# **RESEARCH ARTICLE**

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# Petrographic and SEM-EDX characterization of Mousterian white/beige chert tools from the Navalmaíllo rock shelter (Madrid, Spain)

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# Abstract

Studying lithic raw material sourcing, processing and distribution is helpful when trying to reconstruct the territory, ecology, and cultural practices of Neanderthal groups. The use of multiple methods in such analyses allows for more refined characterizations to be made, helping to distinguish between materials better than any single method. Although 85% of the raw materials making up the Mousterian assemblage at the Navalmaíllo Rock Shelter (Pinilla del Valle, Madrid Region of Spain) correspond to the available local geological resources, 10% is made of white/beige chert, which is not registered in regional geological cartography. Petrographic and scanning electron microscopy with energy dispersive X-ray spectroscopy analyses were performed to determine the origin of this white/beige chert and thus shed light on the procurement strategies of the Neanderthal groups that occupied the rock shelter. The results show this chert to correspond to three different types of rock: quartz-kaolinite rocks from dykes, cretaceous marine cherts, and quartz fillings of cavities/fractures. These findings are in accordance with the geological features and formations present in the Lozoya valley, as recorded during a geoarchaeological survey, and indicate that the Neanderthals occupying the center of the Iberian Peninsula possessed a detailed knowledge of the landscape that allowed them to exploit its resources during MIS5a-early MIS4.

#### KEYWORDS

lithic raw materials, Navalmaíllo rock shelter, Neanderthal, petrography, SEM-EDX

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# **1** | INTRODUCTION

The study of raw lithic materials can provide assistance in the reconstruction of resource availability (Knutsson, Knutsson, Molin, & Zetterlund, 2016 Marks, Shoker, & Zilhão, 1991), exploitation, and economics (Aubry, Luis, Mangado Llach, & Matias, 2012; Moreau et al., 2016; Pereira & Benedetti, 2013), the perception of the landscape (Clarkson, 2008; Hiscock, 2014), and the movement patterns of past human groups (Delagnes & Rendu, 2011; Eixea, Roldán, Villaverde, & Zilhão, 2014; Manninen & Knutsson, 2014; Peresani, Boldrin, & Pasetti, 2015). Additional techniques such as petrographic microscopy can also vield valuable information about the environment in which the rocks used to produce stone tools were formed, helping to identify sourcing areas (Biró, 2011; D'Amico, 2005; Doronicheva, Nedomolkin, Kulkova, & Gerasimenko, 2017; Mangado, 1998, 2005, 2006; Terradas, 1995; Roy Sunyer, Vinagre, Benito-Calvo, Mora Torcal, & Martínez-Moreno, 2013). However, this is a destructive method rarely used to study artefacts directly. Fortunately, nondestructive methods that require no specialized sample preparation have recently become available (Campbell & Healey, 2016; Carvalho & Pereira, 2017; Constantinescu, Bugoi, & Sziki, 2002; Eixea et al., 2014; Frahm, 2014; Nadooshan, Abedi, Glascock, Eskandari, & Khazaee, 2013; ten Bruggencate, Fayek, Brownlee, Milne, & Hamilton, 2013), though individually these methods have limitations. The detailed characterization of raw lithic materials, and therefore the identification of sourcing areas, requires complementary methods to be employed (Andreeva, Stefanova, & Gurova, 2014; McDonnell, Kars, & Jansen, 1997; de la Torre et al., 2017). The determination of raw material sourcing areas allows us to establish resource exploitation strategies and mobility patterns during Prehistory. Geneste (1985) established a model for economic zoning distinguishing different acquisition patterns from which several authors have based their methodologies on by subdividing materials according to the mode of acquisition or resource availability. These are defined by determining the distance between the archaeological site and the sources of supply (Fernandes, Raynal, & Moncel, 2008; Geneste, 1985; Park, 2007; Villeneuve, Faivre, Turq, & Guadelli, 2019). Lithic materials found less than 5 km from the site are considered to be local resources, while lithic materials found between 5 and 20 km are considered of medium distance, and lithic materials found >20 km are considered long distance resources. These procurement patterns are defined according to the proportion of a raw material in an assemblage and the distance of the catchment area from the known settlement area (Clarkson & Bellas, 2014; Mester, Faragó, & Lengyel, 2012; Turg, 2005; Wilson, 2007).

