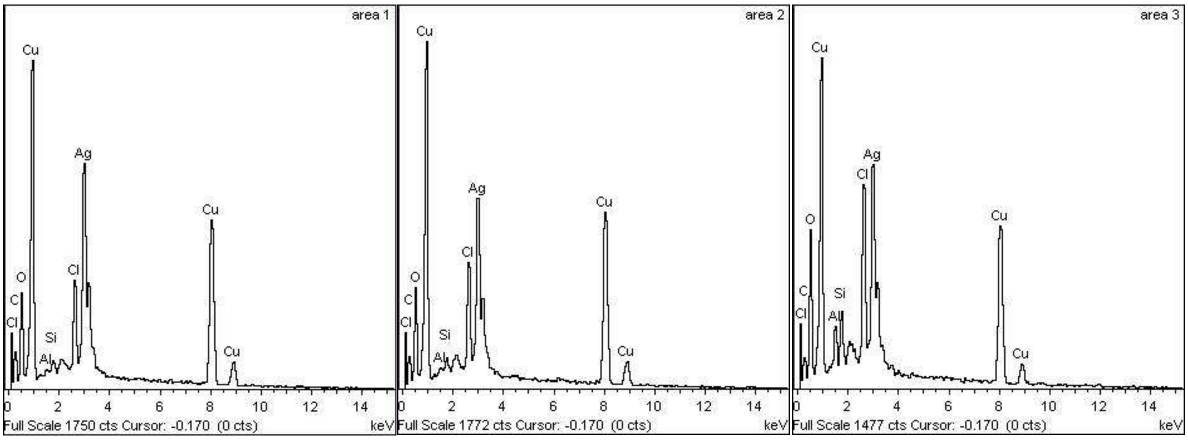

Figure 41: VL 3003, Area A, EDS spectrums.

Area B

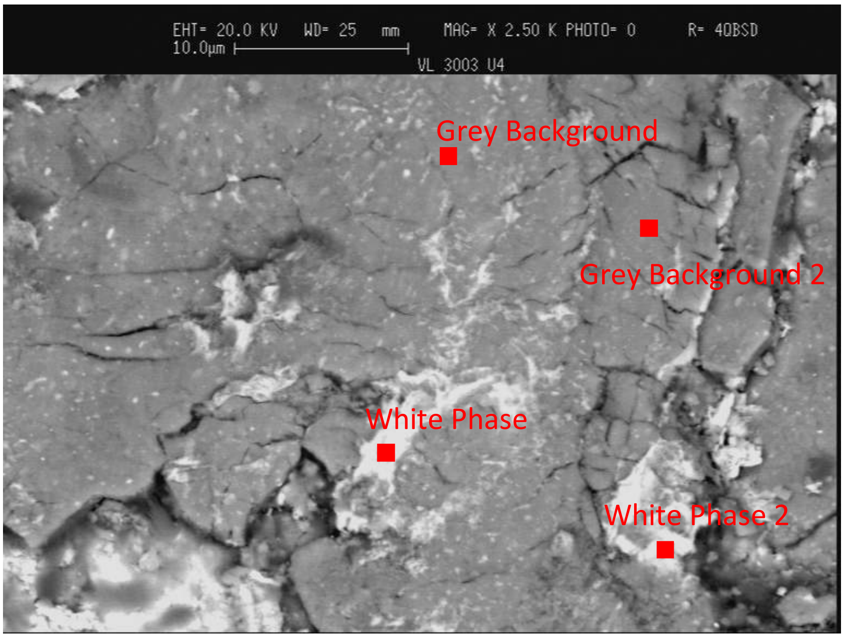

Figure 42: VL 3003, Area B, SEM image.

Area B had 4 points analyzed, two from the background, and two from white areas or phases. The EDS showed C, Cl, Cu, Ag, Au and O in the analyzed artefact. The white phases show higher concentration of silver than copper, and small traces of less than 1% of gold; the grey background areas, on the other hand show high quantities of copper, less than 8% of silver and up to 1.5% of gold. The area, overall contains 43.24% of copper, 32.62% of silver, both intentional in the alloy, and only 1.08% of gold, which probably wasn't intentional. The alloy composition is a silver based tumbaga.

Spectrum	**C**	**Cl**	**Cu**	**Ag**	**Au**	**O**
White phase	1.74%	1.64%	9.07%	74.53%	0.51%	12.50%
Grey Background	3.17%	0.51%	62.61%	7.44%	1.35%	24.93%
Grey Background 2	2.42%	0.23%	66.13%	6.01%	1.50%	23.72%
White phase 2	2.27%	0.96%	35.14%	42.51%	0.95%	18.17%
Area B	2.40%	0.84%	43.24%	32.62%	1.08%	19.83%

Table 28: Results of EDS in Area B of VL 3003.

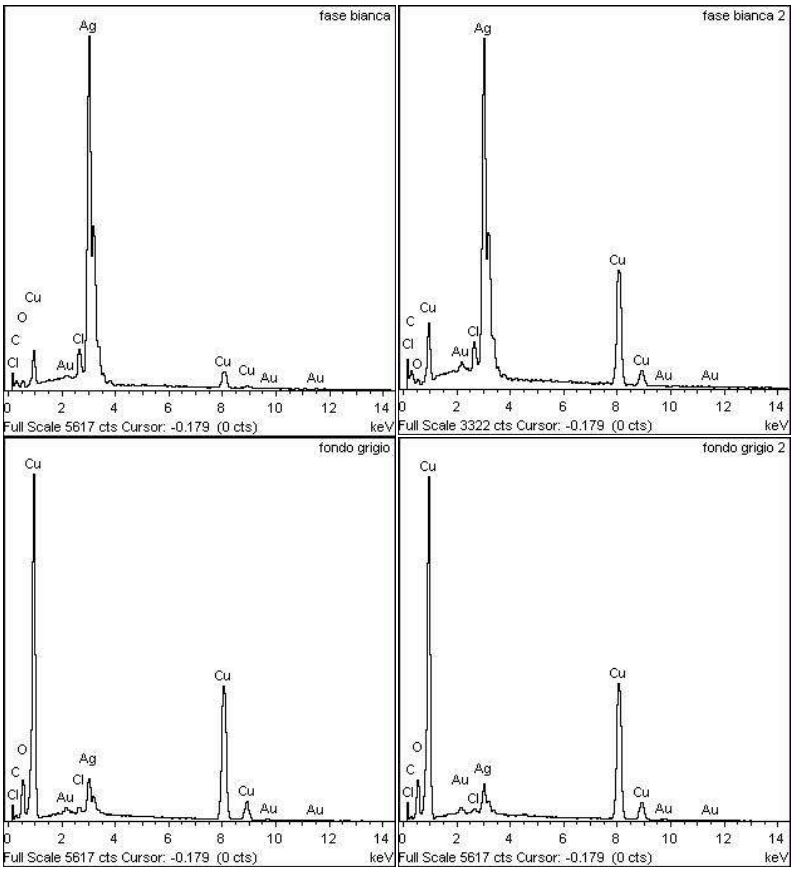

Figure 43: VL 3003, Area B, EDS spectrums.

GROUP 5: VL 036.1

The artefact VL 036.1 is the only artefact identified as part of group 5, which according to the XRF is a copper-lead alloy. This artefact had just one area analyzed and showed presence of C, Si, P, Cl, Ca, Cu and O. The EDS did not show presence of lead, which signifies that that element was not intentionally placed as part of the alloy, or that it was only present in some parts of the surface of the artefact.

Area A

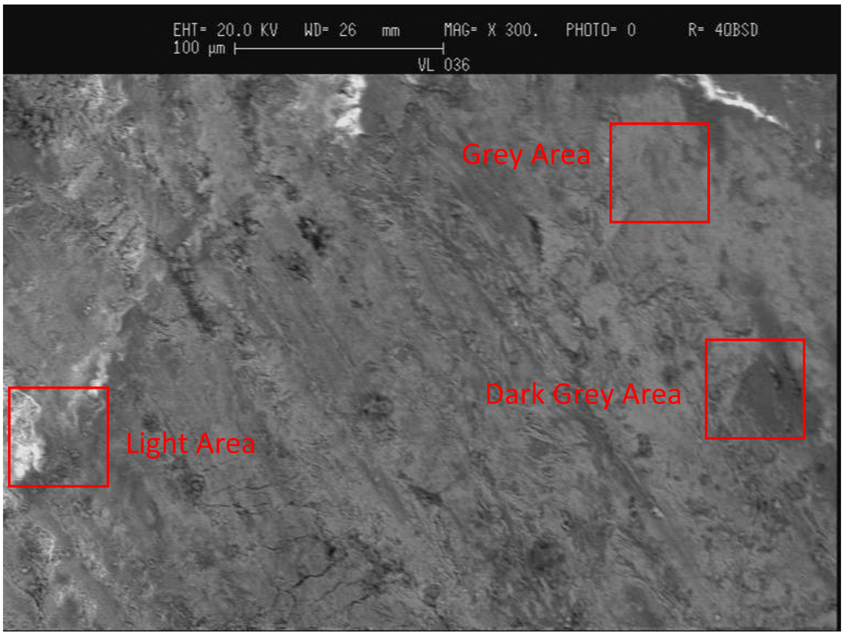

Figure 44: VL 036.1, Area A, SEM image.

The area had three smaller areas analyzed: a dark grey, a grey and a light area. Both grey areas show presence of C, Si, P, Cl, Ca, Cu and O; while the light area does not show Si. The only relevant element shown by the EDS is the copper.

Spectrum	**C**	**Si**	**P**	**Cl**	**Ca**	**Cu**	**O**
Dark Grey Area	10.96%	0.52%	7.40%	2.33%	1.80%	29.50%	47.49%
Grey Area	7.87%	0.59%	4.44%	1.75%	0.68%	45.55%	39.12%
Light Area	12.62%		2.85%	2.48%	1.02%	34.62%	46.42%
Area A	13.71%	0.78%	6.25%	2.82%	2.13%	22.34%	51.97%

Table 29: Results of EDS in Area A of VL 036.1.

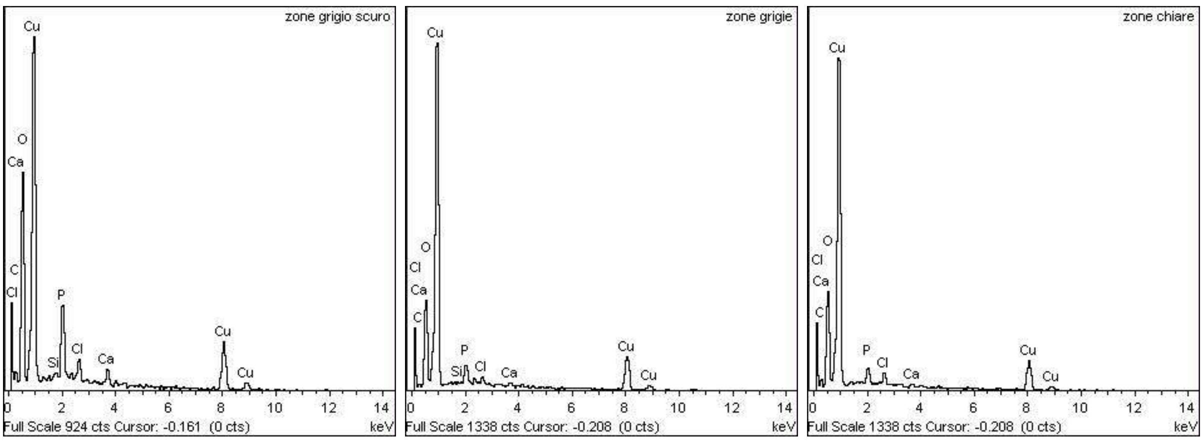

Figure 45: VL 036.1, Area A, EDS spectrums.

GROUP 6: VL 2000.1

Artefact VL 2000.1, the only artefact from group 6, which based on the XRF analysis is a pure copper artefact. One area of this hook was analyzed, and several other elements were discovered including not only Cu, but C, Cl, Al, Si, Ca, P, Ag and O.

Area A

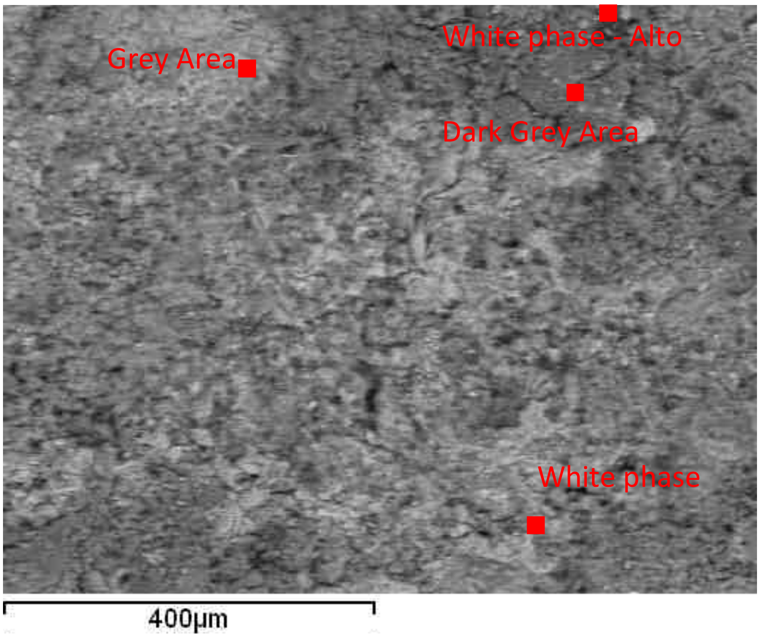

Figure 46: VL 2000.1, Area A, SEM image.

While analysis Area A, 4 points were selected for EDS analysis. These analyses showed presence of copper in all areas and small traces of silver in the lighter colored points. Most of the other elements are related to soil attached to the artefact, the equipment environment and some other trace elements related to the source of the raw material.

Spectrum	**C**	**Cl**	**Al**	**Si**	**Ca**	**P**	**Cu**	**Ag**	**O**
White phase	8.5%	0.7%	1.2%	3.2%	1.01%	1.23%	38.1%	1.65%	40.6%
White phase - alto	19.4%	0.6%	0.9%	3.08%	5.57%	0.71%	4.5%	0.9%	62.%
Grey Area	9.7%	0.3%	1.02%	11.5%	0.64%		27.4%		47.8%
Dark Grey Area	12.1%	0.4%	5.51%	13.1%	0.63%	1.08%	6.9%		56.5%

Table 30: Results of EDS in Area A of VL 2000.1.

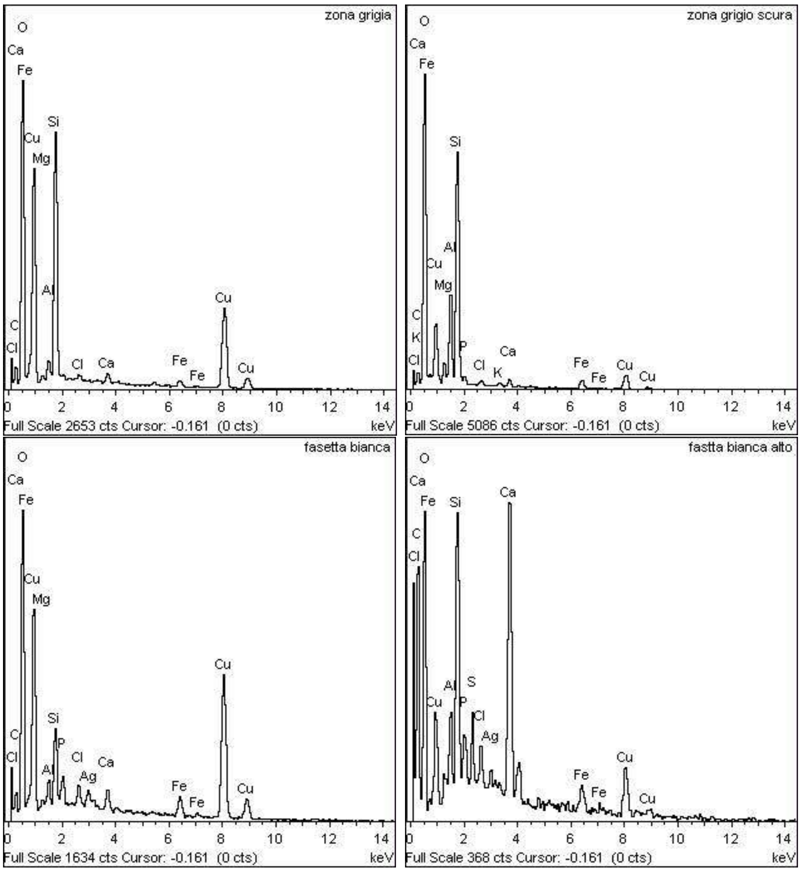

Figure 47: VL 2000.1, Area A, EDS spectrums.

ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE

The Environmental Scanning Electron Microscope was used to analyze 5 animal hair samples and 3 archaeological samples.

CAMELID HAIR SAMPLES

Camelids are herd mammals that were domesticated in the Andes and were of primary importance to pre-Hispanic economy, social and ritual life from modern day northern Ecuador, to the Patagonia (Mengoni 2008). The four main species of camelids found in the area include two domesticated: alpaca and llama; and two wild ones: guanaco and vicuña. For the purpose of this research the Environmental SEM was used to analyze camelid wool samples obtained from Ecuadorian *Lama Glama* and *Vicugna pacos*, more commonly known as Llama and Alpaca. These two animals are the ones with the longest domestication history in the Central Andes, almost 6000 years ago in the case of the alpaca, and between 4600 and 3000 years ago in the case of the llama (Mengoni 2008).

Animal sample	Hair color
Alpaca	White
Llama 1	Grey
Llama 2	Black
Llama 3	White
Llama 4	Brown

Table 31: List of animal hairs analyzed.

Alpaca

Alpacas are small and light domesticated camelids, probably descendant from wild vicuñas, that are traditionally found from the Titicaca basin to northern coastal Peru, but have been also been known to live in central Ecuador (Mengoni 2008). Alpacas posses a fine and silky wool, which varies in fineness, from 9 to 88um, based on the body part it comes from, alpaca wool has been used for wool as it takes dye better and due to its natural elasticity (Lange et al. 1987; Wheeler et al. 1995). It is important to note that alpaca colors range from white to black, and include various shades of brown and gray; but they usually have only one color of wool all over their bodies.

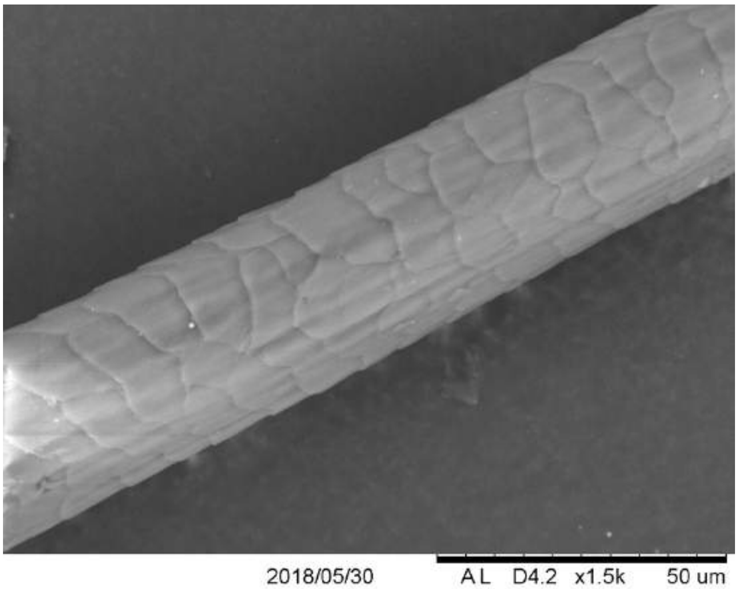

Figure 48: Alpaca hair under SEM.

By analyzing a sample of alpaca hair under SEM, this research attempted to use them as reference for identifying the textiles found attached to the metallic artefacts analyzed beforehand. The alpaca hair, shows, as expected (Tridico 2009), overlapping cuticular scales pointing to the tip of the fiber, on the outermost surface of the fiber in a smooth distant broad petal scale pattern. This hair's width is approximately 28um and white in color.

Llama

Llamas are bigger domesticated camelids, probably descendant from wild guanacos and are commonly found from northern Ecuador to central Chile and northwest Argetina; it is a highly adaptable animal and has been historically used for food, wide, beast of burden and fiber (Mengoni 2008). Llama wool is coarser and stiffer than that of alpaca and can have a width from 8 to 144um; and as in the alpaca, the fiber diameter depends on the area of the body the hair comes from (Wheeler et al. 1995). Llamas colors range from white to black, including several brown and grey shades; but it is important to mention that they sometimes show more than one wool color at once. For this research, four different llama hairs were picked, each of a different color.

Grey llama hair

This llama hair, grey in color was a width of 49.8um and a barely visible crenate distant regular wave scale pattern (Tridico 2009).

