The Uruk Expansion as Dynamic Process: A Reconstruction of Middle to Late Uruk Exchange Patterns From Bulk Stable Isotope Analyses of Bitumen Artifacts.

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Key words: Bitumen; Uruk expansion; Stable Isotope Analyses; Hacinebi Tepe; Tell Brak; Sheikh Hassan; Jerablus-Tahtani; Godin Tepe; Habuba Kabira; Anatolia; Uruk Mesopotamia; Ancient Trade and Exchange; Trade Routes; Natural Asphalt; Trade Goods;

Abstract:

This research examines broad regional patterns of inter-regional trade for the world's first colonial trading system, the economic expansion of southern Mesopotamia into southwest Iran and southeast Anatolia. Stable carbon and deuterium isotope analyses of bitumen artifacts from several Uruk enclaves and colonies show diachronic changes in trade routes as well as changes in the nature of the Uruk expansion from the Middle to Late Uruk periods. Hacinebi Tepe, a Chalcolithic Anatolian site located on the upper Euphrates River had material primarily from northern Mesopotamia in the period before the Uruk expansion. These findings

highlight the importance of sites such as Tell Brak and Hamoukar which reached a high level of complexity in the early fourth millennium BC and influenced societies prior to trade with southern Mesopotamia. In the late Middle Uruk Period, during the Uruk expansion, bitumen found in trade colonies in the northern Euphrates regions derived mainly from central and southern Mesopotamia and an unidentified source area, while later colonies from the Late Uruk period acquired most of their material from the northern Mesopotamian sources. The significantly large quantity of bitumen from the sources near Hit and Khuzistan at the site of Hacinebi during Uruk contact suggests a high volume of trade in this area. The changes over the course of the Uruk expansion demonstrate changing emphases in the upper Euphrates from riverine trade routes oriented north-south back to overland trade routes running east-west. These changes are consistent with the idea that Late Uruk colonies were focused on settlement and colonization rather than exchange. The preliminary results of these analyses demonstrate the utility of bulk isotopic analyses as a first step in the identification of broad regional patterns, which then can be bolstered with detailed isotopic and molecular work on asphaltene extractions of bitumen (Connan and Nishiaki, 2003).

Introduction

This collaborative project focuses on scientific analyses of bitumen (natural petroleum) artifacts from southeast Anatolia, northern Syria, and Mesopotamia to reconstruct the exchange patterns of the Uruk expansion, the world's first colonial

trading system. During the Uruk period (3600-3100 BC), the world's first states and urban centers appeared in the southern alluvium of Iraq and southwestern Iran, (Nissen, 1988; Pollock, 1992) and saw appearance of early administrative/writing systems, social stratification, kingship, warfare, and other key elements of early states (Algaze 2001; Nissen, 1988; Pollock, 1992; Stein, 1999). While arable land, livestock and bitumen were plentiful, vital resources such as metals, stone, and timber were scarce (Algaze, 1989, 2008), necessitating intensive trade with neighboring regions (Algaze, 1989, 2005, 2008). The presence of Uruk style artifacts and architecture in distant areas of southeast Turkey, Syria and Iran during the late Middle and Late Uruk Periods, has been interpreted to be evidence of trading colonies (Algaze, 1989, 2005, 2008). Spanning an area of approximately 5500 km², this trading system, known as the Uruk expansion is regarded by many as the world's first colonial trading network (Algaze, 1989, 2008; Stein, 1999). Based on the ecology of the southern alluvium, many have hypothesized that Mesopotamian exports were grain, textiles and even bitumen (Algaze, 1989, 2008; Stein, 1999).

The evidence for Mesopotamian trading colonies in the areas of Anatolia, Syria and Iran usually include architectural remains, Uruk ceramics, iconography and administrative artifacts of southern origin (Algaze, 2001:38). Some sites contain what appear to be Mesopotamian enclaves or residential quarters established inside a local Late Chalcolithic settlement (Weiss and Young, 1975; Young, 1986; Algaze, 1989; Stein 2002, 1999). Indigenous settlements in Anatolia and northern Mesopotamia (Algaze,

1986, Algaze et al., 1990; Palmieri and Frangipane, 1986; Gibson and Maktash, 2000), show varying degrees of contact with Mesopotamia (Stein, 2012,1999).

Many colonies, except for Godin Tepe in the highlands of Iran, and Tell Brak and Nineveh in northern Mesopotamia, were located on the Upper Euphrates River clustered in north Syria and southeast Turkey (Algaze 1986, 1989, 2005, 2008). The first group was located at a bend in the Euphrates River in the Tabqa Dam salvage area and includes Habuba Kabira South/Tell Qannas, Tell el-Hajj, Jebel Aruda, and Hadidi on the west bank, and Mureybit, Sheikh Hassan and Tannira on the east bank (Schwartz 2001; Boese 1995:171). The second cluster lies to the north in the Tishrin region with the site of Carchemish as the center, in addition to Jerablus Tahtani (Peltenburg et al. 1997, 1996,1995) and Tell 'Abr (Hammade et al. 2006).

Algaze believes that the strong competition between polities in Mesopotamia led to individual Uruk city-states founding their own specific enclaves or outposts to command the critical lines of communication (Algaze 1989, 2008). These colonies were uniformly positioned in a dendritic spatial pattern to effectively control exchange routes but were not designed to control large territories or exploit local agricultural resources. These Mesopotamian colonists and traders naturally gravitated towards preexisting local sites because they were already important indigenous regional centers (Algaze 1989, 2008; Pollock 1992).

The apparent lack of trade goods in archaeological contexts is seen to be problematic (Pollock, 1992, 1994), suggesting that Mesopotamian style artifacts in Anatolia, Syria and highland Iran could have been local imitations and not the result of

trade (Kohl, 1989; Wattenmaker, 1990). Others have seen the presence of Mesopotamian material culture in Anatolia not as evidence for trading colonies, but of displaced people from the southern centers in the Late Uruk Period (Johnson, 1989) establishing large agricultural and pastoral settlements (Schwartz, 2001). Originally, it was believed that the Uruk expansion was a product of the Late Uruk Period and a “rapid and short-term phenomenon” (Rothman 2001: 5); a view that has changed when archaeological evidence was amassed to the contrary. Revised radiocarbon dates show that the Uruk phenomenon lasted longer than previously thought (Wright and Rupley 2001). The School of American Research Advanced Seminar Series successfully integrated this information into the chronology of sites of the fourth millennium and created a new chronology of 5 periods of the Late Chalcolithic (Rothman 2001: 5-9), which this research will refer to. Through examination of bitumen artifacts from Uruk colonies and enclaves from the Middle Uruk Period/LC4 (Sheikh Hassan and Hacinebi) and the Late Uruk Period/LC5 (Jerablus-Tahtani, Habuba Kabira, and Tell Brak), this research can determine how the nature of the Uruk expansion changed over time.