Studies on the Middle Paleolithic occupation of the Central Iberian Peninsula have recently intensified with the discovery of new sites in the Central System mountain range, including Pinilla del Valle (Arsuaga et al., 2012; Baquedano, Márquez, Laplana, Arsuaga, & Pérez-González, 2014), Abrigo del Molino (Álvarez-Alonso, Andrés-Herrero, Díez-Herrero, Medialdea, & Rojo-Hernández, 2016), El Cañaveral (Nieto-Márquez & Baena-Preysler, 2016), Cueva de los Casares (Alcaraz-Castaño et al., 2017), and Jarama VI (Kehl et al., 2013). These sites indicate the continuous presence of Neanderthals in the region from MIS 5 until MIS 3 (spanning between 130,000 and 57,000 BP). Data that might throw light on their behavior and movement patterns, however, remain scarce. Global regional and interregional analyses of resource acquisition and exploitation strategies are rare due to the lack of available published data (Mangado, 2006; Pereira & Benedetti, 2013). It is important to mention that the investment in these analysis during the last two decades indicates an increase in raw material studies to which the present work intends to contribute with the first data for the central peninsular region.

The Navalmaíllo Rock Shelter (NRS; Pinilla del Valle, in the north of Spain's Madrid Region) is an upper Pleistocene archaeological site with a Mousterian lithic assemblage and includes a wide variety of raw materials, including chert. Among this chert, there is an abundance of a white/beige variety, which is currently not registered in the regional cartography throughout the assemblage. Determining the origin and catchment areas of these cherts should therefore provide insight into the mobility and knowledge of landscape resources of the Neanderthals that used them.

The aims of this research are (a) to characterize the types of white/beige chert present in the assemblage, (b) to determine their probable environment(s) of formation, and (c) to understand the procurement strategies followed by the Neanderthals of the central lberian Peninsula. For this, the white/beige chert from the NRS were subjected to macroscopic, mesoscopic and microscopic analyses.

# 1.1 | The geological context

The Central System is a major mountain range of the Iberian Peninsula. It is a pop-up structure formed during the Alpine Orogeny, with an ENE-WSW trend, a length of c. 600 km, and is mainly composed of orthogneisses, granitoids, migmatites, quartzites and schists (Aparicio & García, 1987; Fuster, Aparicio, Casquet, Garcia, & Peinado, 1974; Lopez Ruiz, Aparicio, & García, 1975). The Central System is a predominant feature of the Iberian Meseta, dividing it into the northern and southern submesetas. The mountain ranges of the System can be divided into three sectors: the Guadarrama, Gredos, and Ayllón ranges.

The Guadarrama range (Figure 1a), of which Peñalara is the highest peak (2,487 m), lies between the Gredos and Ayllón ranges, is about 80 km in length, and shows intense regional metamorphism. The Lozova river valley (an intramountain valley flanked to the north by the Montes Carpetanos mountains and to the south by Cuerda Larga ridgeway), in the upper part of which the NRS lies, can be subdivided into four reaches according to its lithology and morphology (Karampaglidis, 2014). This upper reach of the valley corresponds to a NE-SW trending tectonic depression superimposed on the deep-rooted formations of the Centroiberian Palaeogeographic Zone (Julivert, Fontboté, Ribeiro, & Conde, 1974). These formations are composed of Neoproterozoic-Carboniferous rocks, in part metamorphosed and with intrusions of different types of granitoid dated to before the Permian. The oldest exposed rocks are formed from orthogneisses, leucogranites, granitoids, adamelites, migmatites and, to a lesser extent, schists and guartzites. Late quartz, lamprophyres, and porphyry dykes are also present (Aparicio, Barrera, Caraballo, Peinado, & Tinao, 1975; Karampaglidis, 2014;



**FIGURE 1** (a) Map showing the location of Navalmaillo Rock Shelter in the Lozoya river valley in the Guadarrama Mountain Range (Spain); (b) oblique aerial photograph of the Calvero de la Higuera karstic hill with the location of the excavated area of the Navalmaillo Rock Shelter; (c) Geologic map of the region with area of collected geological samples AM 01 to AM08 (adapted from geologic map MAGNA 50 N° 484–Buitrago de Lozoya at a scale 1:50,000). Lithologies: 33-Sand, silt, and gravel (alluvium) 32-Gravel and sand (colluvium); 31-Gravel and sand (terrace); 30-Sand, silt and gravel (glacis); 29-Cobble, gravel, and sand (debris cone); 28-Cobble and gravel (debris cone) 27-Quartzite and schist cobbles and gravel; 26-Gneiss and granite cobbles and gravel 25-Polymictic conglomerate and carbonate cemented sand; 24-Sand, clay, and gypsum; 23-Cavernous dolostone and marl; 22-Dolostone and dolomite cemented sandstone; 21-Dolomite cemented sandstone; 20-Laminated dolostone; 19-Gray sand, carbonate partial cemented sand; 18-Sand, clay and cobbles (Utrillas facies); 17-Sand, siliceous cemented sand, clay and gravel; 13-Calcsilicate and amphibolite rocks; 12-Leucogneiss, schist, and metapsamite; 11-Leucogneiss and glandular leucocratic orthogneiss; 10-Biotitic banded orthogneiss; 9-Microglandular orthogneiss; 8a-Glandular orthogneiss; 8b - Mojada del Cojo facies; 7-Mesocratic glandular orthogneiss; 6-Fine and medium grained leucogranite; 5a-Medium and coarse-grained adamellite and biotitic granite; 5b-Medium and coarse-grained adamellite and biotitic granite with megacrysts; 4-Migmatitic granitoid; 3: Quartz; 2: Lamprophyre; 1: Porphyry [Color figure can be viewed at wileyonlinelibrary.com]