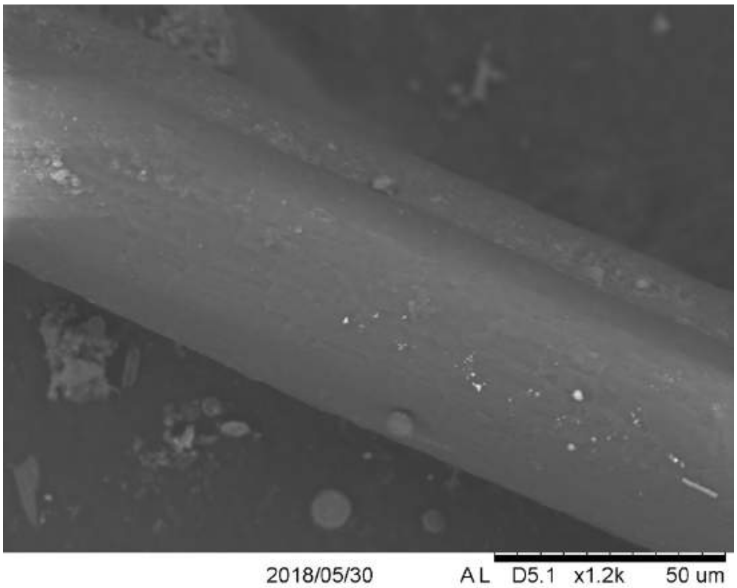

Figure 49: Grey llama hair under SEM.

Black llama hair

The analyzed black llama hair has a width between 46.3 and 54.5um, and presents a barely visible distant broad petal scale pattern. The SEM image also shows some damages to the hair, in the shape of long straight cuts along the length of the fiber.

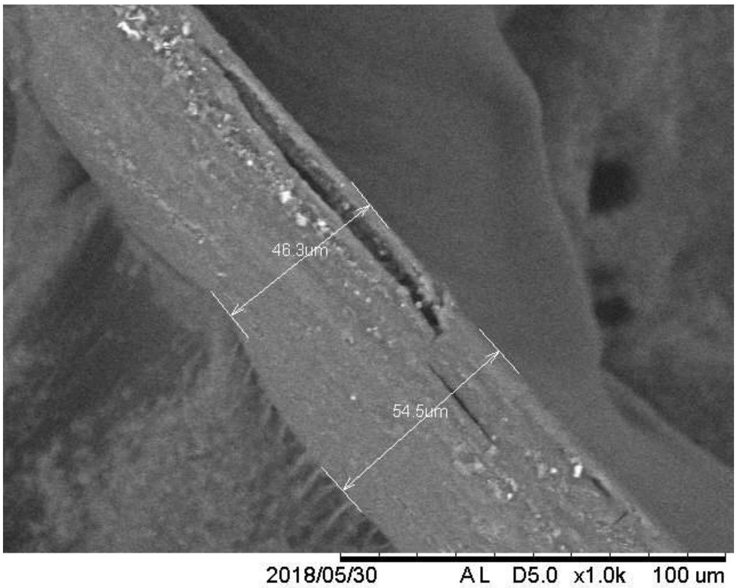

Figure 50: Black llama hair under SEM with width.

White llama hair

The white llama hair analyzed shows a width of 98.5um and a crenate, near and regular scale pattern.

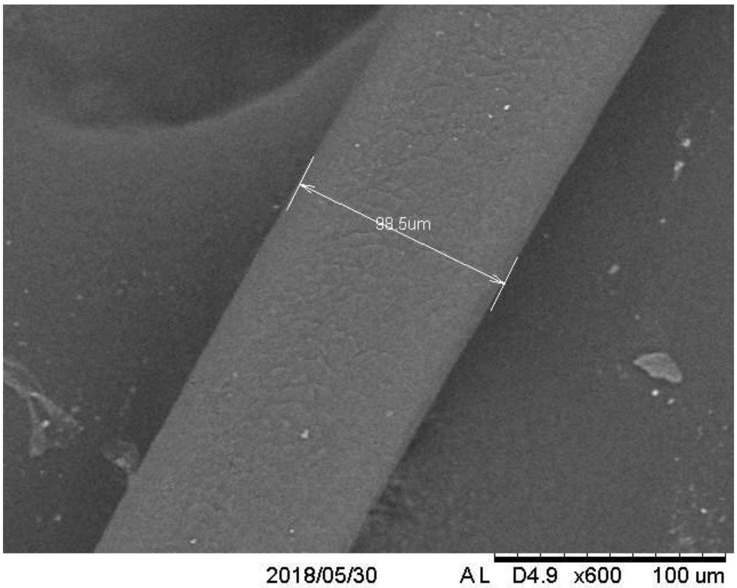

Figure 51: White llama hair under SEM with width.

Brown llama hair

The brown llama hair shows a width of 42.3um and a crenate, near, regular wave scale pattern.

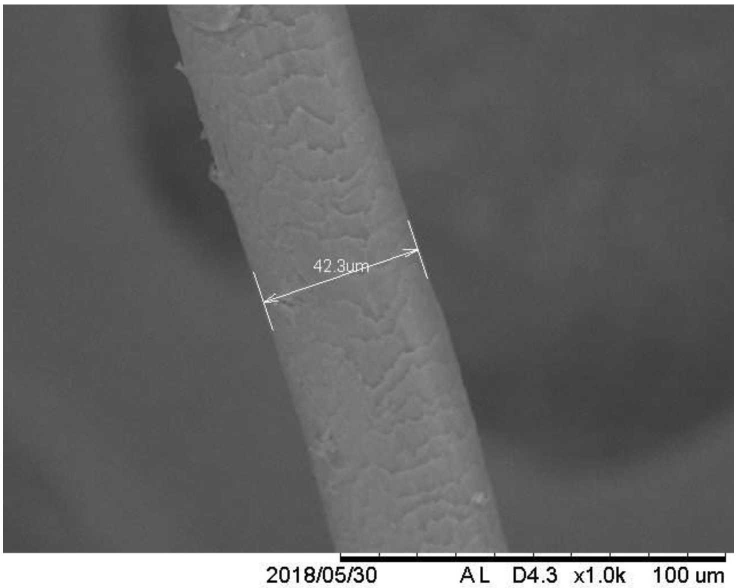

Figure 52: Brown llama hair under SEM with width.

ARTEFACTS ANALYZED

During the first macroscopic analysis preformed on the artefacts, 5 of them were identified as being attached to fibers or textiles. Based on the microscopic analysis, 3 groups were created: threads, braiding pattern textile, and balanced plain weave textile.

ID code	Artefact	Group
VL 010	4 Necklace beads with fiber	Thread
VL 036.1	Bracelet fragment	Float weave or braiding
VL 036.2	Pectoral fragment	Balanced plain weave

Table 32: Artefacts analyzed through Environmental SEM

VL 010

The fibers found in relation to artefact VL 010, were categorized are threads, as they are inside beads. The fibers are partially mineralized, which most likely caused deterioration on the fiber's surface. It seems like the thread was made by coiling together several fibers together and twisting them into shape.

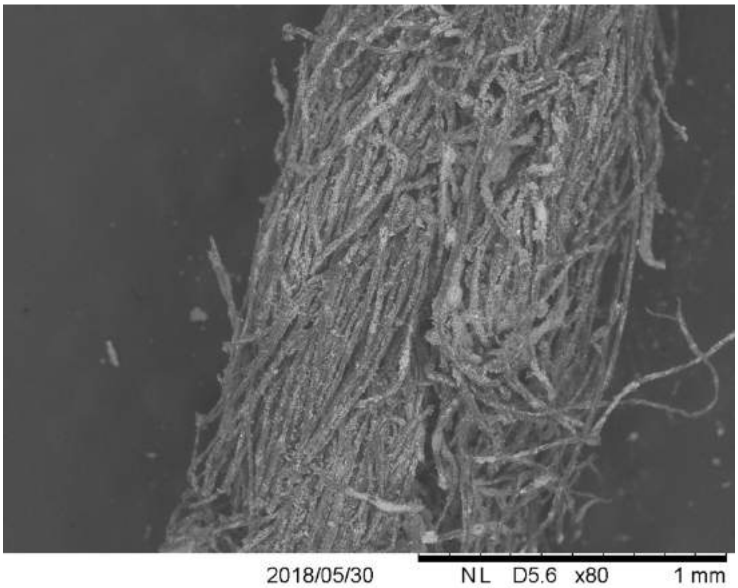

Figure 53: Fragment of VL 010 threat under SEM.

A single fiber was selected and analyzed with the SEM, which showed the width of each fiber at approximately 20.9um. There is evidence of scales, but the pattern is hard to identify, but it seems like a near broad petal or regular wave pattern. It is evident, due to the scales that this fiber is of animal origin, unfortunately, due to the mineralization and degradation process it's impossible to determine if it comes from a llama or an alpaca based in surface analysis, but it's width might suggest it is alpaca hair.

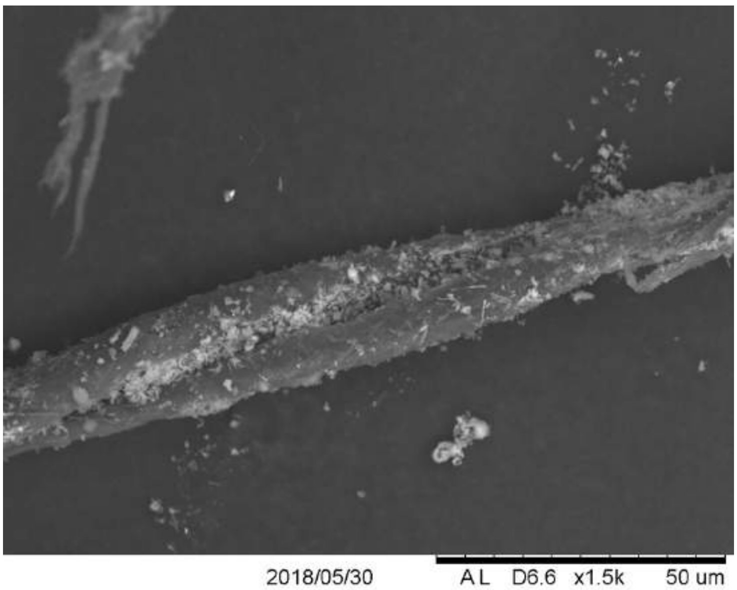

Figure 54: Isolated VL 010 fiber under SEM.

VL 036.1

The textile attached to artefact VL 036.1 seems to have been woven with a braiding or float weave pattern. The SEM was used to obtain images of the weaving pattern, coiling and of a single fiber. The fibers are partially mineralized, decomposed and hold some remains of the soil they where buried in.

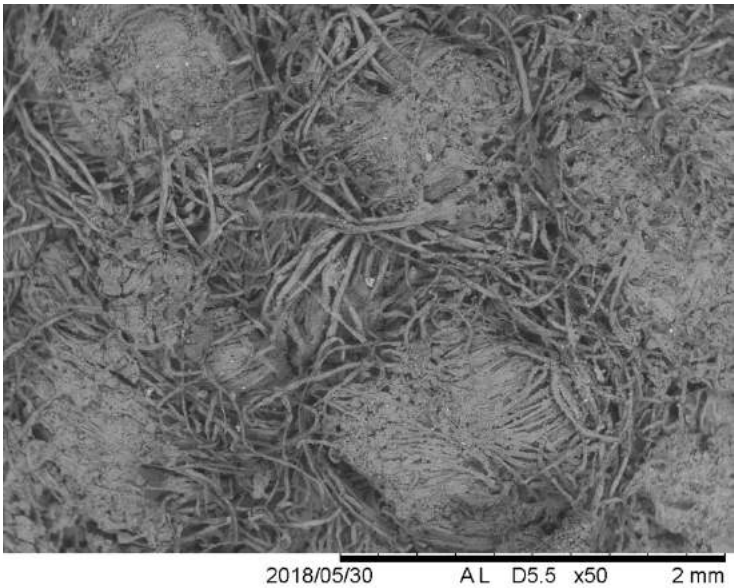

Figure 55: VL 036.1 textile pattern under SEM.

The images obtained through SEM show how the fibers were twisted and woven into shape, in a specific pattern, with fibers being places on top of each other in a braid-like pattern.

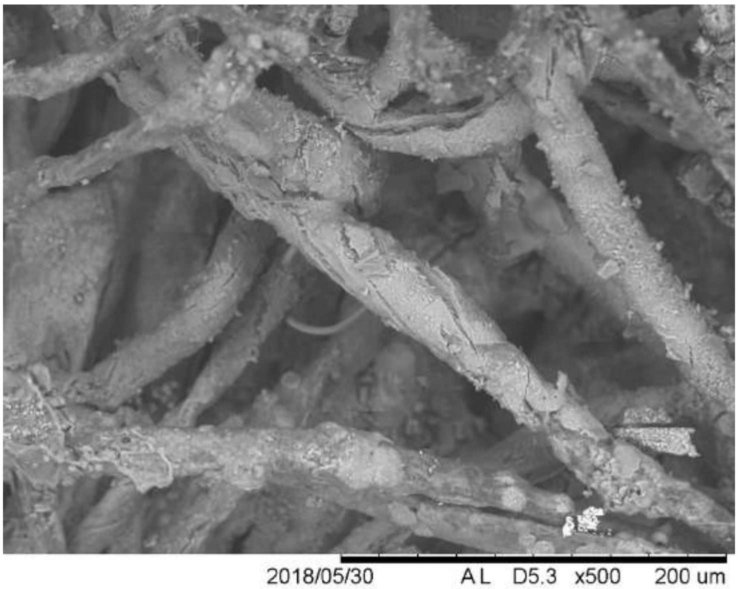

Figure 56: VL 036.1 interwoven fibers under SEM.

When analyzing the image of the isolated fiber or hair, it was determined that its width was approximately 38um. The scale pattern was difficult to determine, due to the mineralization and soil attached to the fiber, as well as the natural degradation process, but it seems to be a crenate, distant broad petal scale pattern. Additionally, the fiber shows evidence of rupture in the shape of long straight cuts, which might have happened during coiling. Based on these characteristics, this fiber could be identified as a finer llama hair or an alpaca hair.

Figure 57: Isolated VL 036.1 fiber under SEM.

VL 036.2

Artefact VL 036.2, which had a piece of textile attached to it was analyzed for this research. During this analysis it was determined that the fibers were coiled together and then woven in a balanced plain weave pattern. The textiles and therefore the fibers show evidence of mineralization, degradation and soil.

Figure 58: Coiled VL 036.2 fibers under SEM.

The SEM images allowed to determine the width of this fiber was 22um approximately, and that it lacked the presence of scales.

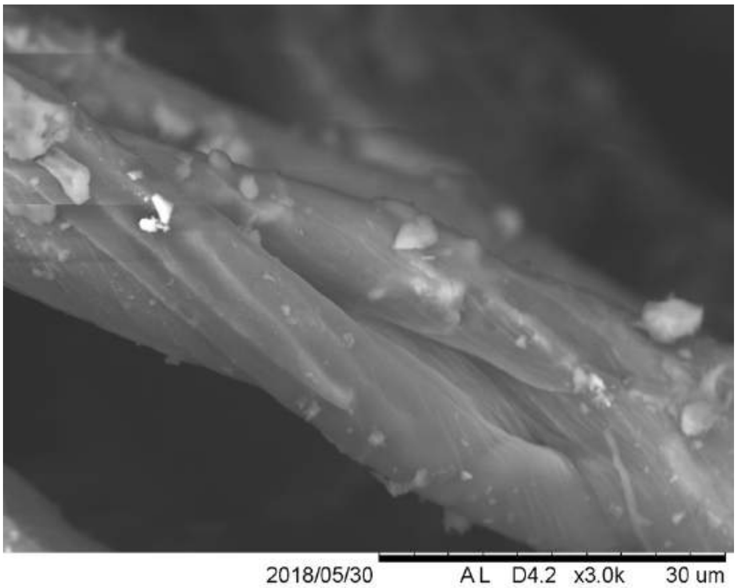

Figure 59: Isolated VL 036.2 fiber under SEM.

This observation lead to the use the EDS of the equipment, which showed presence of elements related to vegetable fibers like C, K, O, P, Ca and Cl; as well as Cu and Al, which could be related to the mineralization process. Animal fibers contain N, which wasn't present in this analysis, and lead to the conclusion that this fiber was of vegetable origin.

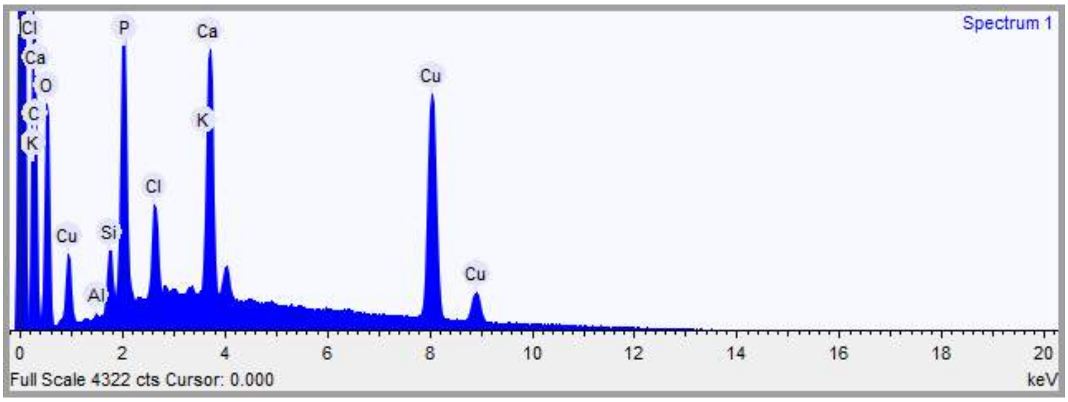

Figure 60: VL 036.2 EDS spectrum.

Finally, the SEM images of the textile attached to artefact VL 036.2 was compared to SEM images obtained from other publications (Gordon 2009; Kan et al. 2012), and was identified as cotton, due to the way the fiber is twisted, the surface morphology with uniform, long lines that go all the way from beginning to end of the fiber in a longitudinal manner and their approximate width of 25um.

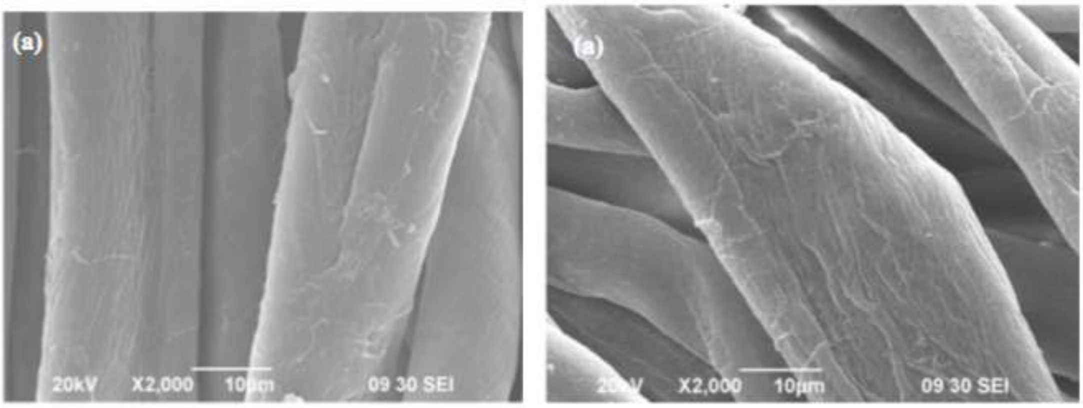

Figure 61: Images of cotton under SEM. Fibers pre-treated for analysis with 10% BTCA (left) and by plasma (right). (Kan et al. 2012).

CHAPTER 8: DISCUSSION

Milagro-Quevedo metal artefacts can be classified in different ways, based on shape, use, color, raw material, manufacturing technique and even stage of manufacture. The preformed analyses allowed us to determine the existence of four alloys, four metal colors, three manufacturing techniques, two types of artefacts recovered and it lead to the determination of types of fibers used by the Milagro-Quevedo.

ARTEFACT CLASS AND METAL COLOR

Milagro-Quevedo metallurgical artefacts have been previously analyzed and five classes of artefacts were determined by Sutliff (1989), mainly based on use and stage of finishing: ornaments, implements, semi-elaborate material, unidentified objects and unknown artefacts. The 22 artefacts analyzed for this research came from a burial mound, so they can all be considered grave goods, but can still be classified into two of the five different classes suggested by Sutliff (1989): implements and ornaments.

IMPLEMENTS

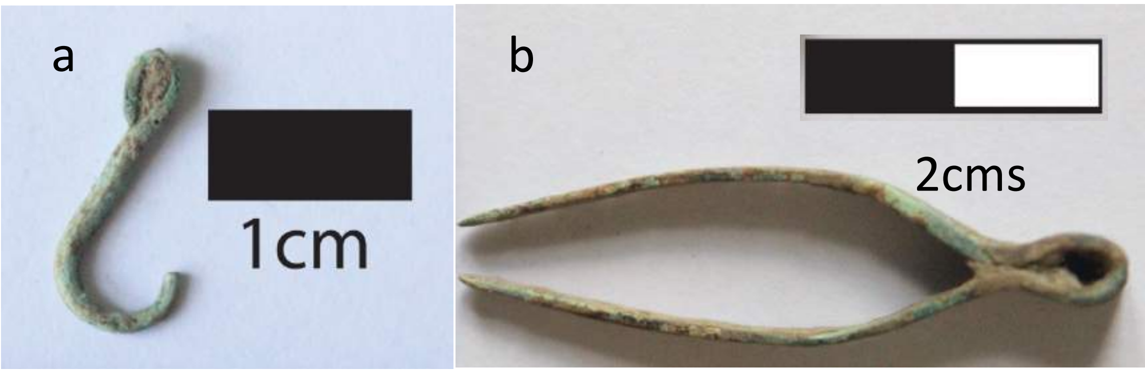

Figure 62: Examples of implements. a-VL2000, b-VL013.