Many of the trade goods that were hypothesized to have been so important in this trading network--textiles from southern Mesopotamia and copper, stone, and timber from Anatolia--are rare in archaeological deposits. Studies of bitumen, a naturally occurring petroleum tar, have great utility because this natural petroleum tar was abundant in the ancient Near East, was used for a wide range of purposes, was traded between different cultural groups, was present in large quantities at many archaeological sites, and is chemically sourceable using molecular and isotopic analyses.

Bitumen as a Marker for Ancient Exchange Patterns

Scientific analyses have made progress in the investigation of trade patterns of utilitarian and prestige goods in the Uruk expansion (Badler et. al., 1996; Blackman, 1984). Analyses of local Uruk period exchange patterns in southeast Turkey using Neutron Activation Analysis (INAA) of pottery and clay administrative sealings proved successful in documenting Anatolian trade with Mesopotamia (Blackman, 1999; Evins, 1998), but sealings are not typically found in large quantities in archaeological sites.

Bitumen, a term used by archaeologists for the natural petroleum tar available from a variety of seepages in the Near East (Figure 1), was utilized primarily for its waterproofing and adhesive properties. There is widespread evidence of bitumen's use as mortar in the construction of important buildings throughout the southern alluvium whose effectiveness is evident from ancient walls in Iraq that remain iron hard even today (Forbes 1936). This substance was collected at ground seepages, in lakes, or from rock outcrops. Bitumen source areas are ubiquitous in Mesopotamia proper and the region of Hit, Ramadi and Abu Jir in central Mesopotamia is believed to have been one of the main sources of bitumen for Uruk city-states (Forbes, 1964). Other significant sources exist in the Deh Luran and Susiana Plains of southwest Iran at the juncture of the Zagros Mountains and the Tigris-Euphrates alluvium. There were also major seepages near Kirkuk and Mosul, in northern Mesopotamia (Marschner et al., 1978; Connan and Van de Velde 2010), a source on the Mediterranean coast of Syria near the modern city of Latakia, significant seepages in the area of the Dead Sea and sources in the very

southern part of Mesopotamia used by the people of Ur (Connan et al., 1999). The presence of material from Bichri in northwest Syria has been documented with molecular analyses for a number of nearby archaeological sites (Hauck et al. 2013; Boëda et al. 2008). In addition, the Burgan source in Kuwait was identified among Ubaid period artifacts at the near by site of H3/as-Sabiyah (Connan et al. 2005; Connan and Carter 2007). The primary sources for the Near East (Figure 2) in general however, were the seepages in central Mesopotamia (Hit), northern Mesopotamia (Magda) and southwestern Iran (Khuzistan) (Van de Velde 2015). There are some indications that bitumen from Hit was purer and of higher quality than the bitumen from northern Mesopotamia (Connan and Van de Velde 2010).

The existence of different bitumen sources and seepages is known through textual records, archaeological evidence, and modern geologic survey. Ancient texts often mention a bitumen source that has not been identified by modern survey and occasionally geologic surveys identify seepages that do not seem to have been used in antiquity. A bitumen well between Kufa and Basra (Forbes 1936) and a pool of bitumen near Samarra (Peters 1897) have yet to be discovered. The abundance of bitumen at Ur would seem to suggest that there was a nearby source and a Sumerian scribe from the Ur III period mentions a source at the nearby site of Eridu (Ferrara 1973).

Archaeological evidence for bitumen processing in Near Eastern sites includes pottery with bitumen dribbles on the inside and/or the outside, and pieces that appear to have been the result of cooled, solidified spills (Figure 3). Ethnographic research

provides a wealth of data on bitumen processing and demonstrates that, besides using containers to heat bitumen, people in southern Iraq would also use a trough dug into the mud at the side of a canal. Igniting a fire in a hole beneath the trough would supply the necessary heat to liquify the bitumen (Ochsenschlager 1992). Written evidence from the Ur III period reveals that the price of mastic (processed bitumen mixed with mineral and organic inclusions) was considerably higher than raw bitumen because of the fuel consumed in the production process. Rock asphalt from outcrops has a much higher melting temperature than liquid bitumen from seepages, which may have been another reason why the people of southern Mesopotamia mainly used the latter kind for their projects (Forbes 1936).

Bitumen artifacts are numerous at many southern Mesopotamian sites in addition to Uruk colonies and enclaves. During the Uruk period bitumen was used primarily in Mesopotamia as: (1) a waterproofing material for pottery, reed matting, baskets and boats (Gregg et al. 2007; Carter 2002); (2) a mastic used in the production of art objects; (3) a mortar for bricks; (4) a pigment for colored wall cones in public buildings; (5) and a hafting for flint blades onto sickle handles (Forbes, 1964: 56-109; Harper et al., 1992; Ochsenschlager, 1992). In addition, it may have been an exchange item itself (Algaze 2008) as well as packaging material for other commodities (Figure 3 and 4). It is hypothesized that Mesopotamians used bitumen on jars, pots, baskets and reed mats to help transport other items (Stein et al. 1996). Furthermore, because different bitumen seepages and deposits have different geologic histories, one can estimate the possible source of individual bitumen artifacts using advanced geochemical techniques (Connan et

al. 1999, Connan and Deschesne, 1995, 1996, 1998). In this respect, bitumen waterproofing can serve as a proxy for archaeologically absent exchange items in sites associated with the Uruk expansion. However caution and a suitably large data set must be employed due to several examples of bitumen samples at archaeological sites not deriving from the closest source but rather from sources further away (Gregg et al. 2007; Van de Velde et al. 2015).