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Pérez-González et al., 2010). Alpine tectonic activity preserved stretches of post-Variscan strata from the Early Cretaceous, composed of sands, clays, and conglomerates from the Utrillas facies (Alonso Millán, & Mas, 1982). On top of this formation, sands, shales and carbonates, sandstones and bedded limestones or dolostones are exposed. The marine sequence ends with a few meters of sand, clay, and gypsum layers. Also present are Tertiary sediments of Neogene age, formed mainly by large granite and gneiss boulders deposits on top of previous formations in angular and erosive discordance (Arenas Martín et al., 1994; Bellido, Escuder, Klein, & Del Olmo, 1991; Polo, Segura, Carenas, Gil, & García-Hidalgo, 2003). The most recent deposits, from the Quaternary, are widely represented and correspond mostly to Late Pleistocene moraines associated with glacial cirgues. These deposits are occasionally molded by fluvial-torrential actions (Karampaglidis, Benito Calvo, Pérez-González, Baguedano, & Arsuaga, 2011; Pérez-González et al., 2010).

The Calvero de la Higuera hill (Figure 1b), where the Pinilla del Valley Upper Pleistocene archaeological sites are located is an outcrop of Upper Cretaceous carbonate rocks running in a W-E direction. The dolostone stratum is associated with karstic processes and fluvial erosion caused by the Lontanar and Valmaíllo streams (tributaries on the right bank of the river Lozoya; Pérez-González et al., 2010). This karst system has three levels distinguishable by their different resistance to erosive agents: (a) a lower level of highly erodible marls; (b) an intermediate level with more soluble and porous carbonates allowing the formation of rock shelters and caves; (c) an upper level with harder cemented carbonates more resistant to erosion, allowing the formation of roofs and overhangs (Pérez-González et al., 2010).

### 1.2 | The Navalmaíllo rock shelter

The Pinilla del Valle archaeological sites (altitude 1,100 m) are located within the partially collapsed karst system of the Calvero de la Higuera hill, which covers an area of some 3 ha (Figure 2b). The Component Camino Cave, NRS, Buena Pinta Cave, Des-Cubierta Cave and Ocelado Rock Shelter sites were occupied by both Homo neanderthalensis and carnivores at different times during MIS 3-6 (Arsuaga et al., 2012; Baquedano et al., 2012).

The NRS was discovered in 2002 and hangs about 8 m above the Navalmaíllo streambed. Some 60 m<sup>2</sup> of its (at least) 300 m<sup>2</sup> have been excavated (Figure 2-a). According to thermoluminescence dating, it was occupied by Neanderthals during MIS 5a and early MIS 4 (UAM Mad-4262 = 71.685 ± 5.08 ka; Mad-3767 = 77.230 ± 6.016 ka; Arsuaga, Baquedano, & Pérez-González, 2011).

Levels D and F in the southern area of the rock shelter have a sandyclay texture and show alterations caused by pressure due to the collapse of the roof (Márquez et al., 2013). The collapses affecting level D created fissures and spaces between the boulders, allowing the vertical hydroplastic injection of sandy clay sediments containing artefacts from level F.

Although both levels D and F are rich in Mousterian lithic artefacts as well as faunal remains that show clear signs of human processing (such as cut marks), the higher density of material in Level F suggest this to represent the main period of occupation (Moclán et al., 2018). Herbivores, particularly members of Bovinae, Cervidae, and Lagomorpha, are the most abundant taxa represented (Arriaza et al., 2015). Both levels D and F have also yielded hearths and burnt bones.

Lithic artefacts represent 60% of the total archaeological assemblage up to 2017. These include a wide variety of rocks (Figure 2a) including quartz (78%), chert (11%), quartzite (2%), rock crystal (2%), and other rocks (total 7%, with each rock type represented by 1% or less). Around 88% of the raw materials in the NRS assemblage are clearly coincidental with the geology of the Guadarrama mountain range, and in particular with that of the high valley of the river Lozoya. They are therefore of local origin (radius < 5 km; Geneste, 1985; Meignen, Delagnes, & Bourguignon, 2009). The white/beige chert component (10%) is of interest (Figure 2b) because there is currently no known source of this material in this region.