The artefacts considered implements are those that were created for a practical, functional purpose, even if they were left as grave goods, and became ornamental as a consequence of their final use. Implements in this case include needles, tweezers, hooks and axes. All but one of these artefacts are bronze in color, and are alloy mixtures of copper, arsenic and lead. The color and alloy mixture show that these artefacts had a more utilitarian purpose, as there is no additional process or metal added to change the final color of the artefact. The only artefact that does not comply to the color and alloy group is artefact VL 2000.1, which according to the SEM-EDS contains traces of silver, and due to corrosion has a surface color that was impossible to determine.

ID code	Artefact	Color	Alloy mixture
VL 013	Tweezers	Bronze	Cu+As
VL 114	Tweezers fragment	Bronze	Cu+As
VL 3021	Needle fragment	Bronze	Cu+As+Pb
VL 3027	2 axe fragments	Bronze	Cu+As
VL 2000.1	Hook	Undetermined	Cu+Ag

Table 33: Artefacts considered implements, color and alloy mixture.

There seems to be a correlation between metal color and use of artefact that can be traced to both the Manteño-Huancavilca (Romero-Bastidas et al. 2017), and several north Peruvian societies (Lechtman 1985, 2014). The bronze color and original use of these artefacts complements Lechtman (1985, 1991) and Sutliff (1989, 1998) ideas about the use of copper for utilitarian and lower class ornaments.

ORNAMENTS

Figure 63: Examples of ornaments. a-Vl1006, b-VL3009.

The artefacts considered ornaments are those that, as expected by the category name were used as ornamentation in any part of the body of the wearer. All these artefacts had ornamentation as their both initial and final purpose. Ornaments include ear and nose rings, bells, necklace beads, bracelets and breastplates. The colors of the analyzed artefacts have a wider variety of colors and shades, as well as a wide variety alloy mixtures.

ID code	Artefact	Color	Alloy mixture
VL 009	Ring	Bronze	Cu+As+Ag
VL 1000	Ring	Bronze	Cu+As+Pb
VL 1017	Rings fragment	Bronze	Cu+As
VL 1033	Nose ring	Bronze	Cu+As
VL 106	Beads with fiber	Golden Bronze	Cu+As+Ag
VL 1023	Bell	Golden Bronze	Cu+As+Pb
VL 1051	Ring fragment	Golden Bronze	Cu+As+Ag
VL 2000.2	Undetermined shape	Golden Bronze	Cu+As+Ag
VL 2005.2	Bell	Golden Bronze	Cu+As
VL 3009	Ring and ring fragment	Golden Bronze	Cu+As+Ag+Au
VL 010	2 bead with fiber	Dark Bronze	Cu+As+Ag
VL 036.1	Bracelet fragment	Dark Bronze	Cu+Pb
VL 036.3	Not identified fragment	Dark Bronze	Cu+As+Ag

VL 1006	Nose ring	Dark Bronze	Cu+As
VL 2005.1	Ring	Dark Bronze	Cu+As
VL 3003	Bead	Silver	Cu+As+Ag+Au
VL 036.2	Pectoral fragment	Undetermined	Cu+As+Ag

Table 34: Artefacts considered ornaments, color and alloy mixture.

In the case of the ornaments, there is no clear correlation between color and alloy mixture, as there are artefacts that look bronze, golden bronze, dark bronze and silver, as well as different mixtures of copper, arsenic, lead, silver and gold. The final color and alloy mixture might have to do with the wearer of the artefact and their importance inside the society (Lechtman 1985). As all of these were grave goods, their purpose was to display wealth and hierarchy in death, so the colors might relate to a significance they were attempting to convey. It is usually considered that pure copper, copper-arsenic and copper-arsenic-lead, and their bronze and darker bronze colors were used for simpler ornaments meant for lower hierarchy members of the group, while the golden colors and alloy mixtures with silver and gold were meant for more elaborate pieces and higher hierarchy members (Sutliff 1989, 1998; Lechtman 1985).

MANUFACTURING TECHNIQUES

The Milagro-Quevedo people manufactured their metallic artefacts in a previously studied and established manner, which starts after the arrival of the raw material to the site, as there is no evidence of mines in the area. In all manufacturing techniques, the process starts with the ingot, goes on to creating a preform, shaping it and finally doing finishing touches and polishing.

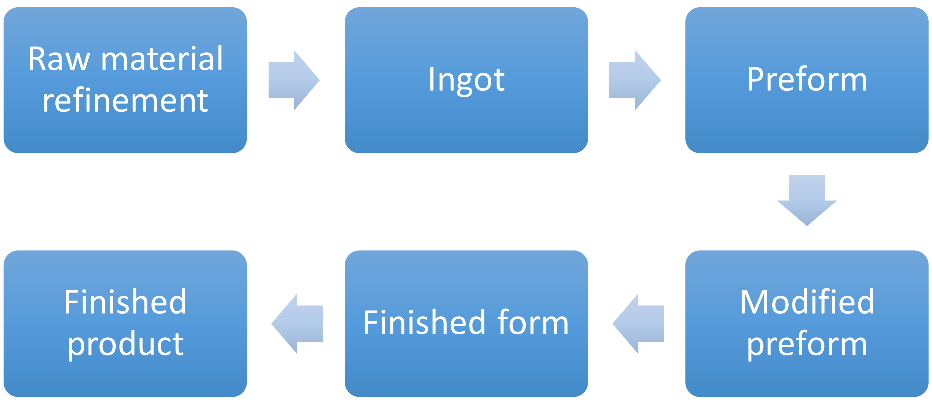

Figure 64: Basic manufacturing process. (Based on Sutliff 1989).

The artefacts were classified based on either surface color process or the shaping methods which include preform creation, modification and finishing, and three groups were created: wire, thin hammered sheets and hollow beads.

COLOR

As mentioned before, color is extremely important in Andean cosmovision, the metal colors had to do with their Gods and nature, and as such the society wished to display them in the best way possible. Each color was meant to mean something specific and convey a message inside the society is was used. In spite of this, the final surface colors depended on the available raw materials and the ingots that arrived from the still undetermined mines. The different alloys were as relevant as the process in the final surface color; the use of small amounts of Au and Ag in copper to create various colors is known all over the Andes, and was used repeatedly to manufacture silver or gold artefacts with a minimum amount of actual gold or silver (Easby 1966; Lechtman 1984a, 1985; Blust 1992).

To obtain the final colors, the preforms went through an extra process before being shaped and polished. This process was either depletion gilding, which involves a chemical treatment with a corrosive solution that would dissolve the copper from the surface or annealing and pickling which enriches the surface with either gold or silver (Lechtman 1984a). Either technique gives copper, the base metal, an appearance of gold or silver, even when the pure metal percentage is very low (Bray 1971). Considering the depletion gilding' corrosive agent would dissolve the silver in the surface, leaving only gold (Lechtman 1984a), and the lack of artefacts with a purely golden surface, we can affirm that the Milagro-Quevedo only used the annealing and pickling process. This process is repeated several times until the desired surface is enriched with the desired element and the desired color is obtained (Lechtman 1984a), which could explain the variety of colors found in these artefacts.

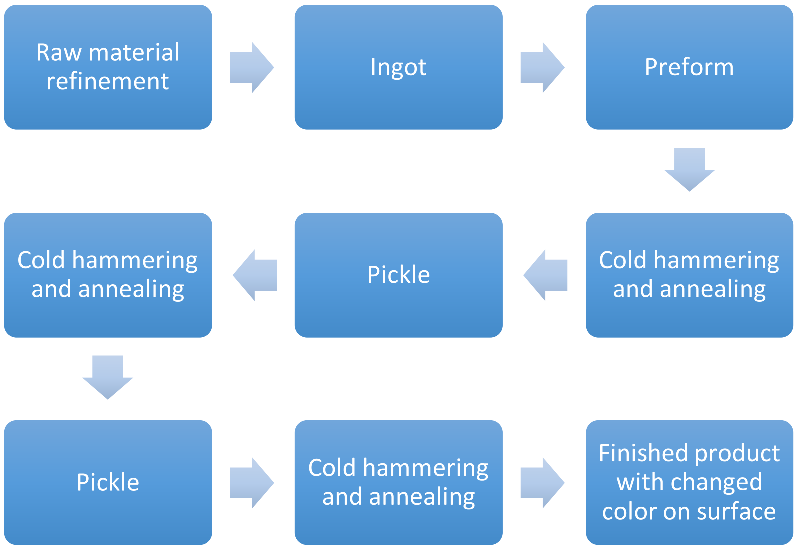

Figure 65: Process of annealing and pickling to change surface color of copper alloys. (Based on Lechtman 1984a, 1984b).

SHAPE

Wire

Figure 66: Artefacts manufactured with wire. Different width rings: a-VL009, b-1017; hook: c-VL2000; needle: d-VL3021.

The metals in this category include all the different width ear and nose rings, the hook and the needle, it is important to note both ornaments and implements are part of this category, so the manufacturing technique is not based on the final use of the artefact, but on the desired shape. The colors and alloy mixtures vary a lot and have no direct relation to the shape.

ID code	Artefact	Color	Alloy mixture
VL 009	Ear or nose ring	Bronze	Cu+As+Ag
VL 1000	Ear or nose ring	Bronze	Cu+As+Pb
VL 1006	Nose ring	Dark Bronze	Cu+As+Pb
VL 1017	Earing fragments	Bronze	Cu+As
VL 1033	Nose ring	Bronze	Cu+As
VL 1051	Earing fragment	Golden Bronze	Cu+As+Ag
VL 2000.1	Hook	Undetermined	Cu+Ag
VL 2000.2	Undetermined shape	Golden Bronze	Cu+As+Ag
VL 2005.1	Earing	Dark Bronze	Cu+As
VL 3009	Earing and fragment	Golden Bronze	Cu+As+Ag
VL 3021	Needle fragment	Bronze	Cu+As+Pb

Table 35: Artefacts made out of wire.

They were shaped through constant hot hammering and annealing until it was brought into a wire of the width desired, afterwards it was cut and brought to shape through cold hammering and manipulating (Sutliff 1998; Shimada & Craig 2013).

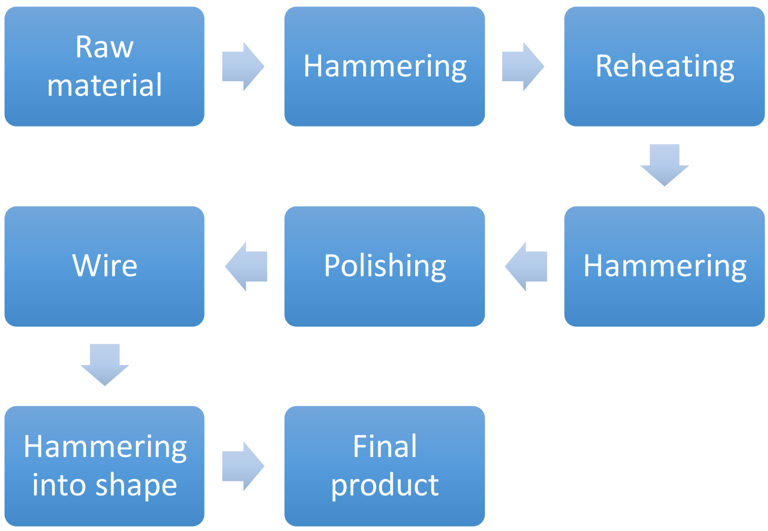

Figure 67: Manufacturing process for wire artefacts. (Based on Romero-Bastidas et al. 2017; Sutliff 1989)

Thin Hammered Sheets

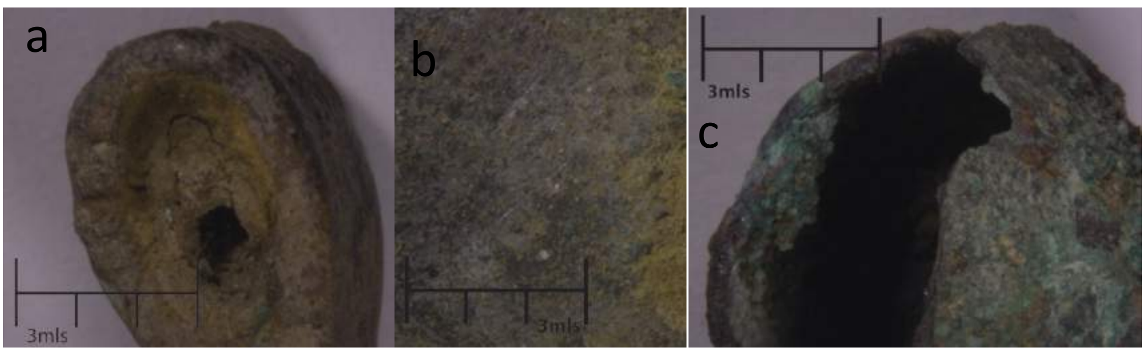

Figure 68: Artefacts manufactured with thin hammered sheets. a-VL013, b-VL036.3, c-VL2005.2.

The artefacts in this category include tweezers, bracelets, pectorals, bells and axes; as with the wire category, both ornaments and implements were manufactured with this technique and the colors and alloy mixtures were also varied and non specific to the manufacturing method used.

ID code	Artefact	Color	Alloy mixture
VL 013	Tweezers	Bronze	Cu+As
VL 036.1	Bracelet fragment	Dark Bronze	Cu+Pb
VL 036.2	Pectoral fragment	Undetermined	Cu+As+Ag
VL 036.3	Not identified fragment	Dark Bronze	Cu+As+Ag
VL 114	Tweezers fragment	Bronze	Cu+As
VL 1023	Bell	Golden Bronze	Cu+As+Pb
VL 2005.2	Bell	Golden Bronze	Cu+As
VL 3027	Axe fragments	Bronze	Cu+As

Table 36: Artefacts made from thin hammered sheets.

The artefacts were manufactured by taking a metal ingot and working it into a thin sheet by extensive cold hammering and reheating; afterwards there would be several series of cutting and shaping, as described by Sutliff (1998), Lechtman (1991) and Romero-Bastidas et al. (2017).

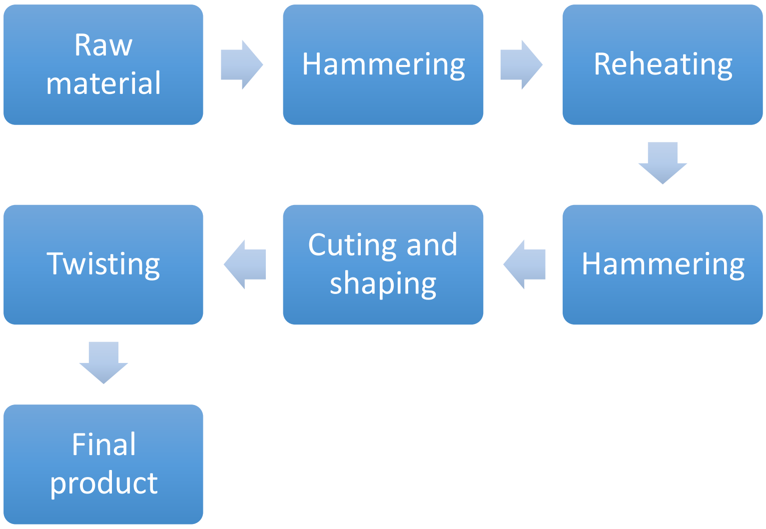

Figure 69: Manufacturing process for thin sheet artefacts. (Based on Romero-Bastidas et al. 2017).

Hollow Beads

Figure 70: Hollow beads. a-VL010, b-VL106, c-VL3003.

The artefacts in this category are all necklace beads, with silver, dark bronze or golden bronze surfaces; and include alloys containing copper, arsenic, lead, gold and silver. Once again, the surface color and alloy mixture does not have a direct relationship to the manufacturing technique. On the other hand, the final use or purpose is the reason behind their manufacturing technique as the bead need to be threated through a thin rope to create a necklace or other ornament.

ID code	Artefact	Color	Alloy mixture
VL 010	4 Necklace beads	Dark Bronze	Cu+As+Ag
VL 106	2 Necklace beads	Golden Bronze	Cu+As+Pb
VL 3003	Necklace bead	Silver	Cu+As+Ag+Au

Table 37: Hollow Bead Artefacts.

These artefacts were either shaped from a metal sheet, beaten into a cylindrical shape, cut, had their edges forged together and then exposed to heat again to eliminate forging marks, or created with heated molds. Considering the tradition they belong to (Sutliff 1998; Shimada & Craig 2013), and the lack of evidence of molds in the area, the first process seems more likely.

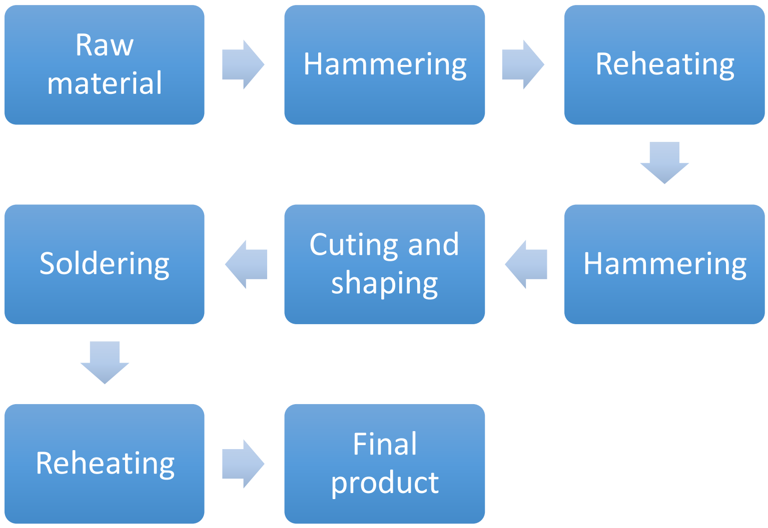

Figure 71: Manufacturing process for hollow beads. (Based on Lleras & Ontaneda 2010; Romero-Bastidas et al. 2017))

ALLOYS

The people of the Andes developed several different alloys before the arrival of the Spanish; some of these alloys were accidental due to lack of purifying techniques for precious metals, while others were purposefully made due to metals importance and final color wanted (Lechtman 1985). The Milagro-Quevedo artefacts analyzed showed the presence of several copper based alloys, which can be classified as either copper-silver or copper-arsenic alloys.

There is just one artefact that can be considered ‘tumbaga’, as it contains copper, silver, gold and arsenic. This artefact has a golden bronze color, and only small quantities of gold, which might suggest it was an accidental alloy mixture. The copper-silver alloys, on the other hand, can be either accidental or purposefully made, as some have large amounts of Ag, while other only show traces of this metal. Finally, the copper-arsenic alloys, which are the ones with the highest presence in the analyzed group, are intimately related to the central Andean pre-Hispanic history.

The existence of different alloys might be related to the importance of color in the Andean world, golden and silver artefacts were meant to be used for ornamental artefacts worn by high hierarchy people; while copper or bronze colored artefacts were used for implements and ornaments for low hierarchy people. Even if gold and silver per se were not valued as a raw material, the final color of the artefact and the shape of the same was (Zevallos Menéndez 2005). In the case of the Milagro-Quevedo and the artefatcs form Vuelta Larga, it seems like alloy composition and color is related to final use of the artefacts.

The use of these alloys, both copper-silver and copper-arsenic can be tracked back to societies all over the Andes, but specially the Central Andes, from Ecuador to northern Peru, with the Moche, Chimú, Chincha and Manteño-Huancavilca being some of the groups that used them. The presence of these alloys in Vuelta Larga show this society’s relationship with the Manteño (Romero-Bastidas et al. 2017), Sicán (Lechtman 1991) and possibly even the Moche, groups that are known to have used these alloys as well. Considering the lack of information about mines in the area, the ingots of the raw material probably came from elsewhere, which confirm that the Milagro-Quevedo and the Yaguachi chiefdom had contact with other contemporary groups in the area.

FIBERS

As mentioned before, pre-Hispanic societies were known for their textile manufacture and their use of camelid wool and cotton for weaving (Ainsworth 1925; Lange et al. 1987). The main camelids used were alpacas and llamas, which were domesticated in the area between 6000 and 4500 years ago, both animals were used for wool, with alpaca wool being finer and softer, while llama is coarser and stiffer.