Bitumen was also an item of exchange both internally in Mesopotamia and externally with communities in Anatolia. Finds of bitumen blocks (Pollock, 1990) and textual records from later periods (Moorey 1994) indicate that Mesopotamians traded bitumen as a utilitarian commodity in the periods after the Uruk expansion if not earlier. Ancient texts suggest that exploitation at the seepages was on an industrial scale during many periods of Mesopotamian history; for example the town of Girsu (Lagash) paid 290,000 kg of bitumen to the king of Ur to be used ‘for the house of the grand vizier’. Other sources mention five shiploads of bitumen being sent down to Ur (Singer et al. 1954). Early European explorers from Rawlinson to Gertrude Bell marveled at the sheer quantity of material that bubbled up from the numerous springs in the town of Hit (Peters 1897; Bell 1911).

Available Data Set

The analyses presented here were part of a larger research project focused on the economic impact of the Uruk expansion on the local community at the site of Hacinebi Tepe in southeastern Turkey. Over four hundred pieces of bitumen from the site of

Hacinebi were compared to comparative material from Near Eastern sources and archaeological sites. Some bitumen was probably brought to Hacinebi in blocks, and then melted down at the site for specific uses by the residents (Stein et al. 1996) evidenced by bitumen residues on the broken surfaces of sherds and residues found in lip spouts of conical cups (Stein et al. 1996). Economic stages of bitumen artifacts from production to finished products are represented at the site of Hacinebi. In addition analyses were performed on Uruk period bitumen artifacts from several colonies and enclaves (Table 1) and these results are the focus of this paper.

If bitumen source samples were not available, bitumen samples from nearby archaeological sites were used as proxies. Material from Kish was used as a proxy for the sources at Hit, Ramadi and Abu Jir based on Kish's proximity to these sources and later historical records describing Hit as a major bitumen source for the site of Kish (Moorey, 1994). The samples from Kish were removed from large mortar deposits on baked clay bricks from the Neo-Babylonian period and since these deposits were so large and were highly utilitarian in function, it is likely that they were obtained from the closest bitumen sources to Kish. Moreover, texts from the period of Nebuchadnezzar identify Hit (Is) as the source for the bitumen used in the docks at the city of Babylon (Peters 1897). Following this example, most of the major ancient source areas were represented in the study (Table 1). With the use of numerous archaeological proxies one could be certain of having material from a source that had actually been used in antiquity. Furthermore, the use of several proxy samples that underwent the same weathering processes as other

archaeological samples represented more accurately the isotopic variability within a source region.

Methods

The development of isotopic and molecular analyses made it possible to identify possible geologic sources for archaeological bitumens, first in the Near East, (Connan et al. 1999, Connan and Deschesne, 1995) and then in the New World (Wendt and Lu, 2006). Archaeologists who have studied obsidian artifacts have realized that it is sometimes preferable to trade accuracy for a larger, more representative sample of artifacts. For example, in the analysis of obsidian from the Neolithic Italian site of Filiestru, an early study of 4 samples yielded the following results for the sources used: 50% SA, 25% SB, 25% SC. A later analysis using 86 samples gave vastly different results: 10% SA, 49% SB, 41% SC (Tykot 2003).

Obsidian is the most reliable substance to source with archaeometric methods. Obsidian sources are limited to a few regions, different sources are chemically distinct from each other, and the rapid cooling and crystallization of lava insures homogeneity in the source material. In addition ancient humans could not chemically alter obsidian (Clark 1981; Glascock 1998). Bitumen artifacts can contain mixtures of sources, making sourcing issues more complicated (Connan and Van De Velde, 2010).

In addition some bitumen seepages known today may not have been used in antiquity, some sources exploited by humans in the past may be absent or still unidentified, and bitumen at a site may have come from further away than the nearest

source (Connan and Van De Velde, 2010; Gregg et al. 2007; Van de Velde 2015; Van de Velde et al. 2015). In southeastern Turkey at the site of Demirköy (one of the archaeological proxies also employed in this study), analyses of bitumen artifacts and material from the nearby oil seep (Boğazköy) demonstrate Silurian origins for both, but molecular data were unable to conclusively show that the artifacts derived from this source (Connan et al. 2006). The analysis of bitumen is complex and detailed study requires great familiarity with petroleum geochemistry (Connan and Van De Velde, 2010). Even with molecular correlations there is still a certain level of uncertainty in source identifications (Connan and Van De Velde, 2010). Weathering effects on archaeological samples may also complicate attempts to draw conclusions of source origin based on chemical similarity to modern samples (Connan et al. 2006). The use of archaeological proxy material allows for comparisons of material that likely had the same weathering effects on their respective chemical compositions.

Bitumen samples from Mesopotamian and Anatolian contexts of Hacinebi as well as samples from source areas and archaeological sites near source areas were analyzed initially for their bulk carbon isotopic composition (Schwartz and Hollander 2008). A select group of samples were tested further with molecular organic geochemical characterization including compound-specific-isotopic-analyses, allowing for more precise comparisons (Schwartz et al., 1999; Schwartz and Hollander 2008). Two samples with similar stable carbon isotope ratios were from Uruk contexts at Hacinebi, and material from the site of Kish (serving as a proxy for the sources at

Hit). Two other samples were from Hacinebi and dated to the era of pre-Uruk contact (Phase A) and the period during contact with Uruk Mesopotamians (Phase B2).

Several biomarkers, specifically types of alkanes, were analyzed: (1) normal-alkanes (n-alkane); (2) isoalkanes (branched alkane or iso- prenoid); and (3) cyclohexanes (cyclic alkane). Extraction of lipids followed the modified procedure developed by Wakeham and Pease (1992). Isotopic compositions of individual compounds were determined on Fison ISOCHROM-GC (GC 8000 Series 8130) and VG Optima Mass Spectrometer.

Neither these intensive methods nor isotope analysis on asphaltene extractions (Connan and Nishiaki, 2003), could efficiently be employed on the entire assemblage, and an approximate measure of source was employed through whole sample bulk stable isotope analyses (Schwartz and Hollander 2008). The project involved the analysis of over four hundred samples from archaeological source proxies and Uruk period sites in southeast Turkey, northern Syria, southwest Iran and southern Iraq to create a representative sample size, crucial for getting beyond simply the identification of trade to examine patterns of exchange. This project represents the largest number of bitumen samples analyzed from one site (423 samples from Hacinebi Tepe). The “interpretive limitations imposed by small data sets” in examining broad regional and temporal patterns of resource procurement can only counterbalanced with a sufficient number of samples (Tykot, 2003).

