Miona Miliša, Josipa Marić, Ivica Ljubenkov

FT-IR analiza uzoraka polikromije s kamenih antičkih skulptura iz Arheološkog muzeja u Splitu

FT-IR analysis of polychromy samples from antique stone sculptures in the Archaeological Museum in Split

Miona Miliša
HR, 21000 Split
Umjetnička akademija u Splitu
Zagrebačka 3
mionamilisa@yahoo.co.uk

Josipa Marić
HR, 21000 Split
Muzej hrvatskih arheoloških spomenika
Stjepana Gunjače 3
josipa.maric62@gmail.com

Ivica Ljubenkov
HR, 21000 Split
Prirodoslovno-matematički fakultet
Ruđera Boškovića 33
ivica.ljubenkov@pmfst.hr

UDK: 77.026.345:730“652”]:069(497.583Split)
Stručni članak
Primljeno: 14. 4. 2020.
Prihvaćeno: 28. 9. 2020.

Miona Miliša
Croatia, 21000 Split
Academy of Arts in Split
Zagrebačka 3
mionamilisa@yahoo.co.uk

Josipa Marić
Croatia, 21000 Split
Museum of Croatian Archaeological Monuments
Stjepana Gunjače 3
josipa.maric62@gmail.com

Ivica Ljubenkov
Croatia, 21000 Split
Faculty of Natural Sciences and Mathematics
Ruđera Boškovića 33
ivica.ljubenkov@pmfst.hr

UDC: 77.026.345:730“652”]:069(497.583Split)
Professional paper
Received: 14 April 2020
Accepted: 28 September 2020

Analize polikromije na kamenu iz prethodnih desetljeća uglavnom su se temeljile na procjeni pigmenata analiziranih golim okom ili mikroskopom, bez kemijske analize koja bi to mogla potkrijepiti. U današnje vrijeme analitička kemija može uvelike pomoći u identifikaciji ostataka izvorne boje, tj. korištenih pigmenata.

Poznato je da se polikromija na kamen nanosila u svim vremenskim razdobljima, ali dobro očuvana polikromija na kamenu uistinu je rijetkost. Najveći problem su antičke skulpture, čija je polikromija često nevidljiva golim okom i moguće ju je pronaći samo uz pomoć mikroskopa. Razlog lošoj očuvanosti polikromije je utjecaj atmosferilija koje uzrokuju kemijske promjene u kamenu i boji. Primjena metoda kemijskih

Analyses of polychromy on stone from previous decades were largely based on the evaluation of pigments analysed with the naked eye or the microscope, with no chemical analyses to substantiate them. Nowadays, analytical chemistry can greatly help in identifying residues of the original colours, i.e. the pigments used.

It is known that polychromy has been applied onto stone in all time periods, but well-preserved polychromy on stone is a rarity indeed. The greatest issue concerns antique sculptures, whose polychromy is often invisible to the naked eye and can only be seen with the help of a microscope. The reason for such poor preservation of polychromy lies in the weathering that causes chemical changes in the stone and

analiza koje se koriste u restauratorskoj struci može pomoći u očuvanju rijetkih primjera polikromije, u izradi vjerodostojne rekonstrukcije polikromije na predmetu te u odabiru prikladnih metoda restauracije i konzervacije. Stalni razvoj novih metoda i tehnika, razvoj računalne i digitalne tehnologije i tehnologije općenito pomaže nam u očuvanju vrlo rijetkih i vrijednih informacija, korisnih za shvaćanje naše prošlosti.

U svrhu istraživanja napravljene su analize uzoraka polikromije s kamenih skulptura iz nekoliko muzeja na hrvatskom području, a ovim radom iznosimo rezultate analize četiri antička predmeta iz Arheološkog muzeja u Splitu. Svrha istraživanja bila je napraviti instrumentalnu analizu pigmenata s kamenih skulptura, odrediti sastav pigmenata, podrijetlo i učestalost upotrebe određenih materijala. Analizom i tumačenjem često vrlo suptilnih razlika u sastavu pigmenata mnogo se može naučiti o vremenu, mjestu i okolnostima izrade određenog umjetničkog djela.

Ključne riječi: *FT-IR, polikromija, pigmenti, kamen, Arheološki muzej u Splitu*

colour. The application of chemical analyses as used by restoration professionals can help in preserving rare examples of polychromy, preparing credible reconstructions of polychromy on objects and selecting suitable methods of restoration and conservation. The constant development of new methods and techniques, as well as computer and digital technologies and technology in general help to preserve very rare and valuable information, useful for understanding our past.

For the purpose of the research, polychromy samples from stone sculptures from several museums in Croatia were analysed. This paper presents the results of the analyses of four antique objects from the Archaeological Museum in Split. The purpose of the research was to make an instrumental analysis of pigments from stone sculptures, and to determine the composition of pigments, the origin and frequency of use of respective materials. There is much one can learn about the time, place and circumstances in which a work of art was produced through analyses and interpretations of often very subtle differences in the composition of pigments.