#### Sources of chert 1.3

The information provided in the literature indicates that the closest primary chert sources to the NRS are Cretaceous deposits in Segovia and Madrid regions (c. 25 and 45 km, respectively), cherts in Cenozoic deposits of the Madrid and Duero basin (c. 50 and 65 km, respectively), and dykes and lenticular bodies of guartz included in the granitic and metamorphic rocks of the Central System.

In the Tabladillo Valley (Segovia), a Campanian age marine formation called "dolostones and marls of the Tabladillo Valley", contains chert (Arenas Martín et al., 1994). This unit consists of cream-colored dolostones, gray and red clays. The first correspond to carbonate mudstones and wackestones with calcareous fossil remains affected by dolomitization. Gypsum crystals of the "selenite" type and some silicifications are also described. After dolomitization, diagenetic processes such as dedolomitization, carbonate dissolution, and silicification occurred. Cherts constituted by mosaics of quartz, length fast, and length slow chalcedony are formed by silicification.

Detritic rocks, clays and gypsums with chert are recorded in the North and South areas of the Central System (Del Olmo Sanz & Martínez Salanova, 1989). The age of this unit would correspond to the interval between the upper Cretaceous (Maastrichtian) and the lower-middle Oligocene. The sedimentary environment in which siliciclastic materials could coexist with detritic-evaporitic materials, both with tidal structures and where, in addition, edaphic processes occurred, could be fan delta type (Del Olmo Sanz & Martínez Salanova, 1989). At the south of the Central System, the detritic-evaporitic unit with chert is described near Torrelaguna (c. 25 km in a straight line from NRS; Nodal & Agueda, 1976).

Cherts in Cenozoic deposits are frequent in the Madrid Basin. Considering only those outcrops with cherts closest to the NRS, we can find cherts in the Paleogene in Torremocha de Jadraque (Bustillo, Arribas, & Bustillo, 2002) and in the Miocene in Brihuega-Jadraque and surroundings, (Bustillo, 1976; Peréz Jiménez, 2010). Both are located c. 74 km from the NRS. All cherts mentioned were formed by silicification of palustrine or lacustrine deposits (limestones, dolostones and gypsums). Depending on the silicified rock, the quartz textures are different, showing mosaics of quartz, length fast, and length slow chalcedony.



FIGURE 2 (a) Pie chart with the % of the main raw materials present in Navalmaillo's Rock Shelter Level F lithic assemblage; (b) Examples of the five macroscopic types (A, B, C, D, and E) of white and beige chert lithics from Navalmaillo Rock Shelter [Color figure can be viewed at wileyonlinelibrary.com]

As in the Madrid Basin, the presence of chert is relatively frequent in Cenozoic deposits of the Duero Basin (Fuertes, Neira, Fernandez, Goméz, & Alonso, 2016). In the southern area of the basin, the outcrops of chert described, closer to the Central System, are located at c. 65 km from the NRS in Sacramenia, Maderuelo and surroundings (Armenteros, Bustillo, & Blanco, 1995 and Bustillo & Armenteros, 1991). Cherts show textures typical of evaporite replacements (from microcrystalline to macrocrystalline) mosaic quartz and length slow chalcedonies.

Dykes and lenticular bodies of quartz are formed by mosaics of quartz crystals and chalcedony (length slow or length fast), and therefore can have a mechanical behavior similar to chert. Numerous dykes/lenticular bodies of quartz are included in the granitic and metamorphic rocks of the Central System (Aparicio et al., 1975). Its composition is highly variable, showing in the Sierra de Guadarrama small amounts of other minerals in addition to quartz, as muscovite, fluorite and Fe oxides (Martín Crespo, 2004). The presence of quartz dykes with kaolinite only have been described in the Ávila province, by Ubanell, Garzón, de la Peña, Bustillo, and Marfil (1978) and Marfil, Bustillo, and Palacios (1980) at 60 km from the NRS.

# 2 | MATERIALS AND METHODS

Over the last decade, raw material characterization and sourcing studies have become easier and cheaper to perform thanks to the appearance of non-destructive, high-resolution techniques. However, none of these methods alone covers all the diagnostic features that characterize rocks and minerals and their sub-types.

White/beige chert archaeological samples from the lithic assemblage of NRS levels F and D were subjected to macroscopic, petrographic and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) analyses to determine their origin(s).

#### 2.1 | Macroscopic analysis

Macroscopic analyses were performed at the Regional Archaeological Museum of Madrid. Such analysis is essential when inspecting and classifying lithic raw materials (Aubry, 2005; Moreau et al., 2016). Macroscopic analysis (Table 1) provides a rapid, inexpensive, non-destructive means of comparing large assemblages, even in the field. The comprehension of the results, however, can be a little subjective since they record qualitative traits; their correct interpretation therefore depends on the analyst being experienced (Agam & Wilson, 2018).