The bitumen artifacts and source samples were analyzed through a combination of stable carbon and deuterium isotope analyses employing a VG Optima

Mass Spectrometer and High Temperature Elemental Analyzer (College of Marine Science at the University of South Florida). With the High Temperature Elemental Analyzer (TCEA), each sample was pyrolyzed in 1400 degrees Celsius anoxic conditions, producing pure hydrogen gas, which could then be sent directly to the mass spectrometer, allowing faster processing than standard offline preparation (Schimmelmann and DeNiro, 1993). All of the isotope ratios were calibrated according to recognized standards, n-36 Alkane for Deuterium analyses and Urea for stable carbon isotope analyses. Samples were prepared by removing contaminated outer surfaces, pulverization and removal of visible elements such as chaff and limestone.

Artifacts were assigned typological designations based on visible impressions. Bitumen bearing traces of matting or baskets may have been part of containers used in the packaging of other trade goods. Archaeological evidence from Tell Brak attests to the use of woven reeds to package or carry caches of precious stones (Emberling and McDonald, 2002). Bricks of bitumen with mat impressions were most likely ingots used as a unit of transport (Marschner et al. 1978; Wright 1969). There is also the possibility that waterproofed reed mats were used for roofing material (Van de Velde 2015). Bitumen on the interior of storage jars and juglets may have been used as waterproofing for the transportation of precious liquids but may also have resulted from the processing of bitumen. Deposits on the interior of bevel-rimmed bowls may have had a utilitarian function. Amorphous bitumen pieces, most likely the result of bitumen processing, were classified as production debris.

Previous Results

Gas Chromatography-Mass Spectrometry and Compound Specific Isotope

Analyses

Previously published molecular organic geochemical analyses (Schwartz et al. 1999; Schwartz and Hollander 2008) of branched and cyclic compounds (specifically three diagnostic hopanes) showed a correlation between an archaeological proxy sample from southern Mesopotamia (Kish) and an Uruk context (associated with Mesopotamian pottery) sample from Hacinebi that had similar whole sample stable carbon and deuterium isotope ratios (Schwartz and Hollander, 2008; Schwartz et al. 1999). One Pre-Contact (Phase A) and one Local Contact sample (Period B2 and associated with Anatolian pottery from the site) selected for these analyses were distinct from the Kish and Uruk samples, as well as from each other (Figure 5).

In this suite of analyses, the most abundant diagnostic compounds of the Kish and Hacinebi samples were used for the determination of isotopic compositions. Cross-plot (Figure 6) of the $\delta^{13}\text{C}$ values of 28-Nor-17 β H-hopane and 7 α H-21 β H-hopane shows differences between the Kish /Uruk group and the Local samples on both axes. Thus, the sample from Mesopotamian contexts at Hacinebi correlates strongly with the sample from Southern Mesopotamia on two separate, highly characteristic planes and the sample that is significantly similar to a sample from central Mesopotamia is also chemically distinct from Local Hacinebi samples (Schwartz et al. 1999; Schwartz and Hollander 2008).

Bulk Stable Isotope Analyses

Bulk isotope analyses on whole samples were performed and source groups were estimated. Initial visual determinations of bivariate plots had ninety percent confidence interval ellipses constructed around the greatest cluster of points to account for the density of points and the slope of the regression line through these points--the standard method for creating source groups in obsidian studies and metallurgical analyses (Glascocock et al., 1998).

Bivariate confidence intervals provide a way of quantifying perceived differences between different geologic samples and/or archaeological proxies. While there was overlap, there was distinction between material from southern Mesopotamian archaeological sites used as source proxies, and material from the Anatolian site of Hacinebi in purely Local contexts (Figure 7). In general, bitumen artifacts from Local contexts at Hacinebi are isotopically distinct from bitumen from central and southern Mesopotamian archaeological source proxies. This Mesopotamian source group is composed of seventy-five samples from nine sites from both southern Iraq and southwestern Iran, many of them from contemporaneous levels (Table 1). Some of the materials (Kish, Sakhieri Sughir, Ur for example) were from later periods. This material seems to form a well-defined group with only a few outliers, (outliers defined as “specimens that chemically differ from all others and have a disruptive influence on the shapes of sample distributions,” Glascocock et al., 1998: 25). These samples were then compared to modern source material and archaeological proxies from northern Mesopotamia and southeastern Anatolia (Figure

8). These added samples further confirm the initial group designations. The source group associated with southern and central Mesopotamian sites overlaps to a degree with modern source material from southwestern Iran.

Source proxies near the northern Mesopotamian/Magda sources (specifically Nuzi) fall in the isotopic range of the first source group from Local contexts at Hacinebi. There is also possibly a second southern Mesopotamian source group with one archaeological sample and one source sample from Masjid-i-Suleiman. Two samples from Hacinebi are similar to an archaeological sample from the site of Demirköy in southeastern Anatolia.

Ninety percent confidence ellipses were then constructed around these hypothesized source groupings (Figure 9). There appear to be three different source groups represented in Local Hacinebi contexts that are discrete from the two southern Mesopotamian source groups. The isotopic discrimination between these source groups correlate in general with archaeological context. The source group with the greatest number of samples contained material from the archaeological site of Lidar less than 100 kilometers upstream from Hacinebi. There was also a sample from Kenan Tepe and three samples from the Late Bronze Age site of Nuzi. Of the remaining archaeological samples, only Nuzi is near a documented ancient bitumen source in northern Mesopotamia near modern Kirkuk and known as Magda in the cuneiform texts (Moorey 1994). It is most likely that this source group represents material from northern Iraq and the bitumen samples found in Lidar and Kenan Tepe were derived originally from this source. The second source group from Local

Hacinebi contexts had an isotopic signature that between the range of the central and southern Mesopotamian source proxies and the modern source sample from Masjid-i-Suleiman. There is unfortunately too little evidence from our study to speculate as to the origin of this source group. To examine the changes that occurred from the Middle Uruk to the Late Uruk period, the study derived rough approximation of source origin for artifacts found in the Middle Uruk sites of Sheikh Hassan and Hacinebi, and the Late Uruk sites of Jerablus-Tahtani, Habuba-Kabira, Tel Brak and Godin Tepe.