Key words: *FT-IR, polychromy, pigments, stone, Archaeological Museum in Split*

Uvod

Još je od 18. stoljeća poznato da je rimska skulptura izvorno bila bogato ukrašena bojama, pozlatom i srebrom[1]. Rimski umjetnici koristili su širok spektar pigmenata, a autori kao što su Plinije Stariji[2] i Vitruvije pružili su važne informacije o tim materijalima.[3] Svrha i važnost polikromije u antici, ali i u drugim umjetničkim razdobljima često je podcijenjena i nedovoljno istražena, zbog krhkosti bojanog sloja koji često ostaje sačuvan tek u mikroskopskim tragovima.[4] Razlozi upotrebe polikromije bili su raznovrsni, ali bojila su najvjerojatnije bila odraz ekonomskog statusa naručitelja određene skulpture. Upotreba bojila bila je povezana sa simbolikom boja u ikonografiji.[5] Navedeni razlozi daju joj veliku važnost u klasifikaciji dragocjenih predmeta.[6] Moguće je istražiti posebno značenje različitih boja u mitovima ili ritualima i prikazima društva koja su poznavala pismo i društva s bogatom i detaljnom ikonografskom tradicijom. Razumijevanje simbolike boja teže je u slučajevima pretpovijesnih društava, ali proučavanjem okolnosti i konteksta prikaza ili opažanjem sličnosti prikaza s prirodom moguće je i u nju steći određeni uvid. Kontekst nas može navesti na zaključak da se crveni oker na prikazima ukopa poistovjećuje s krvi, uobičajeno iskustvo nas navodi da je žuta boja povezana sa suncem, a plava s nebom ili morem. Crvena označava dobročinstvo i mučeništvo, pakao, ljubav, mladost, žar, hvalisanje, grijeh i pomirenje.[7] Crvena je boja vrhovničke vlasti u Rimljana, a grimizna crvena boja uzeta je za boju kardinalske odjeće. Prikazi svetog Ivana Evanđelista odjevenog u crveno označavali su njegovu spremnost na djelovanje. Crna boja označava smrt i podzemni svijet, žalovanje, neuspjeh i bolest. U poganskim običajima crna se životinja žrtvovala za umirenje božanstava podzemlja. U kršćanskoj simbolici crna boja je boja kneza tame, a u srednjem vijeku se povezuje s „crnom magijom".[8]

Drevni Egipćani koristili su se polikromijom kako bi ukrasili zidove i stvarali realističnije portrete, zbog čega boja nije bila novi koncept u antičkoj Grčkoj.[9] Grčki umjetnici koristili su paletu živih boja koja se sastojala od žutih, crvenih, zelenih, plavih, smeđih i crnih tonova. Grčke skulpture često imaju tragove pigmenta na kosi, očima, odjeći i ukrasima, ali postoje rijetki dokazi koji ukazuju da se i koža bojala.[10] Rimljani su preferirali pozlatu određenih dijelova skulpture, polikromija se

1 Brinkmann, Wünsche 2007, str. 8; Miliša 2018, str. 46.
2 Brinkmann 2008, str. 30.
3 Deming 2016, str. 28–29.
4 Fejfer 2008, str. 152–157; Ambert, Simpson 2005, str. 1–2.
5 Pliny 1961, str. 263–65; Miliša 2014, str. 175.
6 Østergaard 2018, str. 1; Brecoulaki 2014, str. 4.
7 Gage 1999, str. 109–110.
8 Badurina 2000, str. 208.
9 David 2000, str. 18.
10 Richter, Hall 1944, str. 235–236.

Introduction

Ever since the 18th century, it has been known that Roman sculptures were originally richly decorated with colours, gilding and silver[1]. Roman artists used a wide range of pigments, and authors such as Pliny the Elder[2] and Vitruvius provided important information about such materials.[3] The purpose and importance of polychromy in antiquity, as well as in other artistic periods, is often underestimated and underresearched, due to the fragile nature of coloured layers, which often remain preserved in microscopic traces only.[4] The reasons for the use of polychromy were varied, but the colourants were most likely a reflection of the economic status of the clients in each particular case. The use of colouring matters was associated with the symbolism of colours in iconography.[5] These reasons give it great importance in the classification of valuable objects.[6] It is possible to study the special meanings of various colours in myths or rituals and depictions in literate societies and societies with substantial and detailed iconographic traditions. It is harder to understand the symbolism of colours in the cases of prehistoric societies, but it can be achieved by studying the circumstances and contexts of depictions, or observing their similarities with nature. Contexts may lead us to conclude that red ochre in burial depictions is equated with blood, while common experience tells us that yellow is associated with the sun, and blue with the sky or the sea. Red signifies benevolence and martyrdom, hell, love, youth, zeal, boasting, sin and reconciliation.[7] Red was the colour of supreme power in Rome, while scarlet red was the chosen colour for cardinals' robes. Depictions of St. John the Evangelist dressed in red represented his readiness to act. The colour black signifies death and the underworld, mourning, failure, and disease. In pagan customs, black animals were sacrificed to propitiate the deities of the underworld. In Christian symbolism, black is the colour of the prince of darkness, and in the Middle Ages it was associated with "black magic".[8]