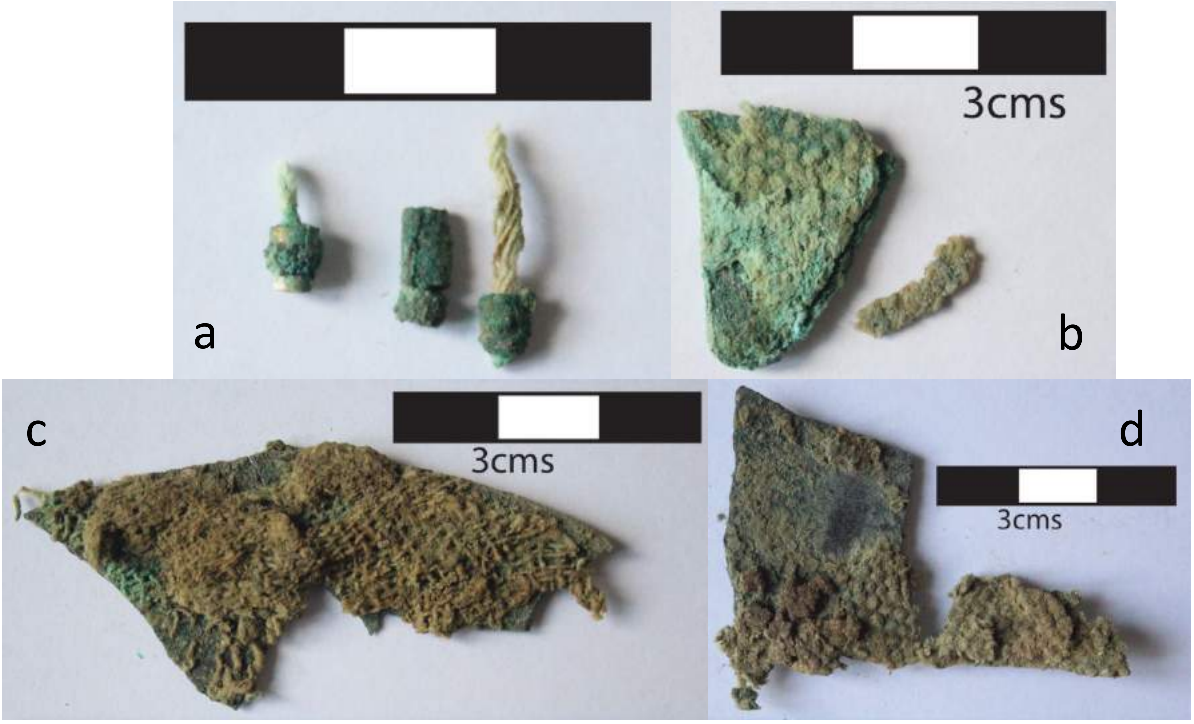

Figure 72: Artefacts with fibers. a-VL010, b-036.1, c-VL036.2, d-VL036.3.

Through Environmental SEM imaging it was determined that the Milagro-Quevedo used three different fibers for their artefacts: cotton, alpaca and llama wool. The fibers, found in relation to burials, were coiled together to create threads and then either used for textile weaving or as jewelry threads, for beads (Lange et al. 1987). Each type of fiber seems to have been used for an specific purpose: cotton was used for clothes, while llama and alpaca wool was used for ornamentation bases. Clothes were made with cotton and woven in a plain weave pattern; while jewelry used camelid hairs for either threads or to be woven in a float weave pattern. It was impossible to determine what specific camelid the fibers analyzed came from, but it could be affirmed that the thread came from an alpaca, and the textile fiber came from an alpaca or the finer area of the llama.

CHAPTER 9: CONCLUSIONS

This research attempted to approach Ecuadorian metallurgy of the Guayas Basin through a metallurgical analysis of artefacts found in a Milagro-Quevedo burial mound in Vuelta Larga, through the analysis of 22 artefacts recovered during excavations preformed by Florencio Delgado, PhD. and his team in the late 90s. The artefacts were analyzed with a stereomicroscope, before going through XRF, SEM-EDS and Environmental SEM analysis. The analysis showed the different manufacturing techniques of both metals and textiles, the possible purpose of each artefact, the alloys they were made of and helped determine possible connections of the Yaguachi chiefdom with other contemporary groups or societies in the area.

The stereomicroscope and the macroscopic analysis helped determine the different manufacturing techniques that were used for all artefacts indistinctively and their possible original purpose: either implements or ornaments. The analyzed artefacts included nose and ear rings, bells, bracelets, breast plates, tweezers, needles, some beads and a hook; and were classified based on purpose and manufacturing method. The Milagro-Quevedo would use constant hammering and reheating for both shaping these artefacts and changing their surface color. These techniques have been known to be used by the Manteño-Huancavilca, and by several northern Peruvian groups.

By the use of XRF and SEM-EDS it was determined that all of the artefacts were copper alloys, containing various amounts of arsenic, lead, silver and gold. Presence of Fe, Ca, Cl and Si was also detected, which was related to the soil the artefacts were found on. All of the artefacts identified as implements were made with Cu-As, or Cu-As-Pb; while some ornaments are the artefacts with Au and Ag in them. These alloy mixtures also reflect on the surface color of the artefacts, which include bronze for all implements and some ornaments, gold bronze, dark bronze and silver for the rest of the ornaments.

The last artefacts to go through analysis were the ones with fibers. These artefacts were analyzed both by stereomicroscope and environmental SEM, which was preformed in both 3 artefacts and on 5 fresh hair samples for comparison purposes. After analyzing the artefacts under stereomicroscope, three different patterns were identified: thread for beading, plain weave for bracelet lining and float weave for clothes. The SEM imaging, on the other hand, helped compare the artefacts with the fresh hair samples, and allowed the determination that the fibers used for the jewelry cords were made with alpaca hair, while the textiles were either made with cotton, in the case of bracelet lining or with alpaca or thin llama hair, in the case of the breastplate lining.

The results showing the alloy mixtures found in the site and the textile techniques help determine the existence of a possible connection between the site the artefacts come from and the Manteño-Huancavilca and the Sican, as both groups are known to use copper-arsenic in the manufacturing of their metallic artefacts. The mines, the ingots the Yagiachi chiefdom used came from, are still unknown, but would probably connect these societies together in some way.

FINAL CONCLUSIONS

The results from the analysis of the 22 artefacts lead to the following conclusions:

a) The alloys used by the Milagro-Quevedo in the Yaguachi area were Cu-As, Cu-As-Pb, Cu-Pb, Cu-As-Ag, Cu-As-Ag-Au and Cu-Ag-Au.
b) The artefacts analyzed were either implements or ornaments.
c) The artefacts were manufactures by first cold-hammering and reheating the raw material ingots, the following steps varied from technique to technique.
d) The surface color was really important for the Milagro-Quevedo, and the colors found in the artefacts vary from bronze, to silver, including dark bronze and golden bronze. The artefact use and wearer were related to their surface color, which was reached through annealing or depletion gilding.
e) The Milagro used alpaca and llama hair, as well as cotton for weaving and cords.
f) The Yaguachi probably had contact with other groups from the area, as the alloys used are quite similar to those found in Manteño and Sican sites.

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APPENDIX A: PHOTOGRAPHED ARTEFACTS

Figure 73: VL009, ring.

Figure 74: VL010, beads with fiber.

Figure 75: VL013, tweezers.

Figure 76: VL036.1, bracelet fragment.

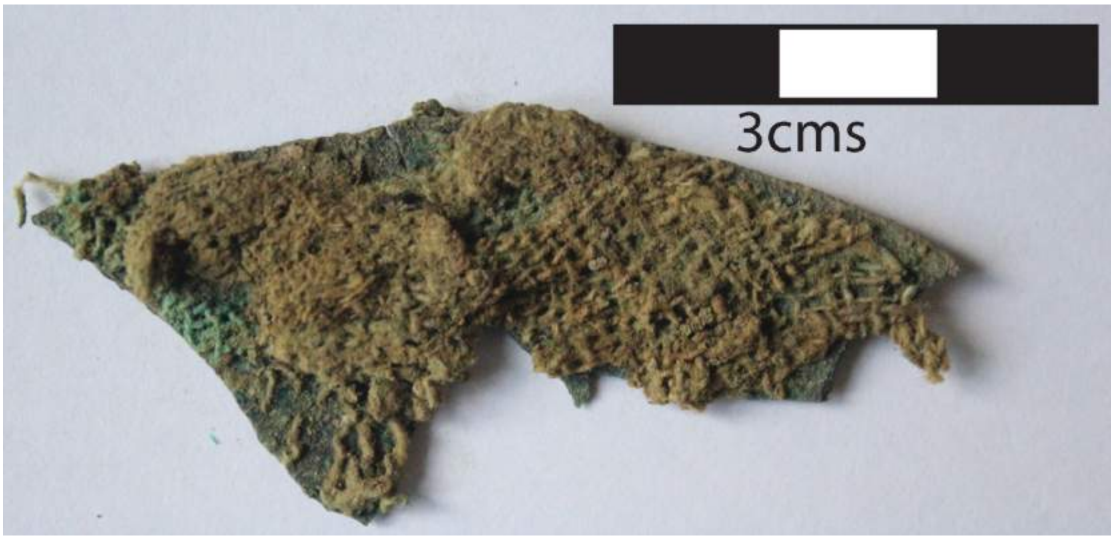

Figure 77: VL036.2, pectoral fragment.

Figure 78: VL036.3, not identified fragment.

Figure 79: VL106, beads with fiber.

Figure 80: VL114, tweezers fragment.

Figure 81: VL1000, ring.

Figure 82: VL1006, nose-ring.

Figure 83: VL1017, ring fragments.

Figure 84: VL1023, bell.

Figure 85: VL1033, nose-ring.

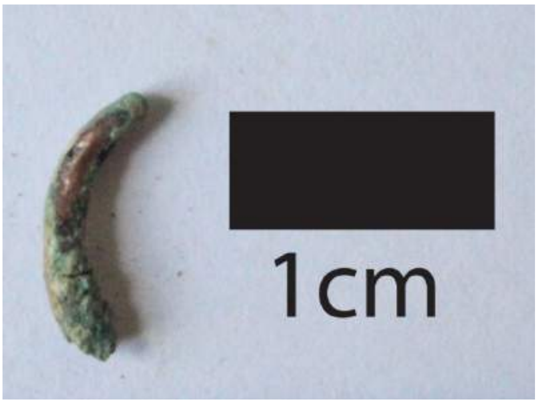

Figure 86: VL1051, ring fragment.

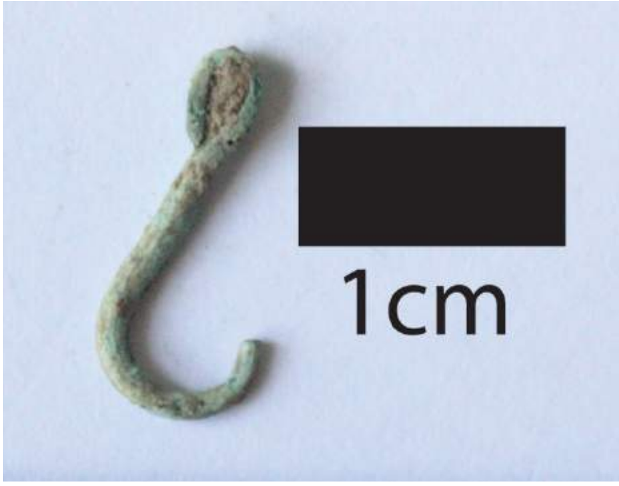

Figure 87: VL2000.1, hook.

Figure 88: Vl2005.1, ring.

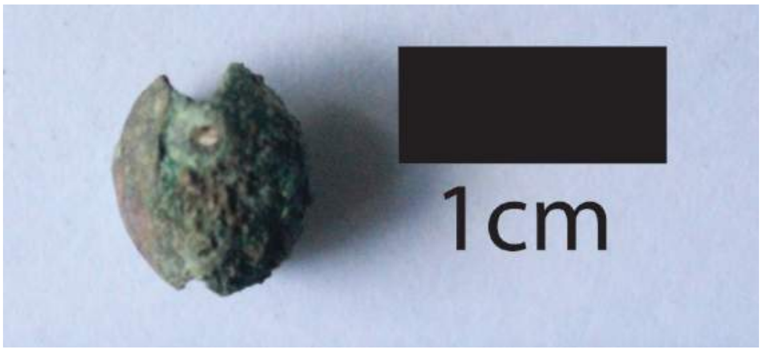

Figure 89: VL2005.2, bell.

Figure 90: VL3003, bead.

Figure 91: VL3009, ring and ring fragment.

Figure 92: VL3021, needle fragment.

Figure 93: VL3027, axe fragments.

APPENDIX B: ARTEFACTS UNDER STEREOMICROSCOPE

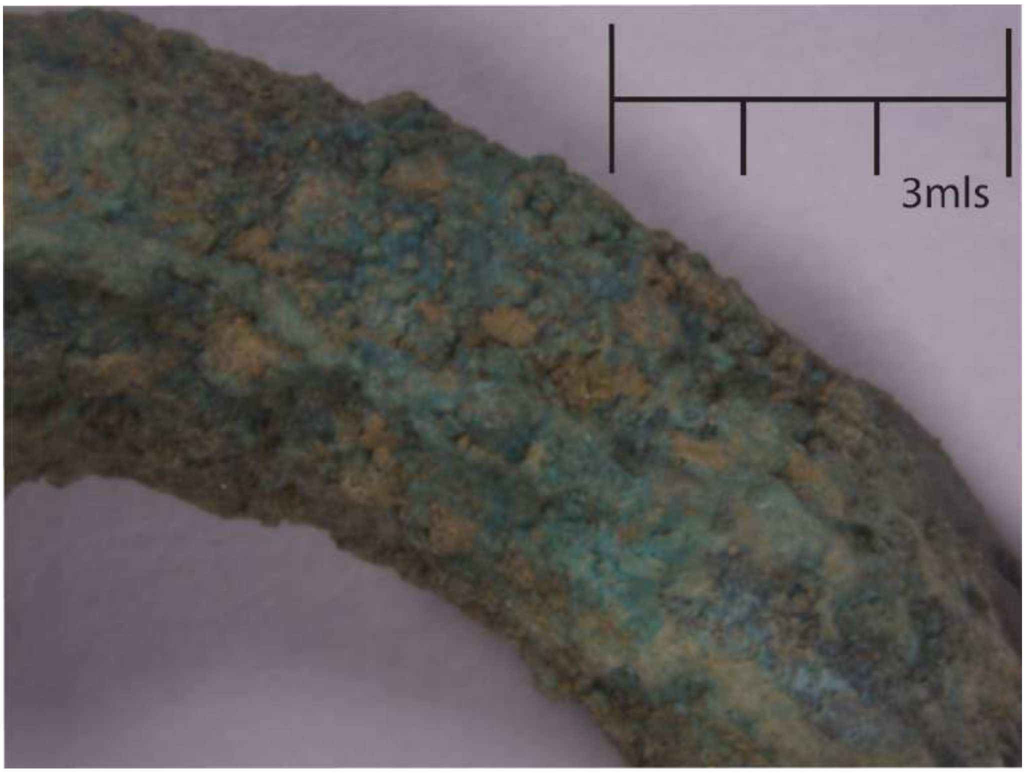

Figure 94: VL009, ring.

Figure 95: VL010, beads with fibers, fiber detail.

Figure 96: VL010, bead with fiber.

Figure 97: VL013, tweezers detail.

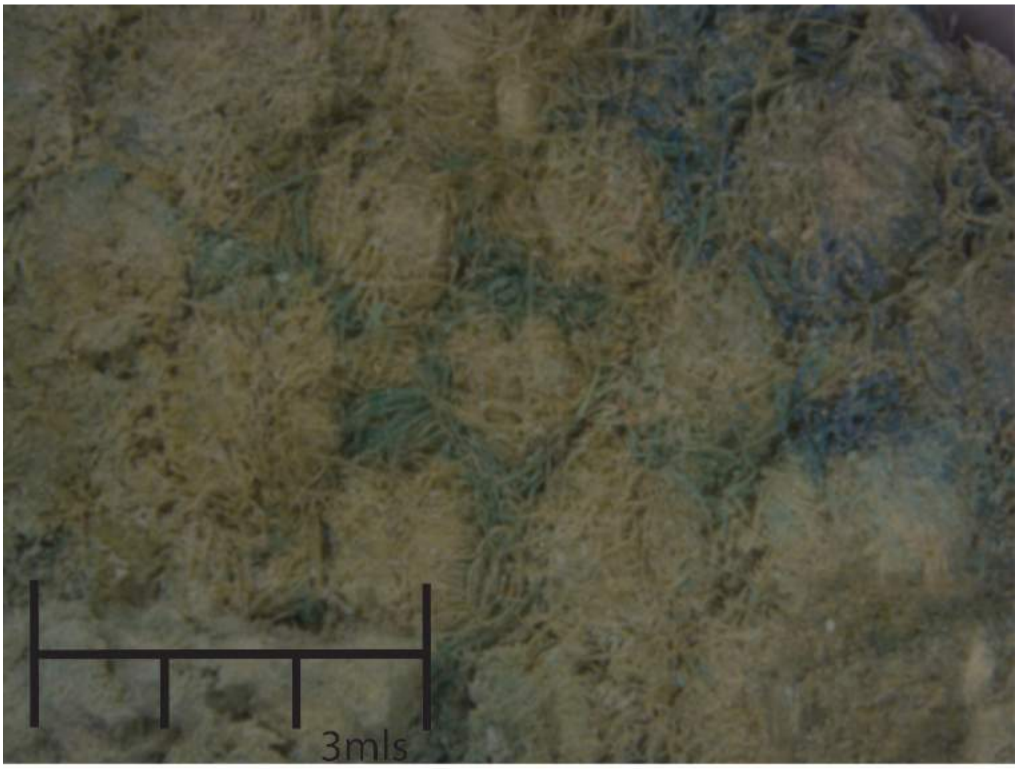

Figure 98: VL036.1, bracelet fragment textile.

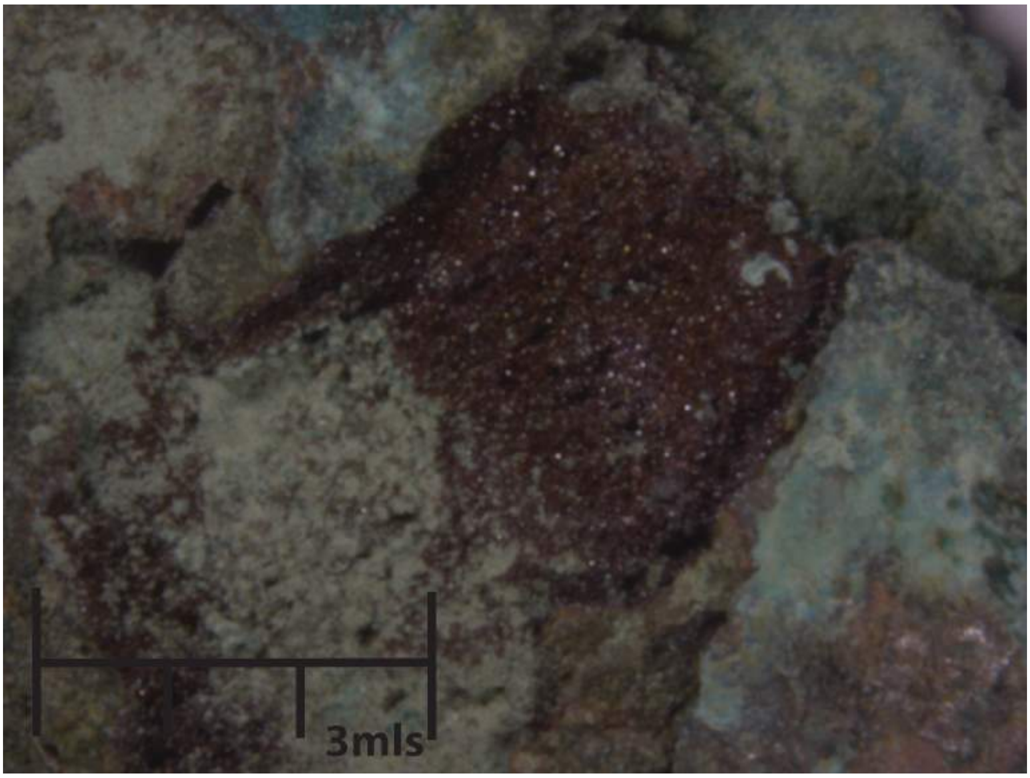

Figure 99: VL036.1, bracelet fragment detail.

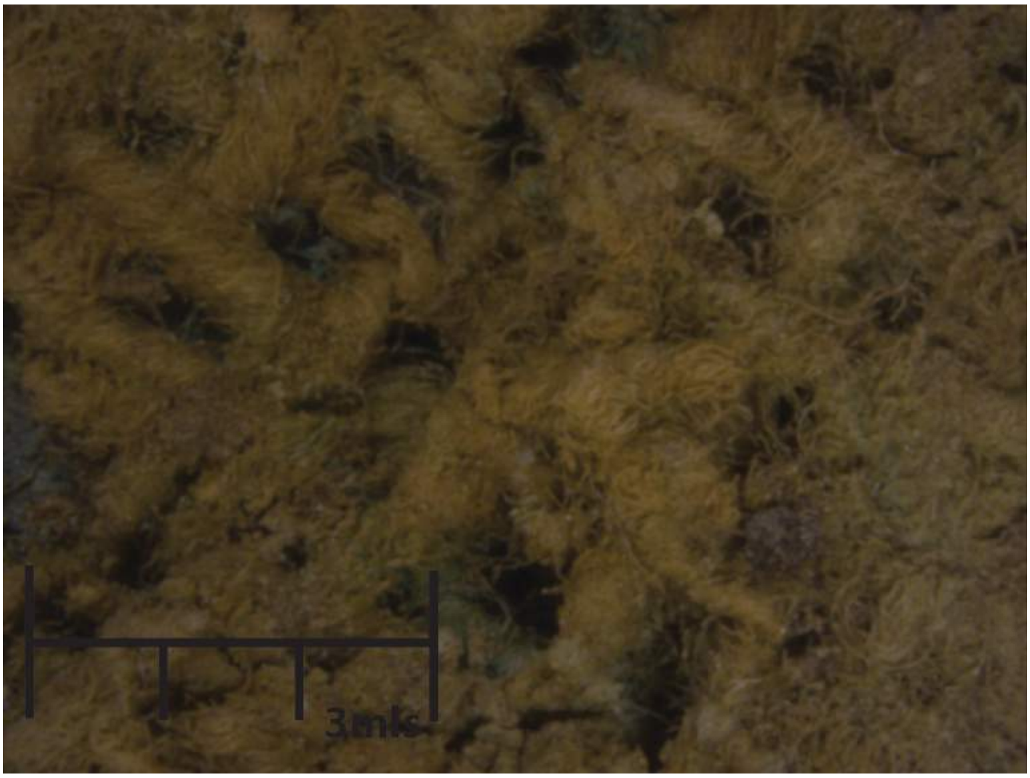

Figure 100: VL036.2, pectoral fragment textile.