Middle Uruk Period Sites

Hacinebi Tepe (n=423)

The 3.3 ha. fortified, local Late Chalcolithic Anatolian settlement was positioned on the Euphrates river at a historic east-west crossing point (Dilleman 1962: 135; Oates 1968: 7; Wagner 1976) and at the head of the main north-south trade route in this region (Chesney 1850: 45; Great Britain 1916: 167; Idrisi 1840: vol.II:137). The earliest occupations at the site (Phase A ca. 4100-3800 BC and B1 ca. 3800-3700 BC) date to the early fourth millennium BC, before the Uruk expansion. The later phase (Phase B2 ca. 3700-3300 BC) consists mainly in a continuation of this local occupation with what appears to be a small Mesopotamian enclave (Stein, 1999; Stein et al. 1996; Stein and Misir 1994). The existence of this enclave was based not merely from the presence of foreign style pottery, but a variety of forms of material culture that document concentrated Mesopotamian activity in the northeast area of the site (Stein 1999). The full-range of Uruk ceramic forms including cooking, serving and storage vessels are

present at Hacinebi and kiln-wasters indicate on-site production. Wall-cones, a distinctly Mesopotamian wall decoration, attest to the presence of Uruk architecture and Mesopotamian style administrative artifacts, baked clay sickles, and grooved stone weights also appear at the site (Stein 1999; Stein 2002).

Results:

Bitumen artifacts from before contact with Mesopotamians (Phase A and B1 LC 1,2,3) seemed to have derived mainly from sources in northern Mesopotamia (Figure 10), which may demonstrate the connection of the site to the east-west overland trade route running from the Mediterranean along the Sinjar to the foothills of the Zagros mountains (Algaze 2008). One deposit of bitumen artifacts at Hacinebi was distinct from others at the site due to the exceedingly high concentration of material in a relatively circumscribed area. This enormous deposit (Operation 7 production area), dating to the period of Uruk contact, was associated with a monumental enclosure wall in the southern area of the site and has evidence of intensive bitumen use and production. In this area over 15,000 grams of bitumen was recovered, more bitumen than found in the rest of the site during all periods, combined (Schwartz and Hollander 2008).

In this production area dating to the period of contact with Mesopotamian merchants, very few of the bitumen artifacts were from the northern Mesopotamian source that was the primary source in earlier phases of the site but instead over half derived from an unidentified source (Schwartz and Hollander 2008). Approximately

6.78% or an estimated 1,031.05 grams out of the total 15,207.3 grams was from northern Mesopotamia as compared to 54.55% or 8,295.58 grams of melted or processed bitumen coming from the unidentified source (Figure 11).

While this production area is very different in terms of the sources present as compared to A/B1 (“Pre-Contact”) Local contexts, it is also distinct from other B2 (“Contact”) purely Local contexts because of the large amount of bitumen from Central Mesopotamian or Khuzistan sources. What is most interesting is that bitumen derived specifically from these source regions is found either in the production area of Hacinebi, or Uruk contexts (Figure 12), and not in Local contexts. The significance of this circumscribed distribution is not entirely clear. However the great amount of bitumen in the production area probably indicates a marked increase in the volume of trade goods both being imported from southern Mesopotamia and exported from the site (Schwartz and Hollander 2008).

Sheikh Hassan (n=10)

Sheikh Hassan was a relatively small (1 hectare) Mesopotamian colony dating to the Middle Uruk period and contained a long sequence of Uruk colonial occupation with domestic and specialized architecture. The city wall with niches and towers was similar to that of its neighbor Habuba Kabira, (Boese 1987, 1995; Schwartz 2001). Sheikh Hassan is unique for its bitumen storerooms containing stacks of mat impressed ingots similar to those of Hacinebi (Boese 1987, 1995). Warehouses or storage areas for bitumen have been noted at other archaeological sites from at least the Ubaid period (Van de Velde 2015). In this respect, this site is particularly

important because it offers the possibility of analyzing a bitumen assemblage for an Uruk colony with no local inhabitants.

Results:

All of the samples analyzed from this site were pieces of mat-impressed bitumen similar to ones found at Haccinebi. Their isotopic signatures values (Figure 13) suggest a majority of the samples from this Middle Uruk site derived from the central and southern Mesopotamian seepages. This is in stark contrast to the results from the Late Uruk colonies, Jerablus-Tahtani and Habuba Kabira.

Late Uruk Period Sites:

Godin Tepe (n=11)

Godin Tepe may represent a colony of a type similar to Haccinebi; an enclave of Uruk settlers within a pre-existing local community. While Haccinebi is located in the upper Euphrates region, Godin is also strategically located, but in the highlands of Iran on the historical Khorasan Road that ran from southern Mesopotamia to the Iranian Plateau (Algaze 2008; Rothman 2013). Uruk Mesopotamian material (Godin VI:1) was found within an oval enclosure inside of the local settlement (Young 1986; Gopnik and Rothman 2011). Like Haccinebi, a local community had existed before Mesopotamian contact. The fort-like structure on top of the site had a range of Uruk Mesopotamian ceramics, in addition to tablets and cylinder seal impressions that included iconographic signs found in the earliest Archaic Texts from Uruk-Warka (Algaze 2001; Young 1986). This Godin Period VI occupation was originally

interpreted to be a colonial outpost made up of a small number of merchants from Mesopotamia (Algaze 2001; Weiss and Young 1975). However ceramic data does not support the idea of a permanent occupation and instead suggests that Uruk pottery was manufactured by locals at the site (Rothman 2013). Contact with southern Mesopotamia is not debated however, and it seems likely that at the very least traders from the Uruk heartland were involved in some trade related activities here (Rothman 2013).

Results:

From the stable isotopic work on samples from Godin VI (Figure 14), one can see a broad distribution of values. Two samples seem to fall in the range of central and southern Mesopotamian values, and two samples are in the borderline area of values close to this source area. Two other samples are in an area that has not been sourced as of yet, but corresponds to values from samples found in Uruk Mesopotamian contexts at Hacinebi. It is possible these samples represent another source from Mesopotamia. For the most part the results from Godin Tepe are inconclusive. It is hoped that future work will allow discrimination between material from Hit and from Khuzistan.