Archaeological specimens were examined (Table 1) for their color, superficial texture, internal texture (after being cut into thin sections), luster, porosity, homogeneity, isotropy in fracture, cortex presence, flaking quality, retouch quality, and transparency (these materials were rated following common mineralogical procedures; Klein & Hurlbut, 1998). Based on the results, the specimens were divided into five different groups with common traits: Macroscopic Types A–E.

#### 2.2 | Petrographic analysis

Petrographic analysis of seven samples of white/beige chert (MNCN 1, MNCN 2, MNCN 3, MNCN 4, MNCN 5, MNCN 6, and MNCN 7) were found to represent all the macroscopic types of white/beige chert detected. Analysis was performed following standard optical microscopy methods at the National Museum of Natural Sciences in Madrid (Spain). During the geological survey of the Lozoya river valley, rock samples from the Calvero de la Higuera and other macroscopically similar to the NRS artefacts were collected for direct comparison (samples AM01 to AM08) with the tools.

Thin sections were studied under plain and cross-polarized light to observe their mineralogical composition, texture, and to define petrological groups. A gypsum plate was used to obtain information

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TABLE 1	. Visual ma	croscopic descr	ription of seve	n white an	d beige chert sample	s from Nava	lmaíllo Rock Shelt€	er ascribed	to macros	copic type	s A, B, C, D and	ЧЕ	
Sample	Color 1 (a)	Color 2 (b)	Superficial texture (c)	Luster (d)	Internal texture (e)	Porosity (f)	Homogeneity (g)	lsotropy in fracture (h)	Flaking quality (i)	Quality in retouch (j)	Transparency (k)	Macroscopic type	Petrographic type
MNCN 1	6N	I	ST3	L2	Microgranular	P4	H1	11	F2	R2-R3	Γ3	A	Type 1
MNCN 2	6N	I	ST3	12	Aphanitic, porous spongiform	P2+P3	H1	1	I		Γ3	в	Type 3
MNCN 3	6 <sub>N</sub>	8X	5T2	2	Aphanitic, sub millimetric/ millimetric ameboid inclusions of microcrystalline quartz, some with microgeodes	P3 (mass); P2 (some quartz inclu- sions)	H2	<u>T</u>	F3-F4		T3 (mass); T2 (inclusions)	۵	Type 3
MNCN 4	10 YR 8/2 (inter- nal); 10YR 7/4 (cortex)	9X	ST2-5T3	2	Microgranular crystalline with submillimetric spheroid particles of quartz (microfossils?)	<b>P</b>	H1	1	F2	R.	ε	U	Type 3
MNCN 5	6 Z	N8&10YR 8/6	5T3	L2	Microvenular with concretion structures involving earthy stains	ЪЗ	H1	1	F2	R1	£	ш	Type 2
MNCN 6	6 Z	5Y 8/1	ST1	L2-L3	Microgranular with transparent sub millimetric particles (microfossils?)	P1+P2+P3	H2	<u>8</u>	F4	R4	T4+T2	۵	Type 3
MNCN 7	6N	I	ST1-STS2	L2	Aphanitic	P4	H1	11	F2-F3	R2-R3	Γ3	А	Type 1
Note: (a,b) N	Munsell rock	color codes of c	dominant and e	ventual dist	inct secondary spots.	(c) Graded in	classes. ST1: gritty;	; ST2: micro	granular; S	T3: smooth	; ST4: shiny. (d)	Graded in class	es. L1: waxy; L2:

imperfect waxy; L3: dull. (e): textural description. (f) Graded in classes. P1: high, considerable water absorption by very fine pores; P2: moderate with disperse coarse pores; P3: barely sensible water absorption; P4: no pores or impervious to water. (g) Graded in classes and textual description. H1: homogeneous in bulk; H2: heterogeneous at millimeter scale. (h) Graded in classes: 11: regular; 12: regular vith imperfections; 13: irregular. (i) Graded in classes: F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: good; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F3: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: fair; F4: weak. (j) Graded in classes. F1: excellent; F2: fair; F4: fair; F4: fair translucid in thin portions; T4: opaque.



**FIGURE 3** Thin section petrography images of seven white and beige chert samples from Navalmaíllo Rock Shelter divided in three petrographic rock types. Type1: MNCN 1 (A) Mega quartz mosaic with fan shaped kaolinites in plane polarized light (PPL) and (B) cross-polarized light (CPL); MNCN 7 (A) fans of platelet kaolinites in PPL and (B) CPL; Type 2: MNCN 5 (A) detail of chalcedony evolving to mega quartz in PPL and (B) CPL; Type 3: MNCN 2 (A) Chert with great micro porosity in PPL and (B) CPL; MNCN 3 (A) General aspect, showing crystalline shapes, non- ascribed ovoid's and possible fragments of bivalves in PPL and (B) CPL; MNCN 4 (A) Spicule of silica sponge in PPL and (B) CPL; MNCN 6 (A) General aspect, showing crystalline shapes, non- ascribed ovoids and possible fragments of bivalves in PPL and (B) cPL; Color figure can be viewed at wileyonlinelibrary.com]

regarding chalcedony elongation (positive or negative) and the corresponding variety.