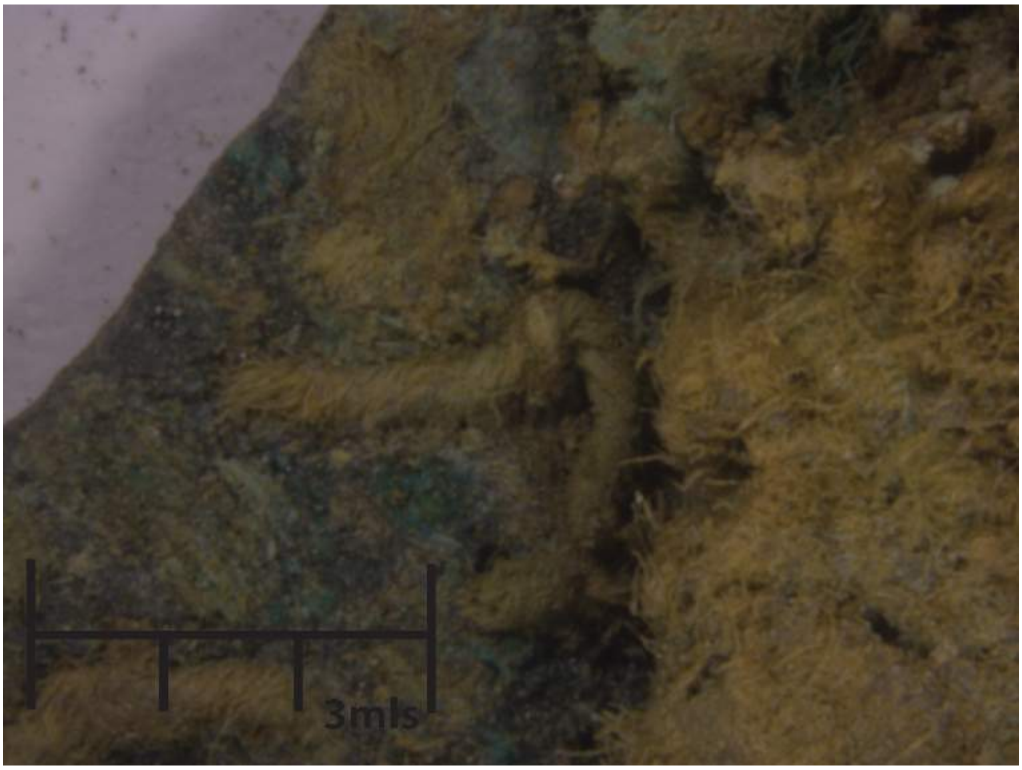

Figure 101: VL036.2, pectoral fragment detail.

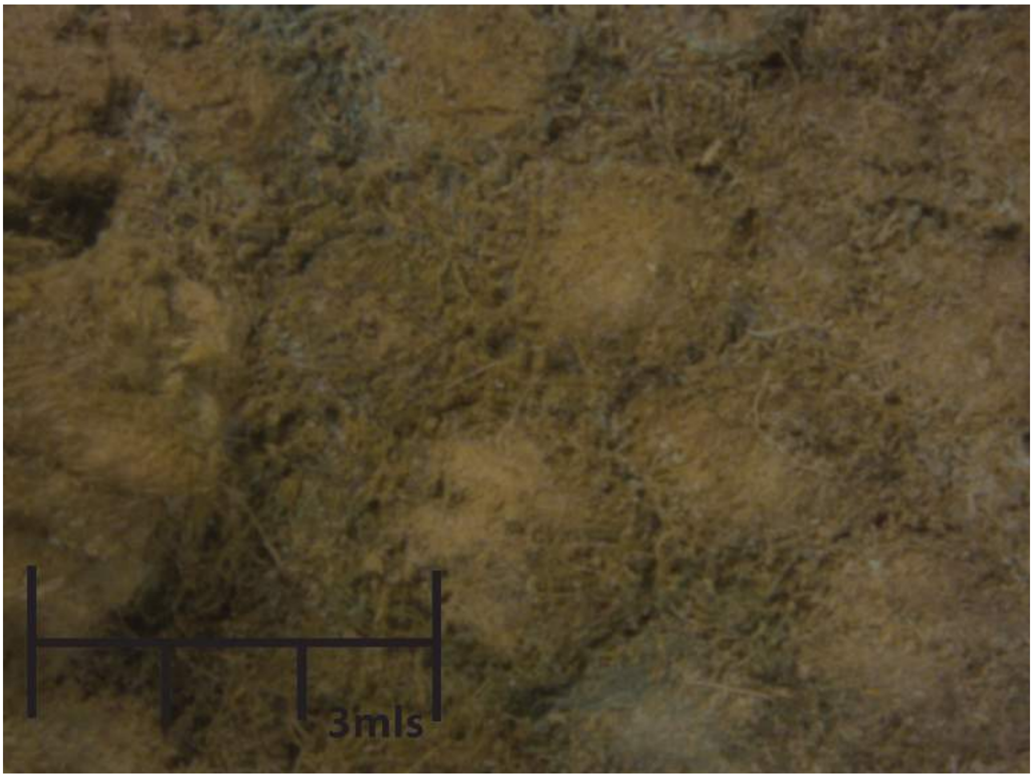

Figure 102: VL036.3, non-identified fragment textile.

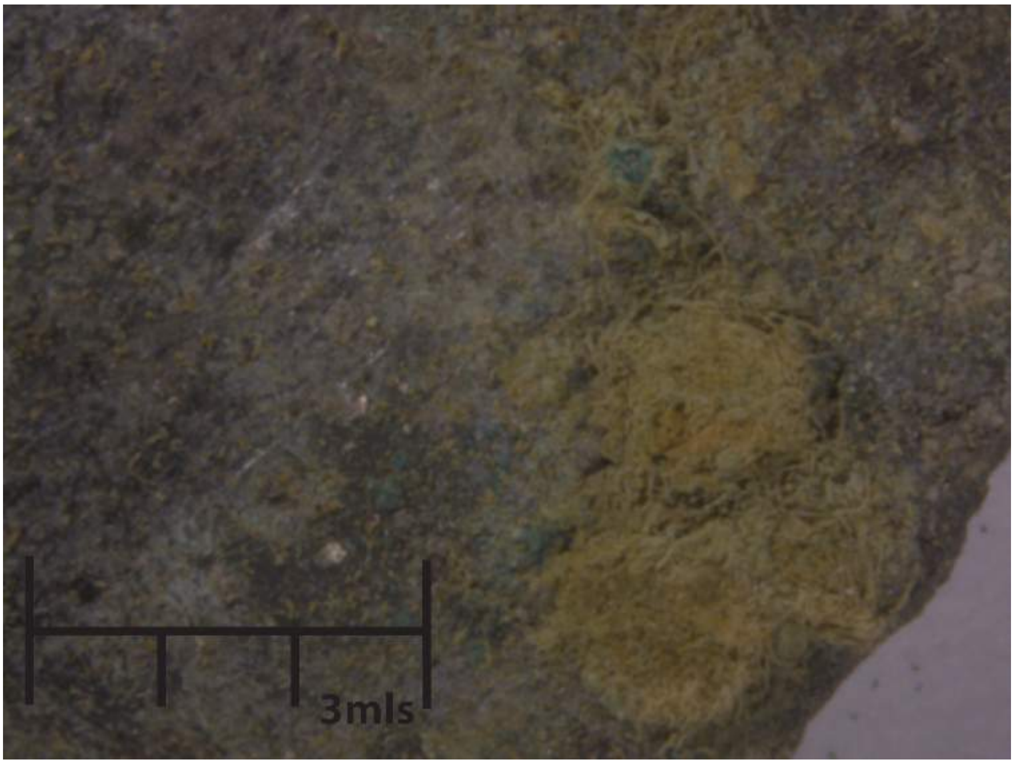

Figure 103: VL036.3, non-identified fragment.

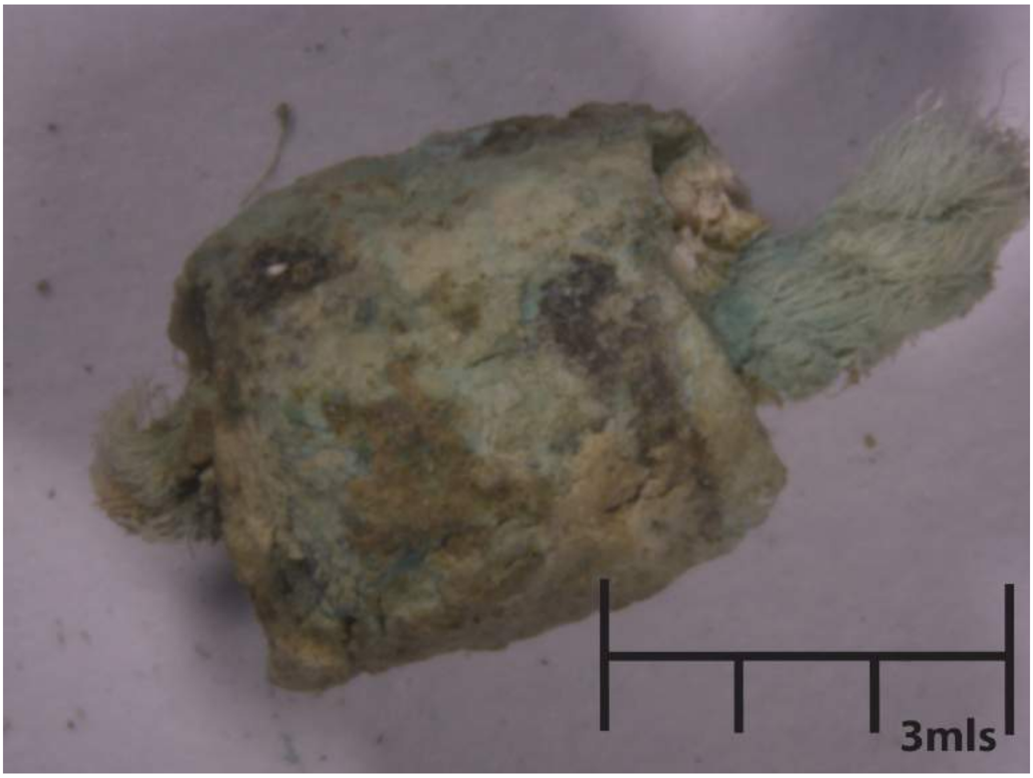

Figure 104: VL106, bead with fiber.

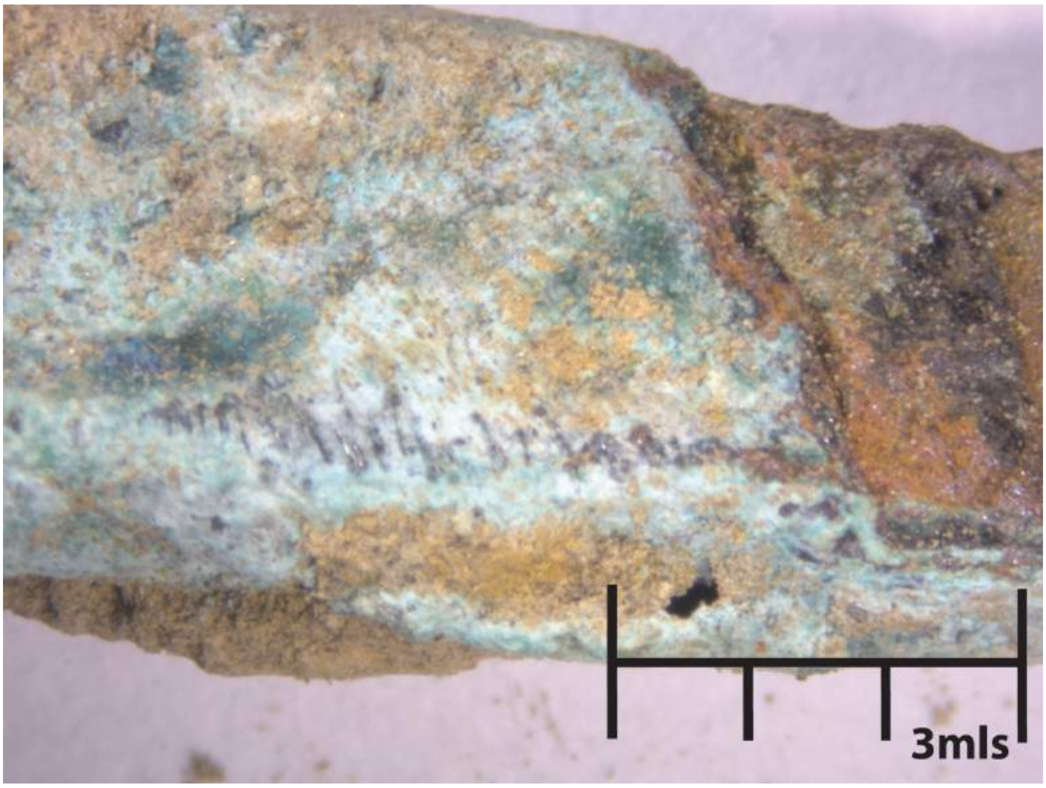

Figure 105: VL114, tweezers fragment.

Figure 106: VL1000, ring polished area.

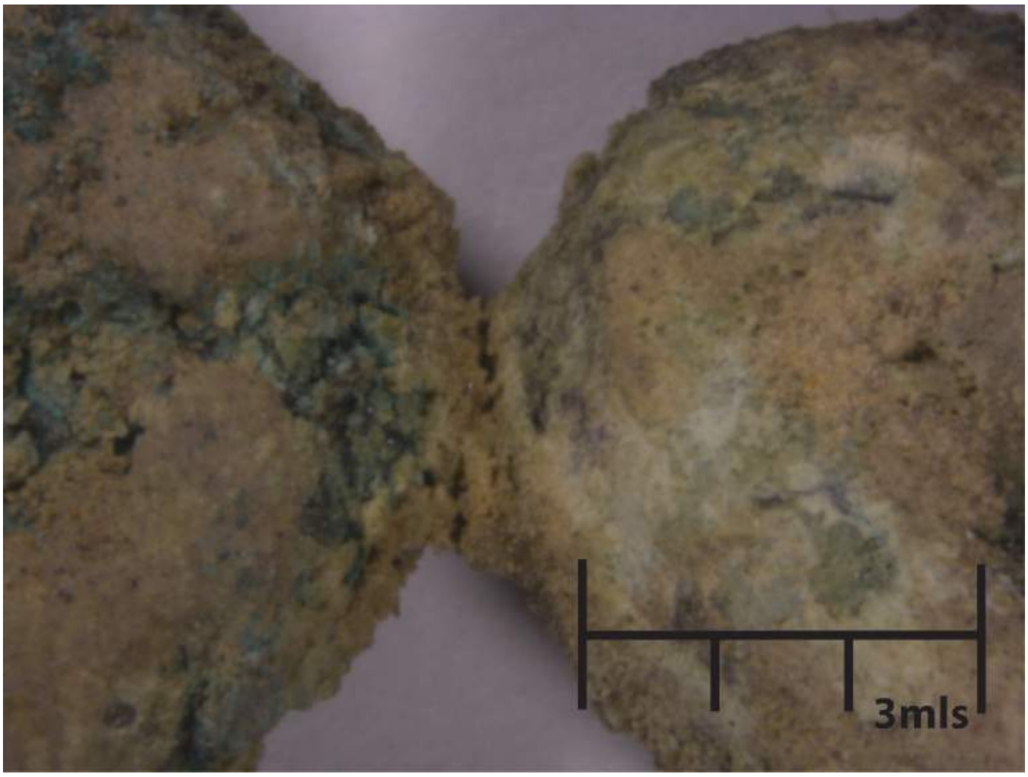

Figure 107: VL1006, nose-ring detail.

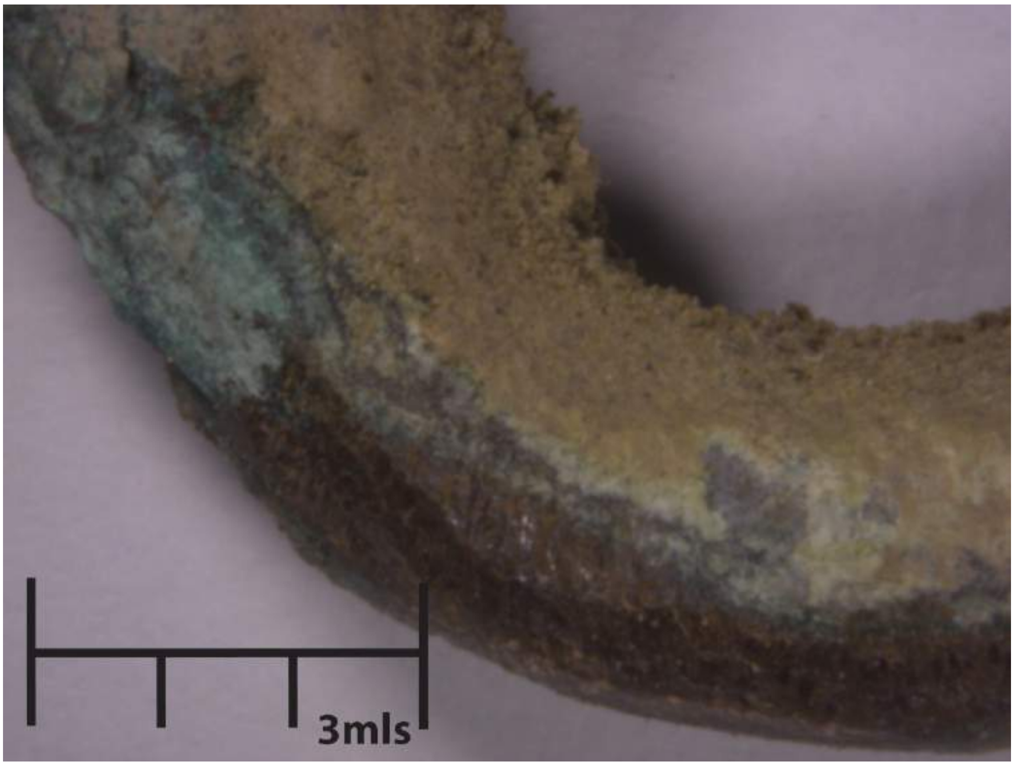

Figure 108: VL1006, nose-ring polished area.

Figure 109: VL1017, ring polished area.

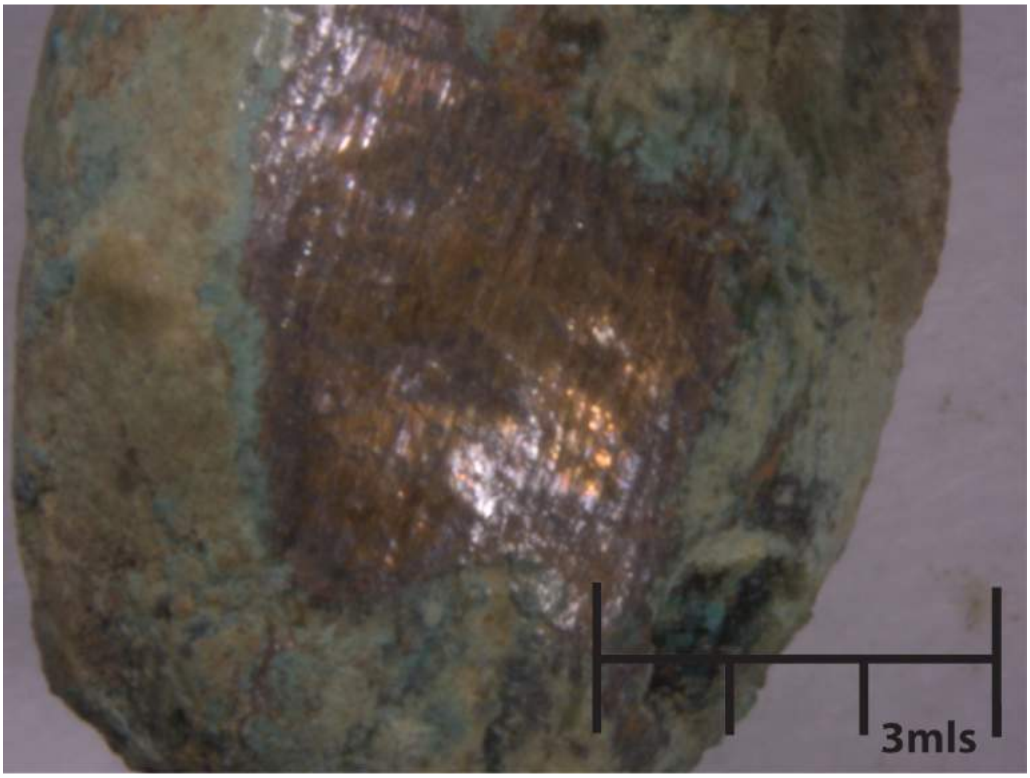

Figure 110: VL1023, bell polished area.