Habuba Kabira (n=6)

Habuba Kabira is perhaps, “our best-documented example of an Uruk colonial emplacement,” (Schwartz 2001: 261). Not only is it a well-researched site with large horizontal exposures, but it is often pointed to as a model for the analysis of other Uruk colonies. The site was most likely an urban community between 18-40 hectares

with a population in the thousands, (Schwartz 2001; Strommenger 1980; Surenhagen 1986). The central mound contained a walled area of 10 hectares with a street and drainage system and impressive fortifications, demonstrating central planning (Schwartz 2001). The architecture of the site was distinctly southern Mesopotamian in style with tripartite plans and rimmed bricks (Surenhagen 1986).

Most of the samples from Habuba Kabira used in this study were not found on pottery. Some were remnants of production debris and others had reed mat impressions similar to the ones found at Hacinebi. From bulk stable isotope analyses (Figure 15), one can hypothesize that two samples derived from central or southern Mesopotamia and the remainder either were from northern Mesopotamia or an unidentified source.

Jerablus Tahtani (n=9)

Jerablus Tahtani was adjacent to the important ancient center of Carchemish and had a number of typical Uruk style artifacts dating to the Late Uruk period (Peltenburg et al. 1995, 1996, 1997). The Uruk levels rest on top of an earlier Local Late Chalcolithic settlement and one section of an Uruk household revealed evidence for bitumen processing as well as textile manufacturing. Most of the artifacts used in this study were bitumen residue samples on Uruk style pottery. In general, the bitumen at the site originated from the northern Mesopotamian sources near Kirkuk (Figure 16). One sample is likely to have been from southern Mesopotamia, while 2 samples may be from an unidentified source. This pattern of bitumen procurement is similar to the other Late Uruk site already discussed, Habuba Kabira.

Tell Brak (n=14)

Tell Brak appears to have been a local northern Mesopotamian settlement that had a significant Uruk Mesopotamian occupation during the Late Uruk period as evidenced by cylinder seals, ceramics and architecture (Emberling 2002; Emberling et al. 1999). Some believe this local center was conquered by Uruk Mesopotamians (Algaze 2008; Emberling 2002) while others believe Uruk material at the site was either the result of local elites imitating foreign styles to increase their status (Schwartz 1989; Wattenmaker 1990) or derived from an Uruk enclave similar to Godin and Hacinebi (Stein 1999).

All of the samples analysed were from area TW, which had clear evidence of an Uruk Mesopotamian occupation (Emberling 2002). The overall picture of Late Uruk Brak isotopic ratios (Figure 17) shows a pattern of use of the northern Mesopotamian sources south of Tell Brak in the Kirkuk/Mosul region with some material from central and southern Mesopotamian sources being very probable as well. There are at least five samples that are in the area of overlap between these two Mesopotamian sources and they could well be from the Hit region. Work by Connan and Van de Velde on bitumen samples from Brak suggest origins of Hit and Kirkuk sources, which are consistent with our findings (2010).

Discussion

The Uruk Expansion in a Macro-Regional and Diachronic Perspective

Based on the geochemical and archaeological data, a macro-regional picture of the Uruk expansion emerges. Some preliminary conclusions may be drawn from the

data, which need to be confirmed with detailed molecular analyses. The predominant source of bitumen at Hacinebi in the “Pre-Contact” phase was not of central or southern Mesopotamian origin but most likely from the Magda/Kirkuk sources, based on the isotopic overlap with bitumen from the northern Mesopotamian site of Nuzi. If this is the case it means that Hacinebi was already an active participant in the east-west overland trade route known from later times, connecting the northern Euphrates to the Mosul region (Algaze 2005: 42). One of the uses of bitumen brought into Hacinebi in this “Pre-Contact” phase was the waterproofing of reed boats (Schwartz 2002) and it seems likely that these boats were used predominantly for ferrying people and goods across the Euphrates as part of trade between northern Syria and the Mediterranean region. This is confirmed by the presence of marine shell (cowrie) beads in Hacinebi (Stein 1999) as well as the significance of the region as a crossing point throughout history (Algaze 2008).

These results further support the recent work that has demonstrated the high level of social and political development in northern Mesopotamia prior to the Uruk expansion. During this period in the early fourth millennium BC, communities in northern Syria had already reached advanced levels of social complexity with little evidence of influence from southern Mesopotamia (Stein 2012). Tell Brak appears to have been a large and complex settlement well before the Uruk expansion (during periods LC1, 2, 3) with a size of up to 100 ha (Emberling and McDonald 2002; McMahon 2013; Oates et al. 2007; Schwartz 2001; Ur et al. 2007). Starting the late fifth millennium/early fourth millennium BC Tell Brak developed many of the

hallmarks of a state society such as monumental architecture, high population density, long-distance trade, craft specialization, and powerful religious institutions (Oates et al. 2007; Ur 2010). Both Brak and other large northern Mesopotamian sites were involved in long distance trade in obsidian from eastern Anatolian sources (Khalidi et al. 2009). Hamoukar/Khirbat-al-Fakhar, while not as densely settled as the city of Uruk, was larger in size at 300 ha (Ur 2010). Hacinebi, while much smaller than these sites, was engaged in long-distance trade in copper, chlorite and cowrie shells, resources from regions to the north, east and west of the upper Euphrates (Stein 2012).

The bitumen evidence from Hacinebi supports the idea that the site and the region of the upper Euphrates were linked through trade to early urban societies in northern Mesopotamia. One could say that before the Uruk expansion, Hacinebi and northern Mesopotamia shared a cultural complex including similarities in pottery, iconography and material style evidenced by glyptic elements on stamp seals (Pittman 1999; Stein 2012), the presence of distinctive “eye idols” (Emberling 2002; Stein 2012) and the ubiquitous chaff tempered “hammerhead” bowls (Brustolon and Rova 2007; Pearce 2000). Interestingly there is evidence for bitumen from these northern Mesopotamian sources reaching as far as sites in the Persian Gulf in the Ubaid period (Van de Velde 2015; Van de Velde et al. 2015).