#### 2.3 | Scanning electron microscopy analysis

Subsamples of MNCN 1-7 were also analyzed by SEM-EDX at the National Museum of Natural Sciences in Madrid (Spain). These observations were made using a FEI INSPECT microscope, working at 30 kV at a distance of 10 mm, in high-vacuum mode, and employing secondary (SSD) and backscattered electron detectors. This instrument was equipped with an Oxford Analytical-Inca X-ray EDX for semiquantitative elemental analysis.

# 2.4 | Geoarchaeological surveys

To determine the likely origin of the examined lithic resources, the availability in the landscape of the rock types from which they were

made was established. This involved (a) the examination of published geological studies on the Provinces of Madrid, Ávila, Segovia, and Guadalajara; (b) a thorough geological survey of the Lozoya valley; (c) surveys of specific areas of interest close to the Lozoya valley. These latter two steps were necessary because outcrops or dykes associated with small geological features tend not to appear in conventional geological cartography - a problem of scale. Samples were then collected from the different areas for comparison with the archaeological assemblage to identify the possible sources of the white/beige chert artefacts.

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# 3 | RESULTS

#### 3.1 | Macroscopic analysis

Macroscopic analysis distinguished five types of white/beige chert, termed Macroscopic Types A to E. Table 1 presents the characteristics of each type.



FIGURE 4 SEM images of seven white and beige chert samples from Navalmaíllo Rock Shelter divided in three petrographic rock types. Type 1: MNCN 7 closeup of kaolinites (scale = 20 µm); Type 2: MNCN 5 (a) General aspect of numerous layers of chalcedony cements (scale =  $200 \,\mu$ m) and (b) bladed independent Fe minerals (hematite) between unaltered mega quartz crystals (scale = 10 µm); Type 3: MNCN 3 (a) Quartz pseudomorph after a possible dolomite crystal (scale =  $100 \,\mu$ m) and (b) Small and irregular guartz crystals from the groundmass with high porosity between them (scale =  $2 \mu m$ ); MNCN 4 Unaltered mega guartz mosaics and Fe oxides spherical aggregates (scale =  $30 \,\mu$ m); MNCN 6 Silica sponge spicules with strong alteration (scale =  $200 \,\mu$ m)

#### 3.2 | Optical petrography of archaeological samples

Samples MNCN 1, MNCN 2, MNCN 3, MNCN 4, MNCN 5, MNCN 6 and MNCN 7 were classified into three Petrographic types (Figure 3):

- Petrographic Type 1. (Samples MNCN 1 and MNCN 7). Silica rocks (not proper chert) composed of macrocrystalline quartz mosaics (from 50 to 200 µm) and kaolinites. The quartz crystals show irregular and interpenetrated edges; some crystals are idiomorphic and show zonation. In plain parallel light, brown kaolinites appear in abundance, randomly distributed, forming fans outside the quartz crystals or included within them. Fe oxide impurities are scarce but, in some cases, mark the edge of idiomorphic crystals.
- Petrographic Type 2. (Sample MNCN 5). Composed of successive layers of quartz cements and a groundmass. The cement layers are mainly made of length-fast chalcedony, although locally the

chalcedonies show both length-slow and length-fast features. Frequently, both types of chalcedony pass laterally to macrocrystalline quartz. Fe oxide appears in the cement but mainly included within the macrocrystalline quartz or within the different chalcedonies, randomly distributed, giving different colors to the textures. The groundmass is mainly composed of macro/meso crystalline quartz mosaics and length-fast chalcedony; locally length- slow chalcedony or a mix of textures of both chalcedonies is present.

Petrographic Type 3. (Samples MNCN 2, MNCN 3, MNCN 4, and MNCN 6). Containing ovoid and spherical forms difficult to interpret (probably grains of carbonates and silicified microfossils), crystalline shapes, and cemented voids. Groundmass made up of micro-cryptocrystalline quartz mosaics with no crystals visible under 30 µm and locally, length- fast chalcedony. Fe oxide accumulations occur in certain zones. Voids, with varying shapes and sizes, occur in samples MNCN 2 and MNCN 4. This porosity may result from an alteration of the samples since some of the voids have the shape of dissolved crystals; their rhombic form is reminiscent of dolomite and gypsum crystals. Ghosts of microfossils are visible, for example, sponge silica spicules, micro-foraminifera, bivalves, and ostracods. Also present are the ghosts of rhombic crystals assignable to dolomite, and occasionally zoned crystals (probably dedolomite) due to dedolomitization (dolomite-calcite transformation). Fe oxides are abundant, occasionally lining the rhombic crystals: these oxides are interpreted as a consequence of the transformation of dolomite (that would include Fe) to calcite (without Fe) during dedolomitization.