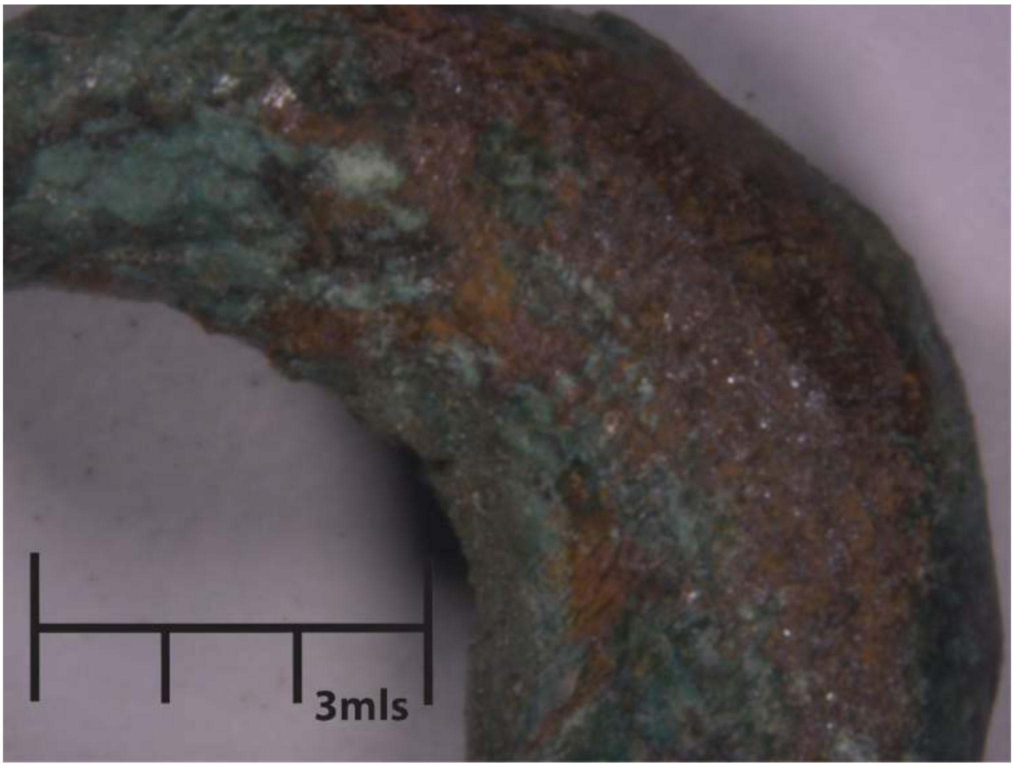

Figure 111: VL1033, nose-ring polished area.

Figure 112: VL1051, ring polished area.

Figure 113: VL2000.1, hook detail.

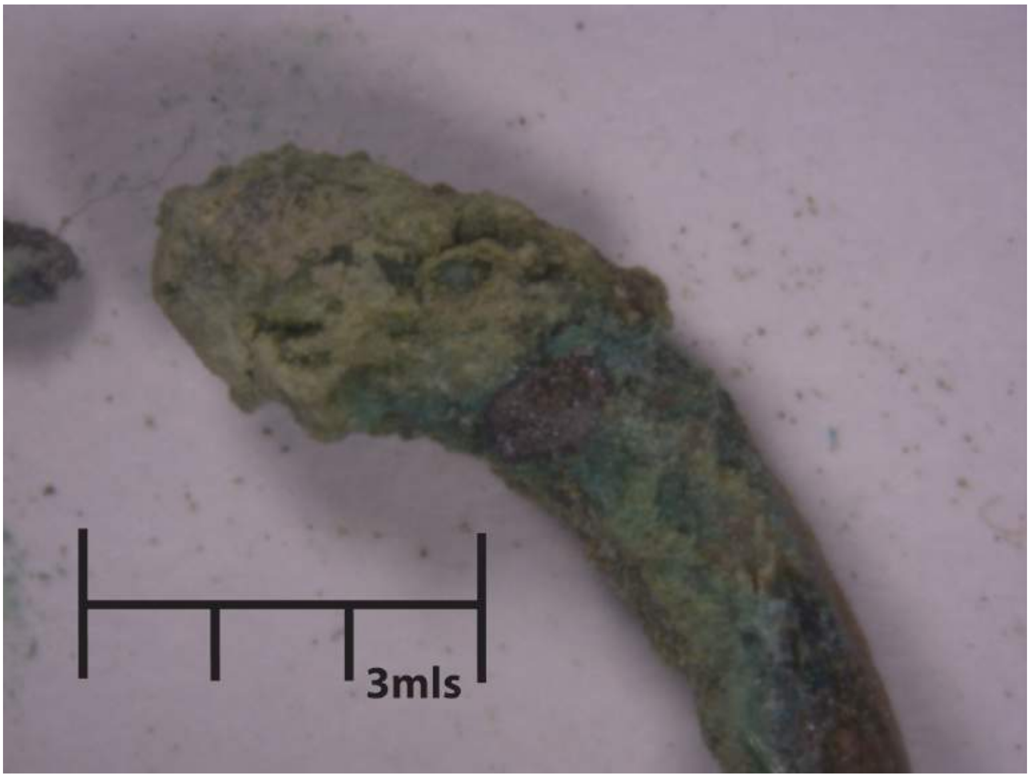

Figure 114: VL2005.1, ring detail.

Figure 115: VL2005.2, bell detail.

Figure 116: VL3003, bead polished area.

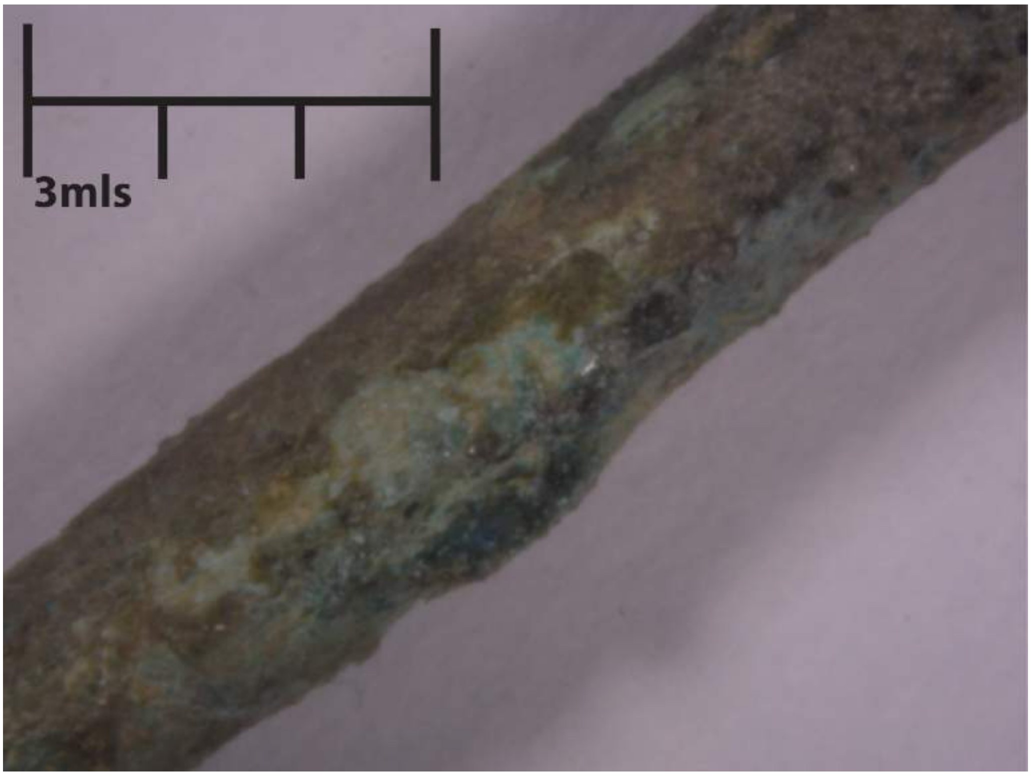

Figure 117: VL3021, needle detail.

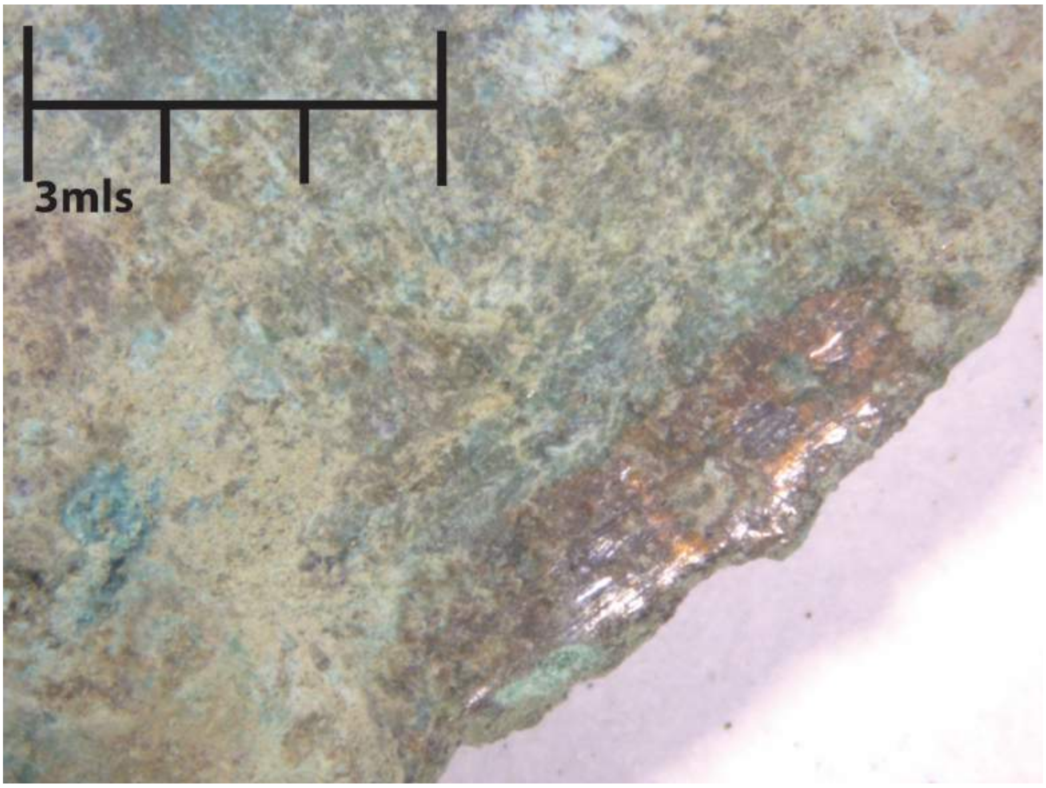

Figure 118: VL3027, axe fragment polished area.

APPENDIX C: ARTEFACTS' SPECTRA

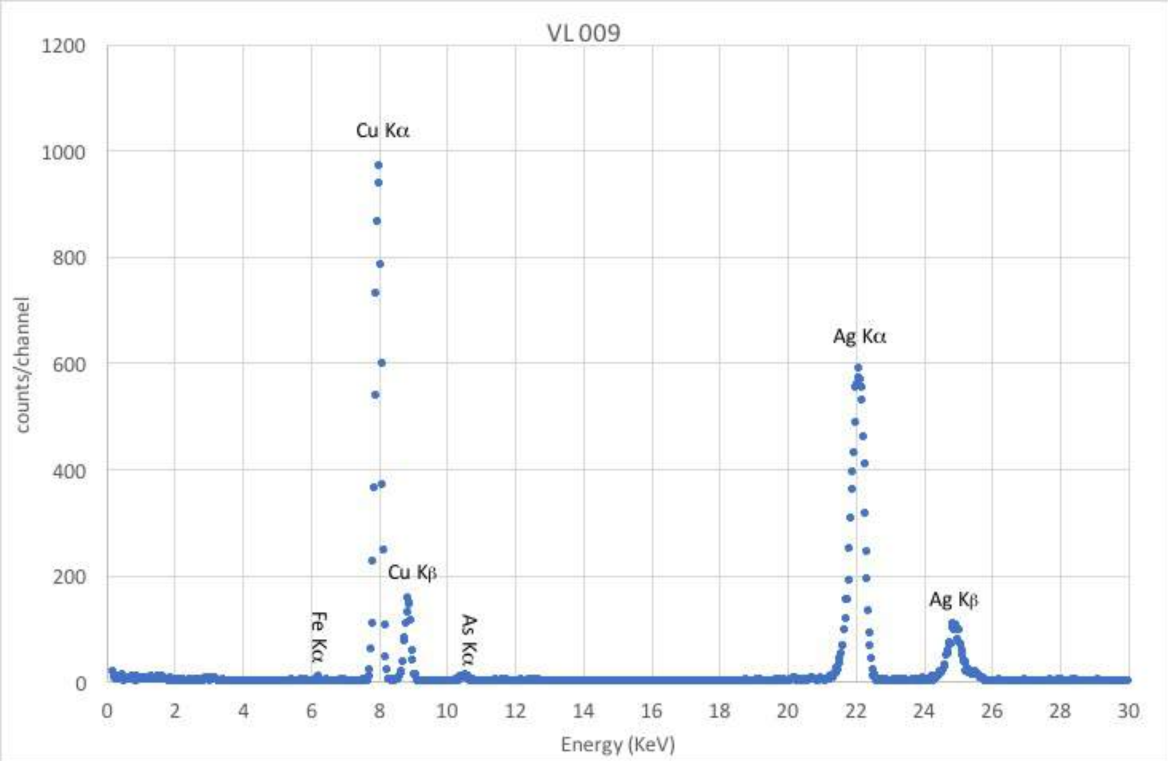

Figure 119: VL009 XRF spectra.

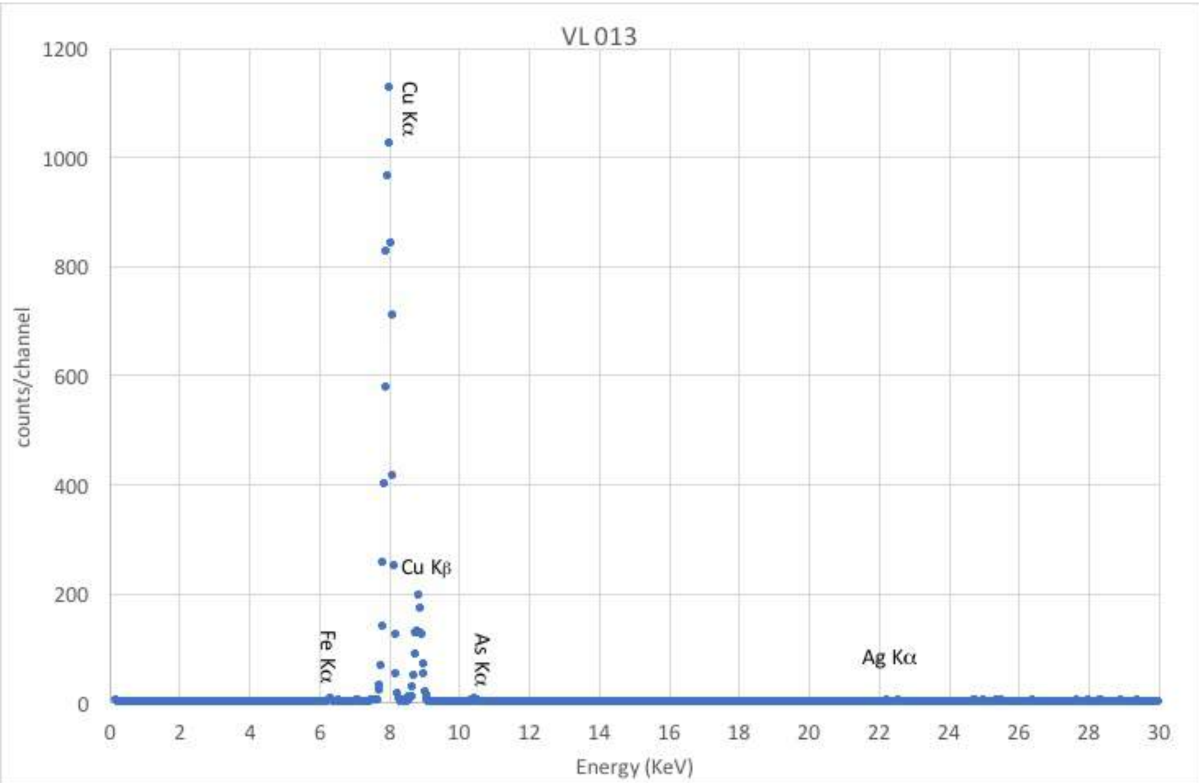

Figure 120: VL013 XRF spectra.

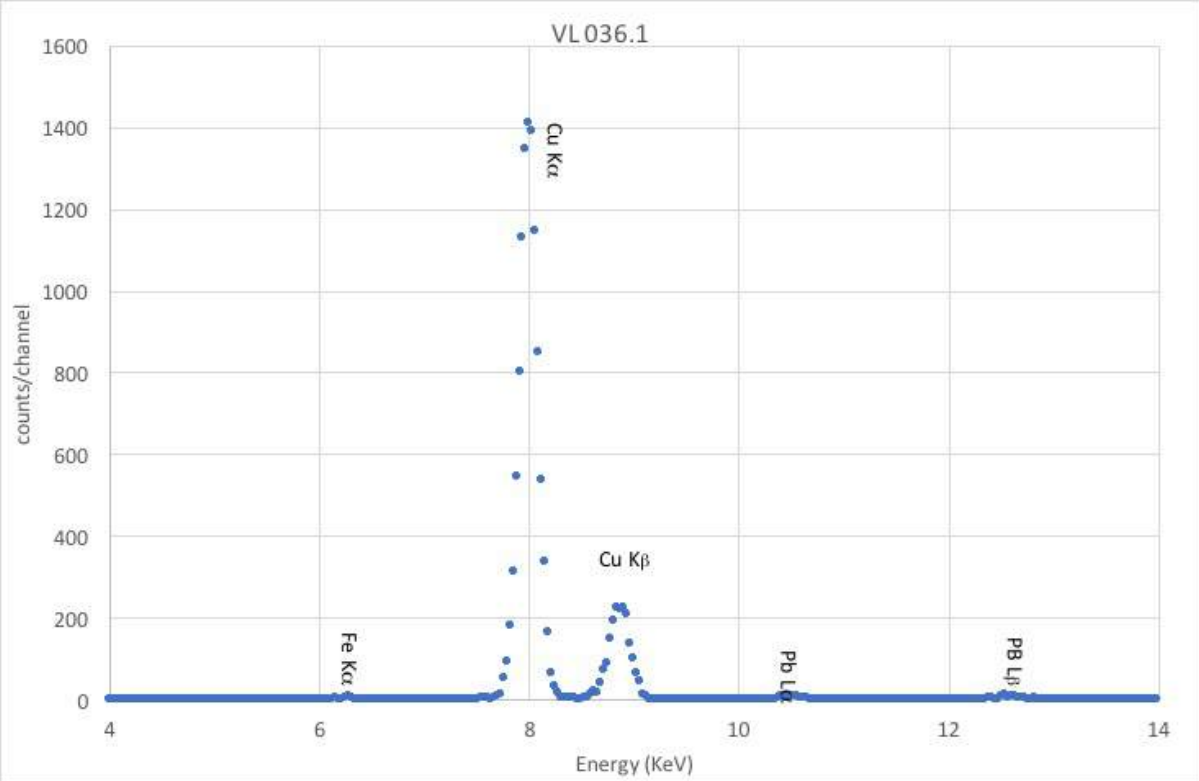

Figure 121: VL036.1 XRF spectra.

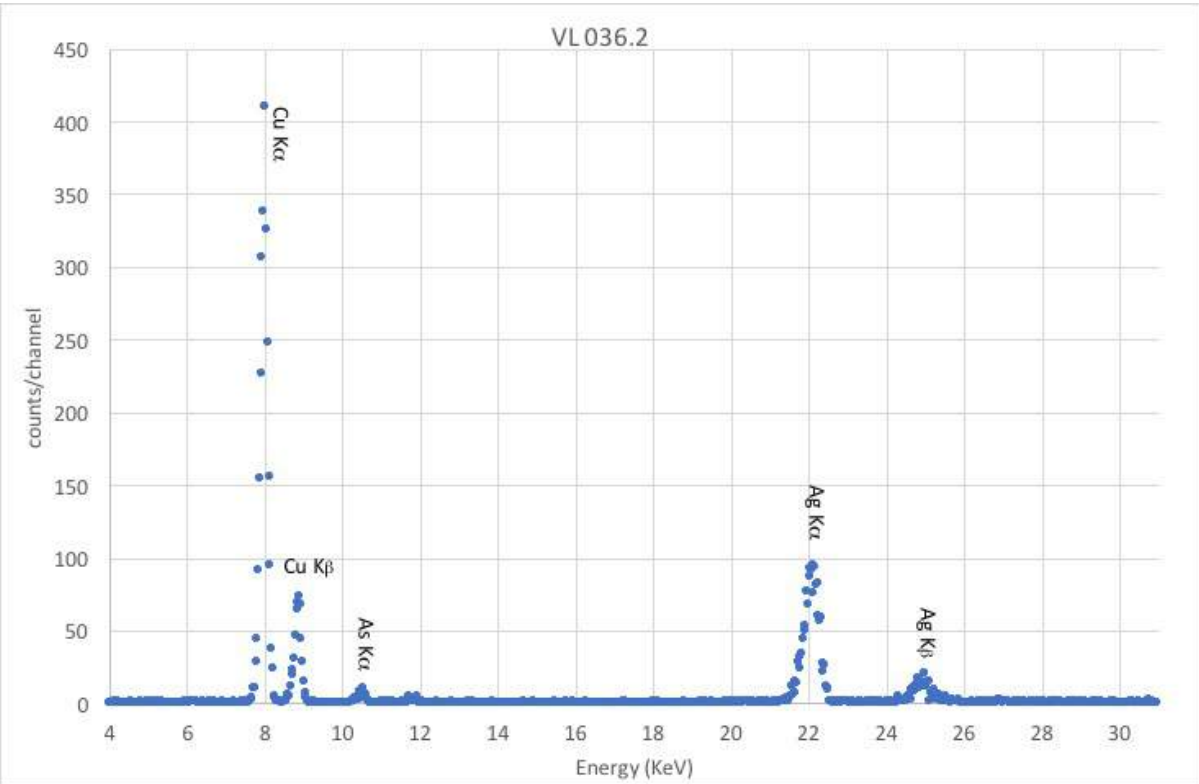

Figure 122: VL036.2 XRF spectra.