During the Middle Uruk Period, there is a sharp drop in the amount of bitumen brought in from the Magda/Kirkuk sources and a significant increase in bitumen brought up from central Mesopotamian sources, sources from southwestern Iran, and a yet unidentified source. During this “Contact” period, the amount of bitumen from

southern Mesopotamia greatly outweighs the quantity of bitumen from northern Mesopotamia. Chemical analyses of sealing clays at Hacinebi indicate Anatolian and Khuzistan sources for these artifacts, further demonstrating that goods were imported to the site from the south, and then other materials were packaged for export back to Mesopotamia (Blackman 1999; Stein 2012). Contemporaneously, at the Uruk enclave of Sheikh Hassan the sourceable bitumen samples also derived from southern Mesopotamia. At Sheikh Hassan archaeologists uncovered a bitumen storage area with stacked ingots and therefore Mesopotamians may have imported the material not only as an item of trade but also for their own use. Based on its location and the presence of this bitumen storage area, Sheikh Hassan may have served as a “supply station” or shipment point for Mesopotamian traders further upstream on the Euphrates at sites like Hacinebi.

With the available data it is still difficult to determine the precise function of bitumen artifacts and their role within the exchange system but they likely were used as packaging material for other trade goods, as trade goods themselves and as waterproofing for reed boats transporting goods within this exchange system. Water transport was undoubtedly used by Uruk traders because it was much more efficient than land transportation (Algaze 2008). In ancient Rome, “it was...cheaper to transport wheat from one end of the empire to the other by sea than to cart it seventy-five miles” (Jones 1966: 311). In addition, a load of salt requiring 10 to 12 pack equids would have only taken up one-third the cargo space of a ship from the third millennium (Potts 1994).

Mesopotamian reed boats made from bundled reeds, rope and bitumen are mentioned twice in Strabo's *Geographikon*, (16. I. 9-10) "...the overflow of the waters (of the Tigris and Euphrates)...forms lakes and marshes and reed-beds, which last supply reeds from which all kinds of reeds vessels are woven. Some of these vessels, when smeared with asphalt, can hold water, whereas the others are used in their bare state". A bitumen artifact from Pre-Contact context Hacinebi was most likely a remnant of waterproofing on a bundled reed boat and suggests that riverine transport was important to the local economy of the region even before Mesopotamian traders arrived in the area (Schwartz 2002). The region of Hacinebi has historically been an important crossing point of the Euphrates on the overland trade routes that ran east to west (Algaze 2008). It seems likely that Mesopotamians constructed or repaired bitumen coated reed boats on the upper Euphrates for the transport of metal and stone downstream to the Uruk heartland. This would require significant amounts of bitumen most of which came from the sources near Hit.

For Hacinebi, the data suggest changes in trade routes and regional interaction from the Early Uruk (Pre-Contact phase) to the Middle Uruk period (Contact Phase) as a result of the economic expansion of southern Mesopotamia. At Hacinebi there is a noticeable change in bitumen sources represented at the site in the "Pre-Contact" versus the "Contact" period suggesting a shift from the east-west trade route to a focus on the north-south Euphrates route, with the arrival of Mesopotamian traders. In the Late Uruk period, there is another shift back to overland trade routes connecting northern Mesopotamia to the Upper Euphrates.

In the Late Uruk period, the bitumen artifacts from the Mesopotamian sites of Habuba Kabira and Jerablus Tahtani on the northern Euphrates, as well as the Uruk enclave of Tell Brak were most likely from the Kirkuk region. These results are consistent with the work of other scholars on bitumen samples from Tell Brak, which demonstrated importation of bitumen from northern Mesopotamian sources and central Mesopotamian sources (Connan, Van de Velde 2010). This shift in trade routes over time is also consistent with other data that suggests the Uruk expansion changed in nature from being a predominantly trading venture to a system of colonial settlement (Figures 18, 19). Accordingly, the connection to the heartland via the Euphrates lessened in intensity while the overland route to the Tigris region predominated.

Some scholars contend Middle Uruk colonies such as Hacinebi and Sheikh Hassan were focused mainly on exchange and transportation of raw materials to southern Mesopotamia while Late Uruk sites such as Jebel Aruda and Habuba Kabira were focused mainly on local agricultural production (Algaze 2008; Schwartz 2001). These later colonies were not focused on trade but on the settlement of southern Mesopotamians to the Middle and Upper Euphrates (Schwartz 2001). From the stable isotope analyses presented here, one can see a greater percentage of bitumen from Sheikh Hassan and Hacinebi deriving from southern Mesopotamian sources with more bitumen from Habuba Kabira and Jerablus Tahtani deriving from northern Mesopotamian sources. While the number of samples analyzed from these sites is small, the data suggest that during the Middle Uruk Period, colonists at Sheikh Hassan

and Hacinebi were transporting bitumen from central and southern Mesopotamia, presumably as part of trade related activities, while during the Later Uruk Period, colonists were supplying themselves with bitumen via the east-west land route to the Kirkuk region of modern day Iraq.

Prospectus for Future Research

Whole sample bulk isotope analyses can provide information on a macro scale of large data sets, which should be then confirmed with detailed molecular analyses. It is hoped that further advancement in petroleum geochemical methodology will allow for less labor-intensive extraction of asphaltene compounds so a greater number of bitumen samples could be analyzed with more reliable measures. This research would also benefit from analyses of more material from Uruk colonies and enclaves. Bulk stable isotope analyses on whole samples were not sufficient in characterizing distinct sources within the region of southern Mesopotamia such as the Hit sources as compared to sources from southwest Iran. Moreover, the identification of the unknown source area characterized by these analyses is an essential step in reconstructing ancient exchange patterns from the Uruk period. The limitations of the analyses confirm that bulk isotope analyses on their own are an approximation of source and are not able to provide the level of detail that molecular analyses can (Connan and Nishiaki, 2003). Even with detailed molecular analyses, “A perfect asphalt- to-archaeological bitumen correlation is not a definite proof of the import of this bitumen to the site” (Connan and Van de Velde 2010: 3). However these

preliminary results have identified several potentially useful avenues for future research.