#### 3.3 | SEM-EDX features

Samples MNCN 1 and MNCN 7 (Petrographic Type 1) showed smooth surfaces of megaquartz crystals and flaky kaolinite crystals (Figure 4). EDX spectral analysis of the kaolinite returned Si, Al, O, and minor amounts of Ca. EDX data revealed the presence of Al in the megaquartz, perhaps due to interference by kaolinite during analysis.

Sample MNCN 5 (Petrographic Type 2) showed numerous layers of chalcedony cements of different size interspersed with cryptocrystalline and macrocrystalline quartz mosaics. The chalcedony cements and the more cryptocrystalline zones showed strong alteration (Figure 5). Fe minerals (hematite) were seen to make up independent crystals or found included within the chalcedonies.

The Petrographic Type 3 samples (MNCN 2, MNCN 3, MNCN 4, and MNCN 6) showed a groundmass of little-altered quartz crystals with great inter-crystal porosity. Chalcedonies were also present and showed strong alteration; larger quartz crystals showed no alteration. Silicified calcareous microfossils and silica sponge spicules with their inner channels were clearly visible although they showed strong alteration. Fe-rich spherical aggregates appeared scattered within the quartz mosaics. The EDX data for the groundmass revealed only Si and Fe.

#### 3.4 | Geological survey of the Lozoya river valley

#### 3.4.1 | Literature study

The information provided in the literature, indicated the existence of the quartz-kaolinite dykes, similar to Petrographic Type 1 crossing the plutonic-metamorphic formations of the Central System as described by Ubanell et al. (1978) and Marfil et al. (1980) in the Ávila province, 97 km SW of the NRS. Until now, no remains of these dykes were recorded in the Lozoya river valley although these can occur because there are dykes associated with plutonic and metamorphic rocks (Martín Crespo, 2000). Cherts similar to Petrographic Type 3 are described in the Upper Cretaceous dolostones and marls of the Tabladillo valley in Segovia (Alonso-Millán & Mas, 1982; Arenas Martín et al., 1994), 25 km NW of the NRS.

# 3.4.2 | Field survey

Rock samples that were the most macroscopically similar to the NRS artefacts were collected for direct comparison. Seven were collected from primary positions among the Calvero de la Higuera carbonate rocks (dolostones/limestones, samples AM01 to AM07), plus one (chert, sample AM08) collected in a secondary position in El Hontanar on the Canencia Horst ridge line 2.5 km away from the archaeological site.



**FIGURE 5** SEM-EDX images of two examples of contamination on the surface of the analyzed lithic tools. (a) NaCl probably from the acid treatment used to clean the surface of lithic tools from Ca concretions; (b) varnish layer from a previous tag on the tool not properly washed away with acetone

AM01-07 corresponded to carbonate rocks that after silicification would translate into Petrographic Type 3 chert. The microfossils, dolomitization and possible dedolomitization detected as ghosts in some of the NRS artefacts were present in the Calvero de la Higuera carbonate rocks; the chert from which these artefacts were made could therefore have come from cherts included in the Upper Cretaceous carbonate rocks in the Lozoya valley.

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AM08 represents a secondary chert with ghosts of stromatolitic laminae and gypsum crusts. These facies are recognized in the Campanian carbonate rocks of the Tabladillo valley (Arenas Martín et al., 1994). Therefore, AM08 sample testifies to the presence of an apparent remnant of the Cretaceous Period, at a nearby distance (2.5 km) of the NRS. This formation can occur in association with the geological outcrops of Petrographic Type 3 chert samples.

Figure 6 compares the characteristics of the examined artefacts and the rocks in the surrounding geological areas.

# 4 | DISCUSSION

Results from this study reveal that 97% of the lithic raw materials used by the Neanderthals of the NRS were collected in the local area. Their intense exploitation of these raw materials, which are only of fair-togood flaking quality, suggests they could adapt their technology to



**FIGURE 6** Correspondence of the results obtained by means of macroscopic and petrographic analysis of archaeological and geological samples [Color figure can be viewed at wileyonlinelibrary.com]

process materials with different knapping potential. This in turn suggests that the availability of other resources on which these populations depended (such as food and water) would be more important when making the choice of where to settle. The archaeological site density of the Upper Pleistocene for the northwest of the Iberian Peninsula and the Central System might therefore be higher than is currently suggested, particularly if there has been difficulty in recognizing sites rich in noncryptocrystalline assemblages.