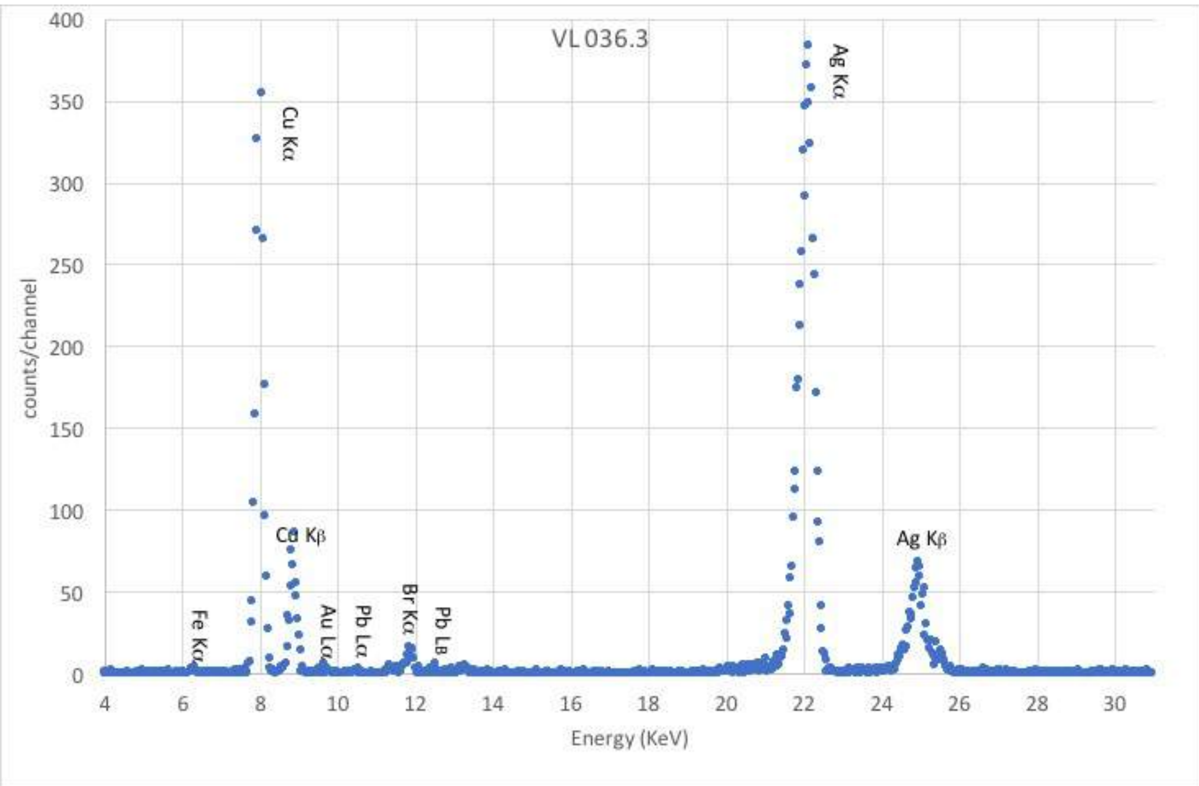

Figure 123: VL036.3 XRF spectra.

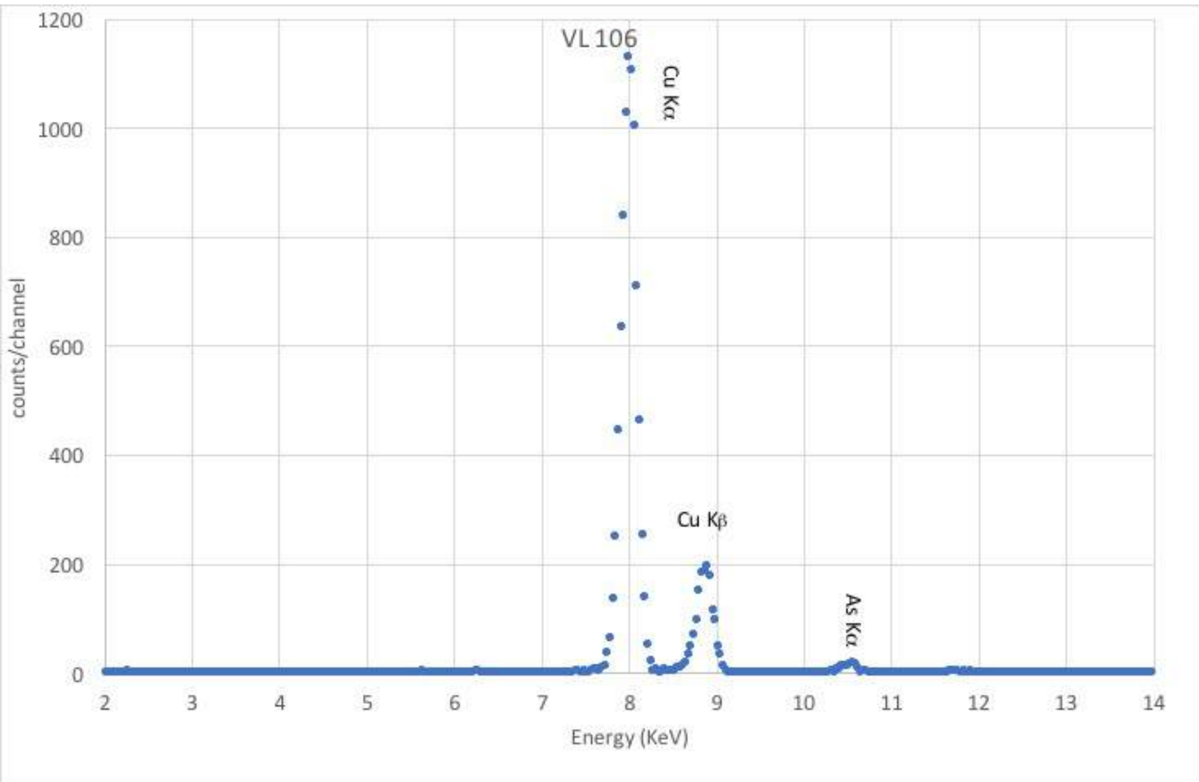

Figure 124: VL106 XRF spectra.

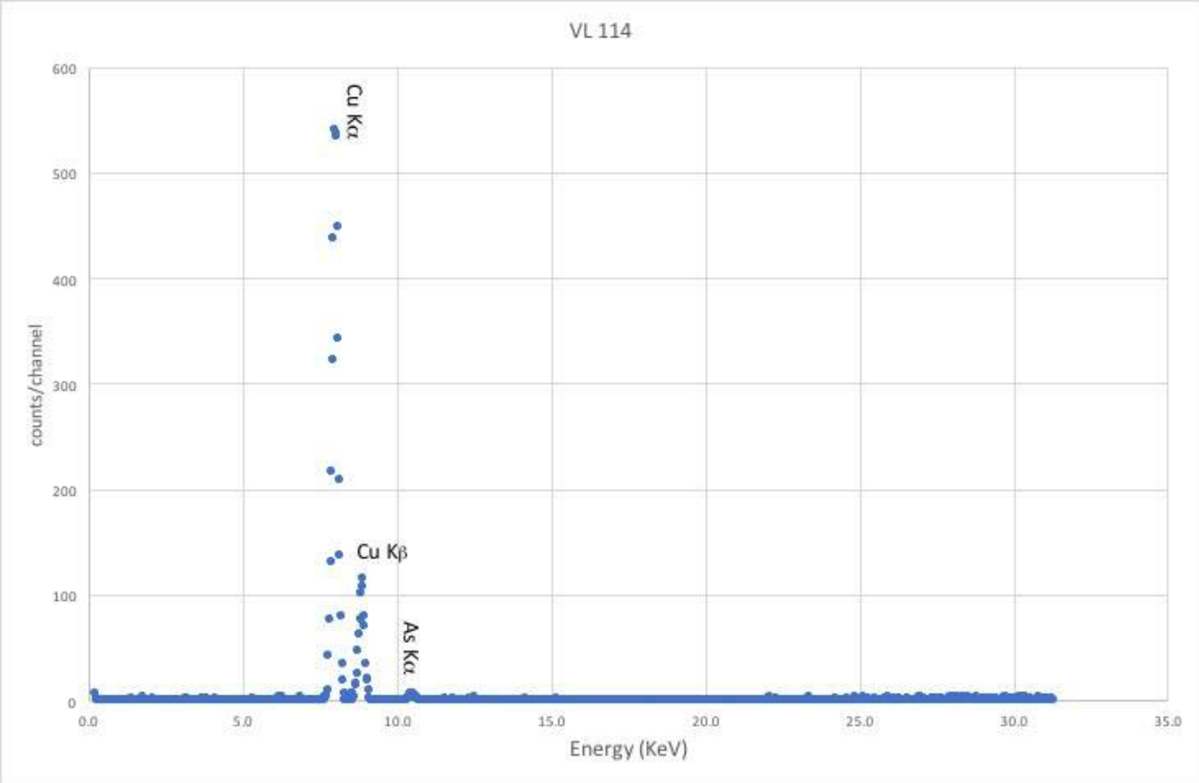

Figure 125: VL114 XRF spectra.

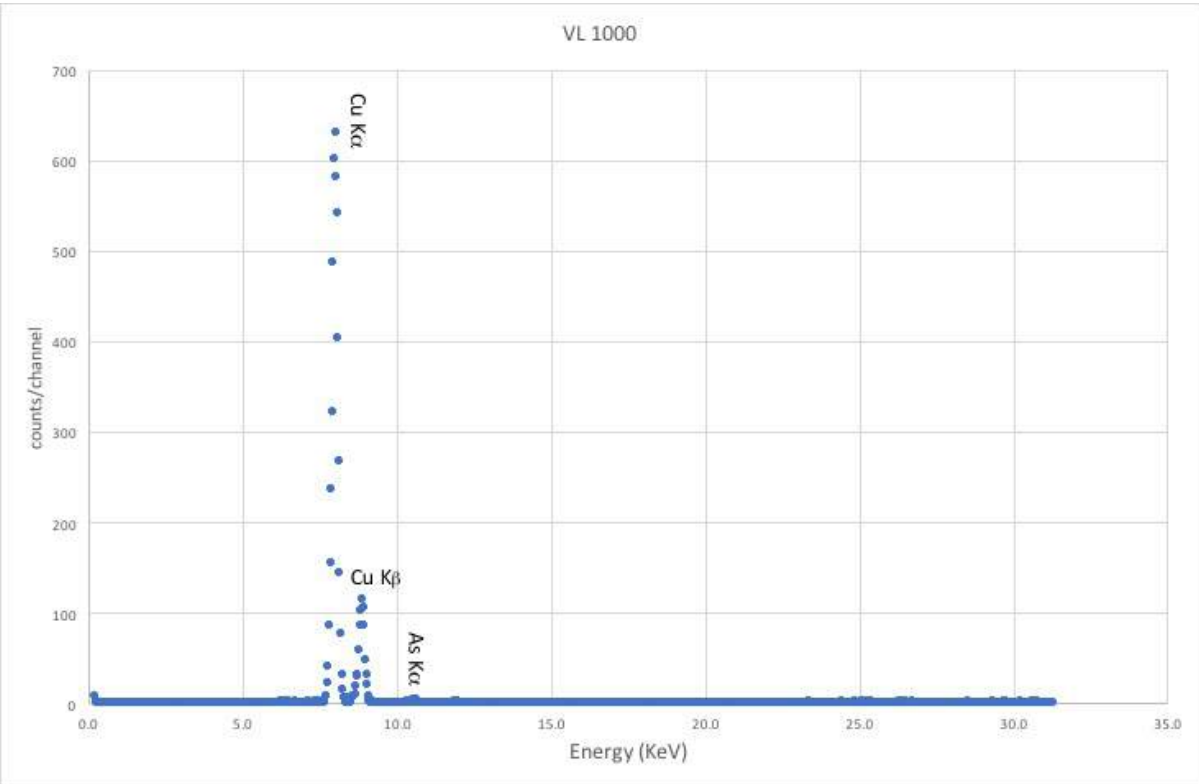

Figure 126: VL1000 XRF spectra.

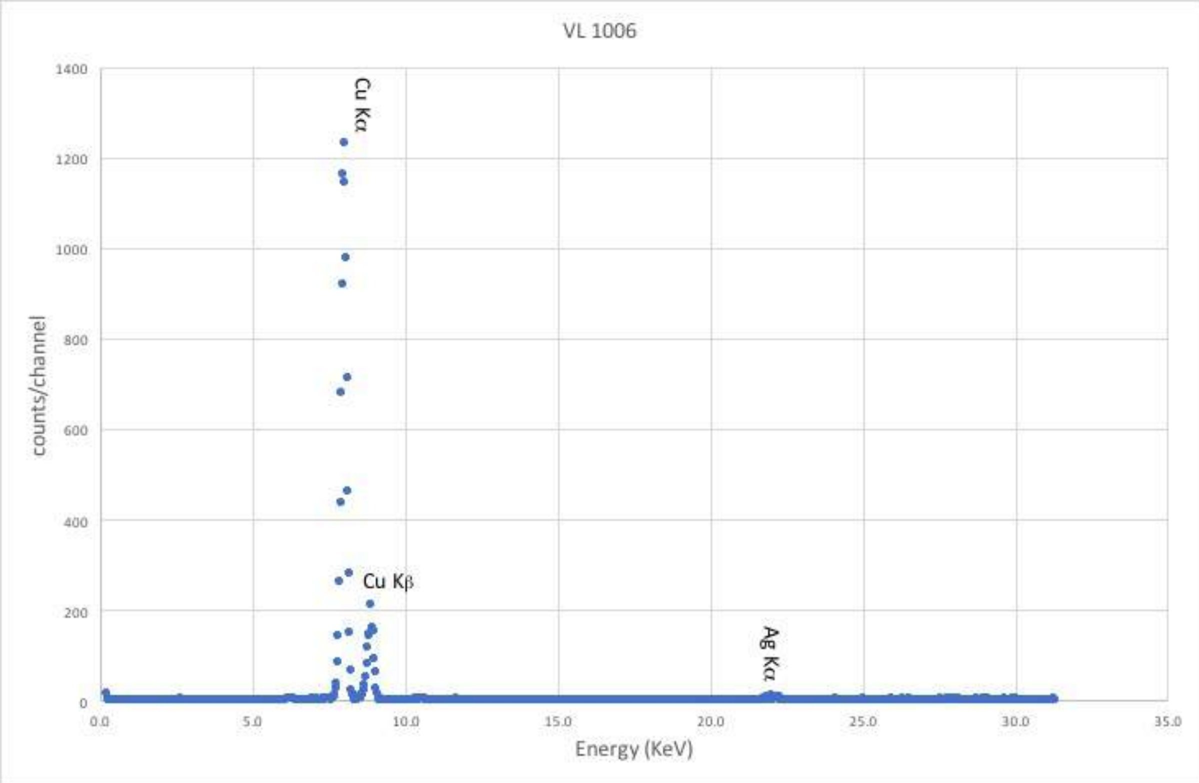

Figure 127: VL1006 XRF spectra.

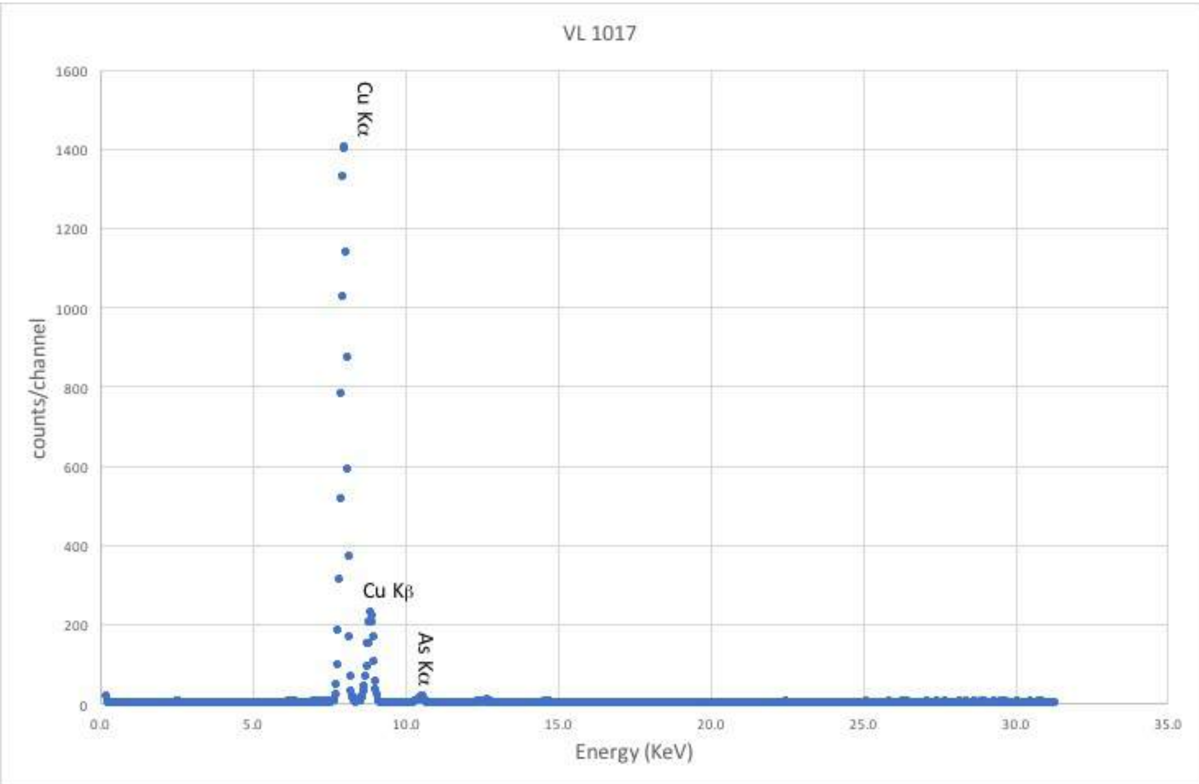

Figure 128: VL1017 XRF spectra.

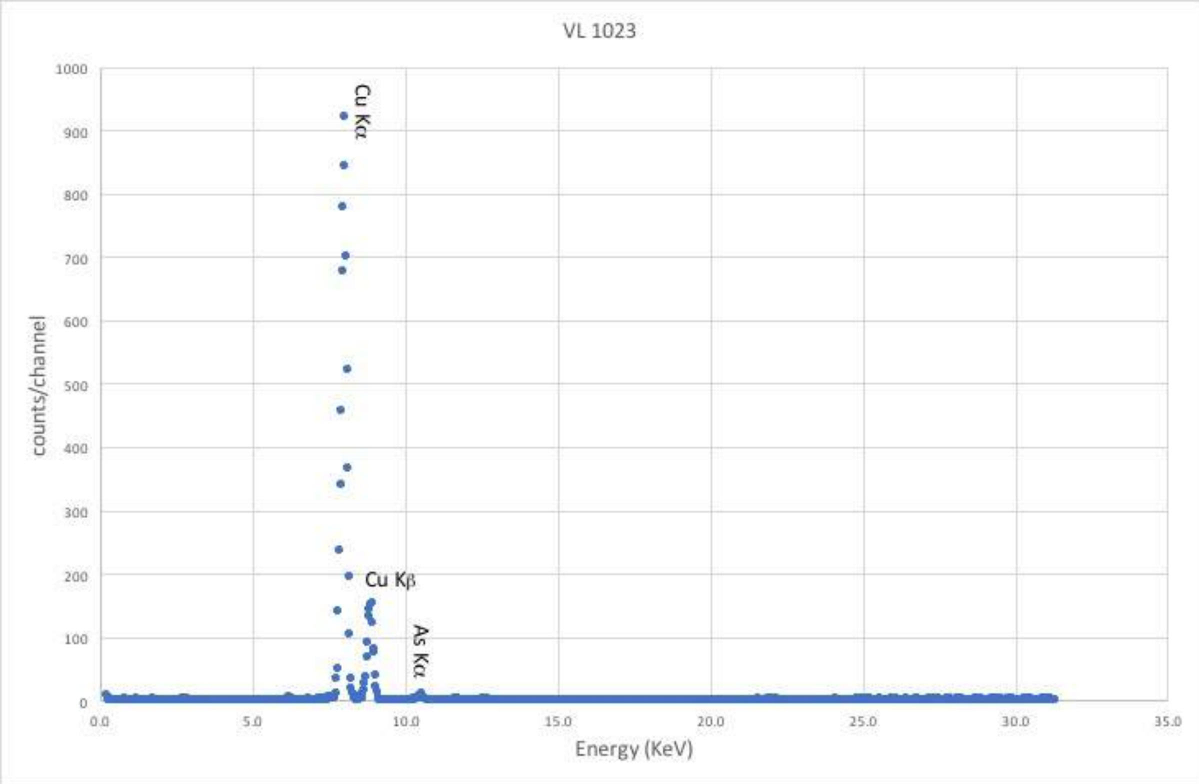

Figure 129: VL1023 XRF spectra.