Acknowledgements

The authors wish to thank Leah Minc and Geoff Emberling for inviting us to be part of an invited session on materials analyses in the Uruk expansion. This research was made possible through a National Science Foundation Doctoral Dissertation Improvement Grant (BCS Award #0115596). We wish to thank the Urfa Museum of Şanlıurfa, Turkey, its former museum directors, Adnan Misir and Eyup Bucak and the Republic of Turkey for allowing the transfer of bitumen samples from Hacinebi Tepe. We are deeply indebted to a great number of individuals, from both museums and academic institutions that provided source and archaeological proxy samples for these analyses. Many of these scholars not only generously loaned us their artifacts for scientific research but also gave advice, new information and great support throughout this project. We are greatly indebted to Professor Gil Stein, director of the Oriental Institute at the University of Chicago and the director of the Hacinebi Research Project for initiating and guiding this work. The authors wish to thank the editors of the Journal of Archaeological Science Reports and three anonymous reviewers for their comments, questions and suggestions. Their insight was of enormous value in the rewriting of this manuscript.

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Figure 1

Map showing bitumen sources and fourth millennium BC sites in the ancient Near East.

Figure 2

The source regions most likely used by people in southern and northern Mesopotamia and traded to other regions. The primary sources are the ones in central Mesopotamia near Hit, the Magda sources in northern Mesopotamia near Mosul and Kirkuk and numerous sources in the Khuzistan and Deh Luran plain of southwestern Iran.

Figure 3

Bitumen artifacts from Hacinebi Tepe showing a range of forms and functional attributes. Clockwise from upper left: Two examples of bitumen artifacts bearing impressions from reed matting. The geometric shape suggests they were ingots transported for trade or later use; artifact bearing impressions of basketry; a shapeless piece, melted in appearance that was most likely debris from bitumen processing.

Figure 4

Bitumen coating on the inside of an Uruk spout jar (left) and Chalcolithic hammerhead bowl (right), both from Hacinebi Tepe, Turkey.

Table 1

Source Proxy samples analyzed in this study.

Figure 5

Gas chromatograms of branched and cyclic alkanes from proxy source sample from Kish (proxy for Hit), and three samples from Hacinebi Tepe. Two samples were associated with Local Chalcolithic style pottery and one was from Uruk context. Compounds are labeled with androstane as the standard.

Figure 6

Bivariate plot of the stable carbon isotope ratios of 28-Nor-17 β H-hopane and 17 α H-21 β □□hopane in the four samples analyzed using gas chromatography. The results show a high degree of similarity between the Uruk context sample from Hacinebi and the sample from Kish in central Mesopotamia.

Figure 7

Hypothesized source groups based on visual comparisons of stable isotope ratios (ellipses represent source groups with outliers included). Proxy material from central and southern Mesopotamia appears chemically distinct from bitumen from Hacinebi Tepe derived solely from Local Chalcolithic contexts.

Figure 8

Source group designations compared to modern source samples and proxy material from northern Mesopotamia and southeastern Anatolia.

Figure 9

Statistical construction of confidence ellipses around source groups (ellipses represent 90% confidence intervals around source groups with outliers removed).

Figure 10

Bulk carbon and deuterium isotope ratios of Pre-Contact (early and early middle Uruk Period, Phase A, B1) local context samples and Contact period (late middle Uruk. Period, Phase B2) local context samples from Hacinebi Tepe (ellipses represent 90% confidence intervals around source groups with outliers removed).

Figure 11

Bulk carbon and deuterium isotope ratios of late middle Uruk period artifacts from Hacinebi Tepe located in the “production area”.

Figure 12

Bulk carbon and deuterium isotope ratios of bitumen samples associated with Uruk contexts from Hacinebi Tepe.

Figure 13

Bulk carbon and deuterium isotope ratios of bitumen samples from Sheikh Hassan.

Figure 14

Bulk carbon and deuterium isotope ratios of bitumen samples from Godin Tepe

Figure 15

Bulk carbon and deuterium isotope ratios of bitumen samples from Habuba-Kabira

Figure 16

Bulk carbon and deuterium isotope ratios of bitumen samples from Jerablus-Tahtani

Figure 17

Bulk carbon and deuterium isotope ratios of bitumen samples from Tell Brak

Figure 18

Bulk carbon and deuterium isotope ratios of bitumen samples from Middle Uruk and Late Uruk Periods.

Figure 19

Maps outlining the evolving exchange patterns between Mesopotamia and southeastern Anatolia during the fourth millennium BC. From left to right, Early fourth millennium and early Uruk Period (Hacinebi Phase A, B1; Santa Fe Chronology LC1, LC2, LC3); Middle Uruk (Hacinebi Phase B2, Santa Fe Chronology LC4); and Late Uruk (Santa Fe Chronology LC5).

Name	Archaeological proxy sources	Museum location	# Of samples	Geographical region	Cultural affiliation	Obtained through the help of:
Kish	Neo-Babylonian ca. 600 BC	The Field Museum, Chicago	10 samples	S Iraq		Prof. Charles Stanish, Holly Lundberg, Dr. Catherine Sease
Ur	Early Dynastic ca. 2600 BC	University of Pennsylvania	Two samples	S Iraq		Prof. Richard Zettler, Dr. Sturart Fleming, Prof. Mitchell Rothman
Sakheri Sughir	Early dynastic ca. 2800–2350 BC	University of Michigan	Four samples	S Iraq		Prof. Henry Wright
Chogha Mish	Archaic periods and early dynastic periods	University of Michigan	Six samples	SW Iran		Prof. Henry Wright, Dr. Ray Tindel, Laura D'Alessandro
		Oriental Institute – University of Chicago	12 samples			
Djaffarabad	Ubaid ca. 5300–3600 BC	University of Michigan	Two samples	SW Iran		Prof. Henry Wright
Nuzi	Akkadian 2300–2100 BC, Sassanian	The Semitic Museum, Harvard University	Two samples	N Iraq		Dr. James Armstrong
Tell Qarqur	Bronze age	Boston University, University of Chicago	One sample	Syrian Levant		Prof. Rudolph Dornemann, Sarah Graff
Lidar	Neolithic	Heidelberg University	One sample	SE Turkey		Klaus Schmidt
Demirkoy	Neolithic	University of Delaware	Two samples	SE Turkey		Prof. Michael Rosenberg
Uruk/Warka	Uruk and early dynastic	Freie Universitat Berlin	Three samples	S Iraq		Prof. Margarete van Ess
Alalakh/Acane	Bronze age	University of Chicago	One sample	SE Turkey		Prof. Aslihan Yener
Kenan Tepe	Hellenistic	University of Utah	One sample	SE Turkey		Prof. Bradley Parker, Andre Creekmore