The collected laboratory data has identified the existence of potential areas of geoarchaeological interest located in previously under surveyed areas surrounding the site. Petrographic Type 3 show ghosts of microfossils (sponge silica spicules, micro-foraminifera, bivalves, and ostracods) and features of dolomitization and dedolomitization. All these features indicate Cretaceous cherts which have the nearest occurrence described in the Upper Cretaceous dolostones and marls of the Tabladillo valley in Segovia (Arenas Martín et al., 1994), 25 km NW of the NRS. On the other hand, microfossils, dolomitization and possible dedolomitization detected as ghosts in some of the NRS artefacts were present in the Calvero de la Higuera carbonate rocks. Therefore, the chert from which these artefacts were made could have its origin in cherts included in the Upper Cretaceous carbonate rocks in the Lozoya valley, that is they are likely to be of local origin.

The existence in the bibliography of potential sources of raw material in Segovia and Ávila regions correspond to a long-distance procurement strategy. Nonetheless, since Cretaceouse cherts can be also found as clasts in the Paleogene deposits in the Lozoya Valley, the same types of rocks were found to be available at a short distance from the NRS which corresponds to a local procurement strategy. Local presence of sources of raw materials, can be considered a "favored sourcing area" as the reduced logistics involved in procurement could be favored by the group (Marks et al., 1991; Ruebens, 2013).

White/beige chert is not easy to find in the Lozoya river valley, showing the Neanderthal population possessed a detailed knowledge of the surrounding mountainous landscape and its resources. Indeed, the difficulty in finding this material today suggests that there may have been only small pockets of it, and that these may have become exhausted (certainly the Lozoya river valley was occupied by Neanderthals over a long period, which may have ensured such an exhaustion).

Recent human activities might, however, also explain the current absence of substantial sources, such as the construction of a dam during the 1970 s that dramatically altered the landscape and access to lithic resources in the region. Most of the area's chert outcrops are now submerged or covered by artificial terraces created by this civil engineering project. Sources of Petrographic Type 3 chert might, therefore, today be under the 38 hm<sup>3</sup> of water in the Pinilla reservoir.

Complementary methods of analysis are required to make reliable comparisons of artefacts and to determine the geological areas from which they might have come. Multiple complementary methods should be used, taking into account the relationship between cost, time and the results that can be obtained (Andreeva et al., 2014; McDonnell et al., 1997; de la Torre et al., 2017). A number of archaeological and geological samples of white/ beige chert were subjected to petrographic analysis. Petrography provides a detailed mineral and textural composition of the sample and allows for intra-specimen analysis, but it is destructive, and can be time consuming and costly.

SEM-EDX analysis was used to corroborate the features detected by petrography and to allow for more detailed observation of the alterations in the interior and on the surface of the lithic artefacts. Though studies have been published on postdepositional changes in cryptocrystalline lithic resources (Caux, Galland, Queffelec, & Bordes, 2018; Fiers et al., 2018) further study is needed to understand the causes and processes of alteration on tool surfaces and the patina that forms on chert.

# 5 | CONCLUSION

Macroscopic analysis of lithic industries based on a qualitative description was successfully used to define different rock types in raw material inventories. Thus, some features may be interpreted as indicators of another rock type in the assemblage's inventory when they are the result of taphonomic processes that alter not only macroscopically but also its internal chemical and physical structure. These changes can only be detected if multiple methods of analysis are applied.

Prehistoric raw material procurement studies are based on current geological resource mapping. If a source is exhausted, destroyed by anthropic or natural causes then it leaves little to no evidence of its existence in its original position. For this reason, petrographic analysis of stone tools is an effective methodology to determine the characteristics of the origin of the raw materials exploited. Subsequently, efforts were directed to target specific areas of interest where remnants of said formations could still exist. The results from this research show how the use of multiple, complementary rock analysis techniques provide for more significant comparisons and more reliable conclusions to be drawn on the origin of lithic materials.

Current research on knapping quality of raw materials in Middle Paleolithic sites located in the center of the Iberian Peninsula indicate new models are needed for Neanderthal mobility and settlement distribution patterns in the region. The present work shows that the Neanderthal population that occupied the Navalmaíllo Rock Shelter possessed an extensive knowledge of the local lithic resources that were available to them and were not dependant on the existence of raw materials with conchoidal fracture. Simultaneously, it reveals a strategy of intense exploitation of local resources which availability and access could have been a significant factor in the occupation of the Lozoya river valley for a considerable span of time.

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#### DATA AVAILABILITY STATEMENT

The authors confirm that the data supporting the findings of this study are available within the article.

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