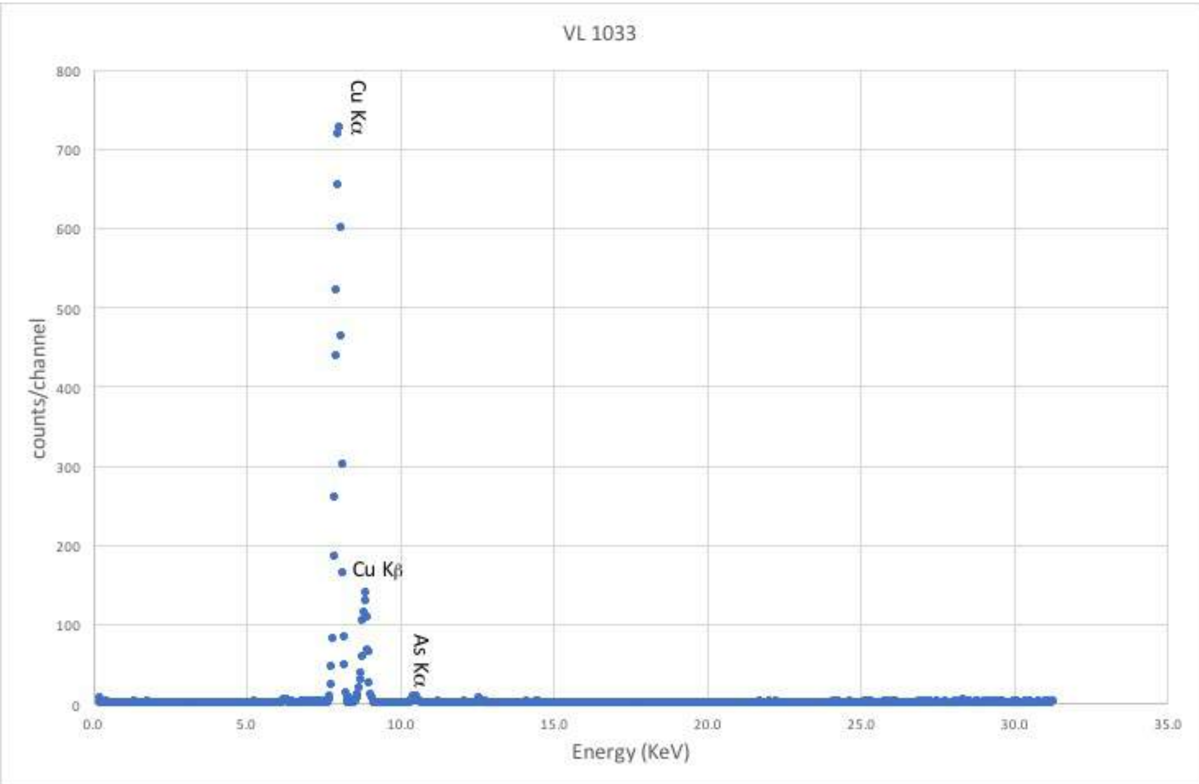

Figure 130: VL1033 XRF spectra.

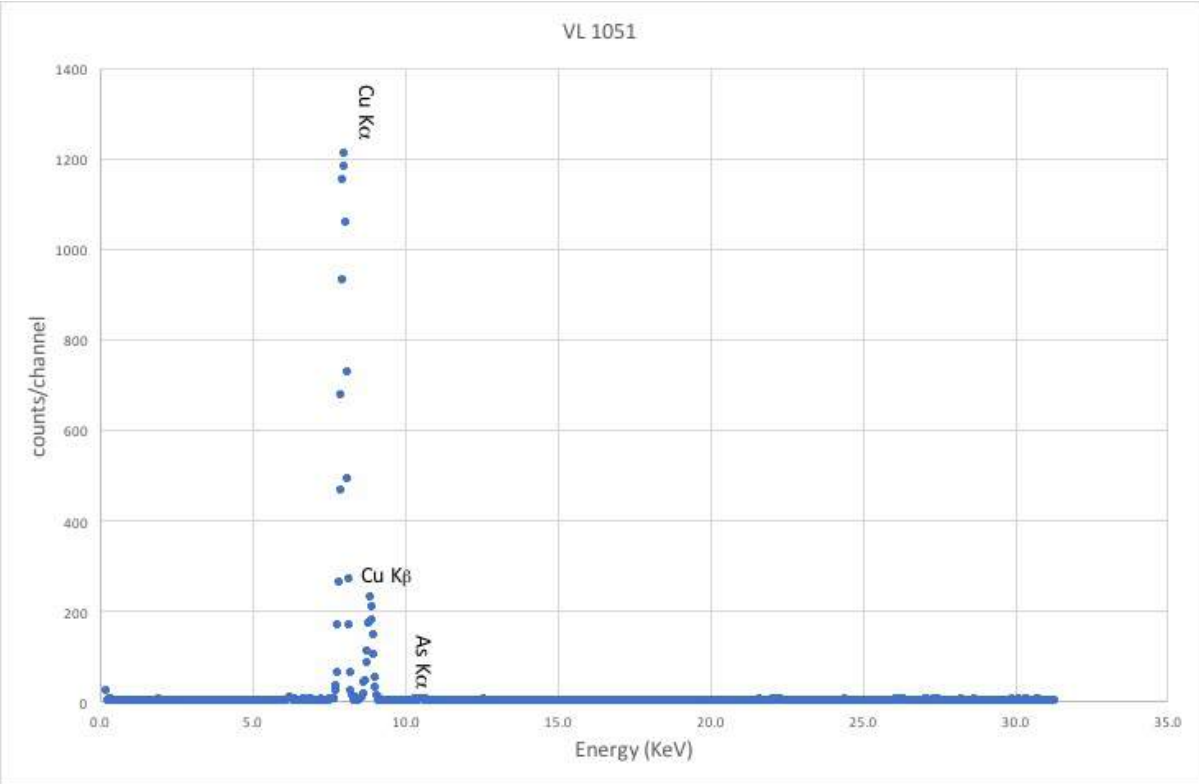

Figure 131: VL1051 XRF spectra.

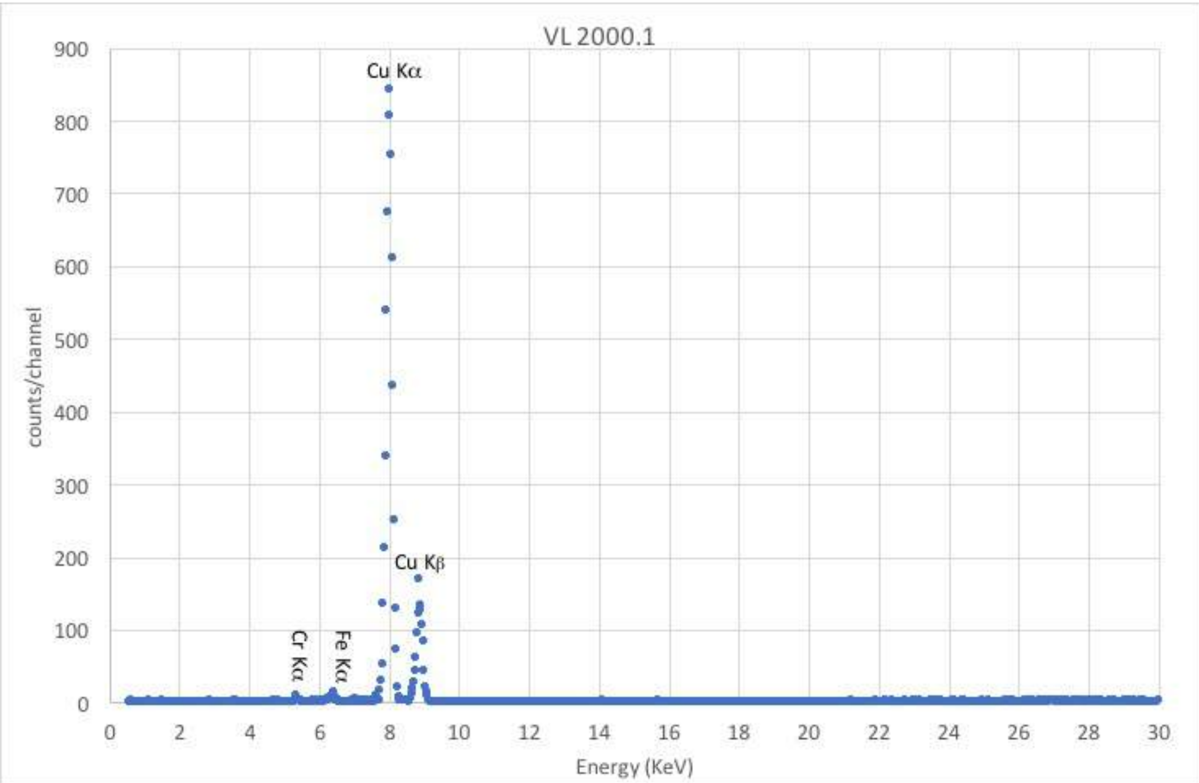

Figure 132: VL2000.1 XRF spectra.

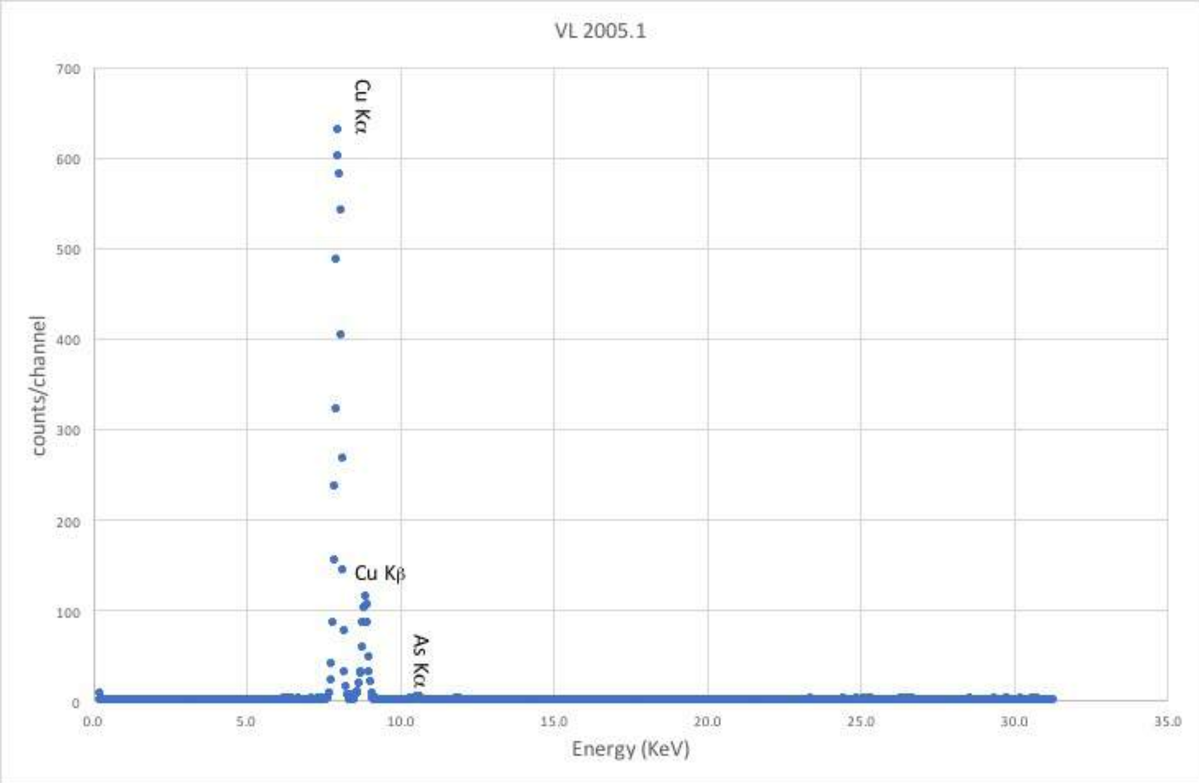

Figure 133: VL2005.1 XRF spectra.

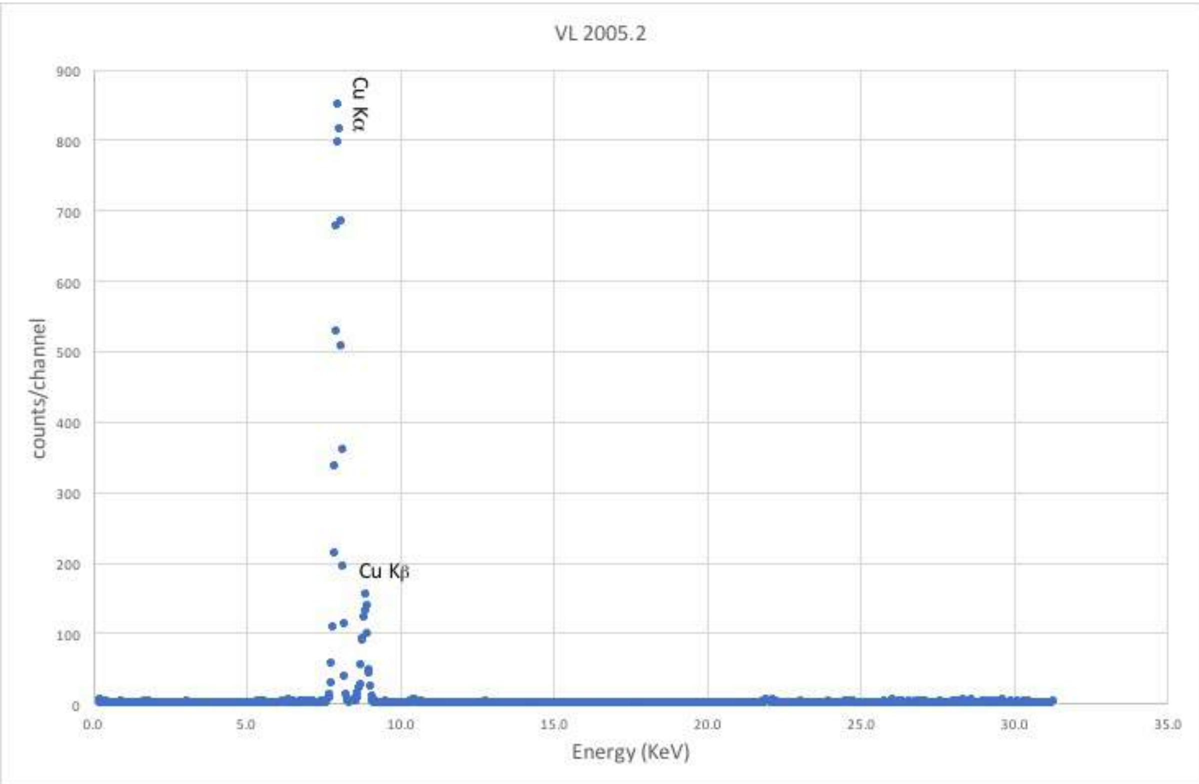

Figure 134: VL2005.2 XRF spectra.

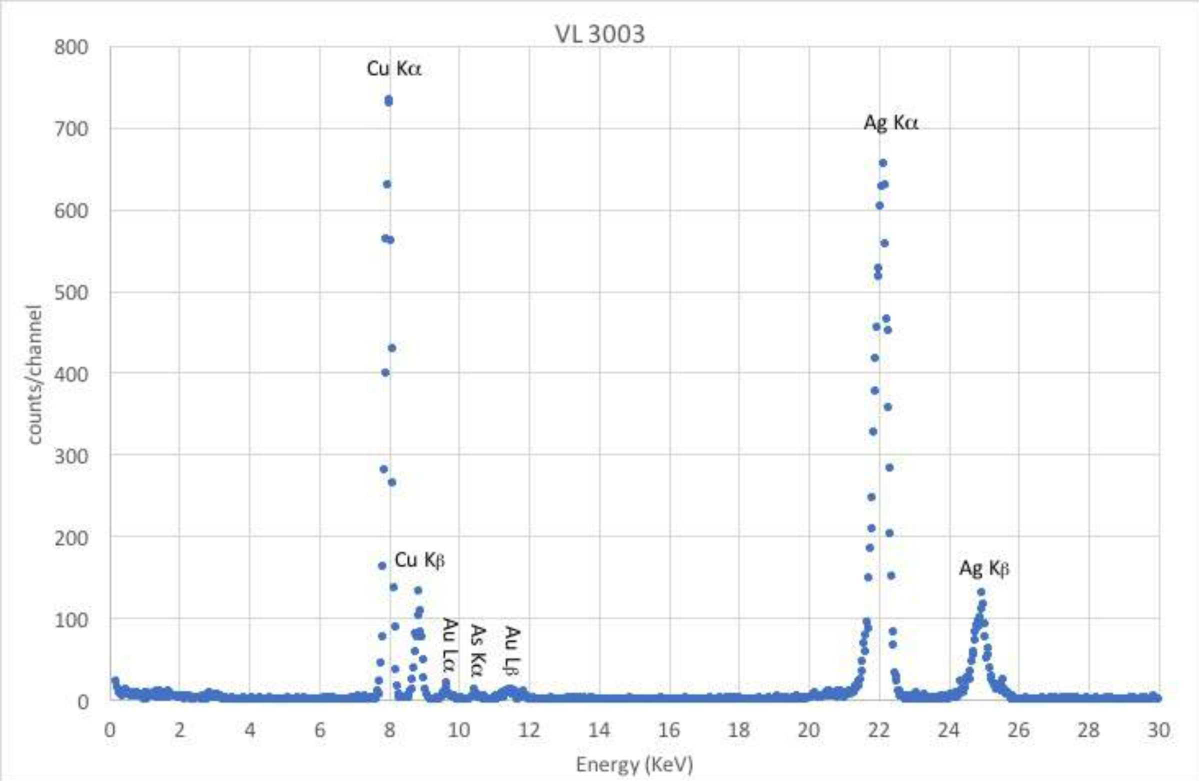

Figure 135: VL3003 XRF spectra.

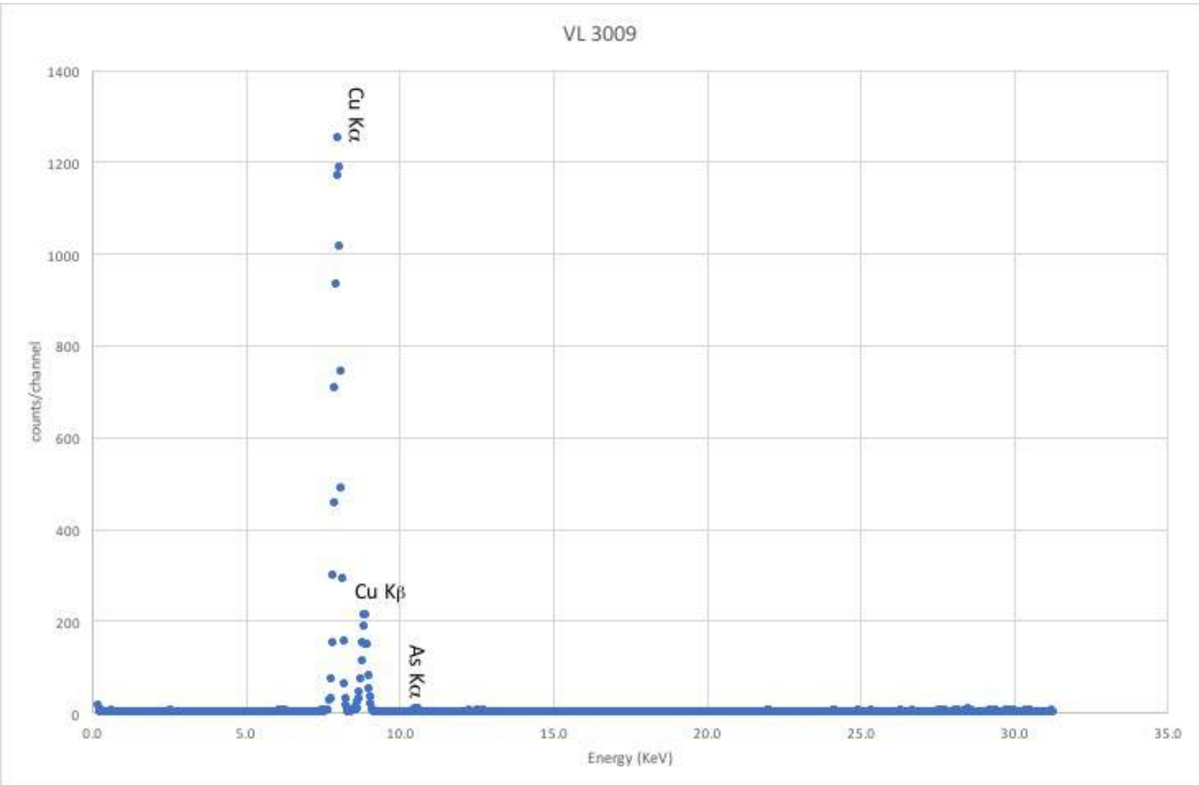

Figure 136: VL3009 XRF spectra.