The ancient Egyptians used polychromy to decorate walls and create more realistic portraits. Therefore, colour was not a new concept in ancient Greece.[9] Greek artists used a palette of vivid colours that consisted of yellow, red, green, blue, brown and black tones. Greek sculptures often have traces of pigment on the hair, eyes, clothes and ornaments. Some rare

1 Brinkmann, Wünsche 2007, p. 8; Miliša 2018, p. 46.
2 Brinkmann 2008, p. 30.
3 Deming 2016, pp. 28–29.
4 Fejfer 2008, pp. 152–157; Ambert, Simpson 2005, pp. 1–2.
5 Pliny 1961, pp. 263–65; Miliša 2014, p. 175.
6 Østergaard 2018, p. 1; Brecoulaki 2014, p. 4.
7 Gage 1999, pp. 109–110.
8 Badurina 2000, p. 208.
9 David 2000, p. 18.

nanosila na odjeću, kosu, oči i usne, ali bojanje kože se izbjegavalo. Najcjenjenija boja u rimskoj paleti bila je purpurna, koja je predstavljala pripadnike višeg staleža i bogove.[11]

Gottfried Semper, njemački arhitekt, likovni kritičar i profesor arhitekture, sredinom devetnaestog stoljeća tvrdi da "bijeli mramor nikada nije ostao gol, čak ni u dijelovima koji su izgledali bijelo; ali sloj boje koji je pokrivao kamen bio je više ili manje proziran, kako bi omogućio da se kroz njega nazire bijela boja mramora".[12]

Da su antičke skulpture bile izrađene od čistog bijelog mramora, vjerovalo se sve do 19. stoljeća, kada su kada su zbog povećanog zanimanja za arheološkim iskapanjima otkrivene skulpture s tragovima originalne polikromije.[13] Unatoč čestim raspravama tijekom devetnaestog i dvadesetog stoljeća o postojanju polikromije na antičkim skulpturama, koja je potvrđena brojnim arheološkim dokazima, teško je bilo odagnati percepciju bjeline kamene antičke skulpture.[14] Razlog teškom prihvaćanju polikromije na kamenu mogli bi biti mnogi zapisi iz razdoblja renesanse i ranoga devetnaestog stoljeća koji opisuju bijele, monokromatske antičke skulpture, te bojanje pripisuju drugim, barbarskim kulturama.[15] Iako se stoljećima smatralo da su rimske kamene skulpture bile klesane bez završnog bojanog sloja, razvoj znanosti nam je omogućio jednostavniju i detaljniju analizu skulptura i time otkrivanje polikromije.[16] Brojne suradnje umjetnika i znanstvenika rezultirale su izradom mnogih rekonstrukcija polikromije antičkih djela, a neke od njih su „Treu Head",[17] Partenon u Ateni[18], kip strijelca „Paris".[19] Nažalost, takve suradnje još su uvijek rijetkost; ostaci polikromije na kamenim reljefima i polikromiranim skulpturama rijetko su temeljito obrađeni i rijetko su takva djela evidentirana kao polikromirana.

Rane metode restauracije i čišćenja kao i trendovi u restauraciji uvelike su smanjili sačuvane količine izvorne polikromije,[20] no na sreću neka djela, iako u mikrotragovima, čuvaju važan dokaz svojega izvornog izgleda. Ostatke polikromije teško je sa sigurnošću identificirati i rekonstruirati jer su često mikroskop-

11 Gage 1999, str. 16.
12 Semper 1851, str. 245.
13 Brinkmann 2008, str. 18, 21.
14 Simmons Stager 2012, str. 16.
15 Combs 2012, str. 26.
16 Kiilerich 2016, str. 1.
17 Verri, Opper, Lazzarini 2014, str. 154.
18 Simmons Stager 2012, str. 122–123.
19 Brinkmann, Wünsche 2007, str. 9.
20 Ljudi su se oduvijek brinuli o svojem kulturnom naslijeđu, ali stav prema zaštiti kulturne baštine mijenjao se u različitim razdobljima. Devetnaesto stoljeće bilo je vrijeme romantičnog shvaćanja kulturnog naslijeđa, te je cilj restauracije bio vratiti građevine u pretpostavljene izvorne oblike. Ideja o polikromiranim antičkim skulpturama nije se lako prihvaćala u 19. stoljeću, što je rezultiralo uništenjem polikromije velikog broja antičkih skulptura. Marasović 1983, str. 33–40; Vokić 2007, str. 152–156.

pieces of evidence indicate that the skin was also coloured.[10] The Romans preferred the gilding of certain sculptural features, and applied polychromy to clothes, hair, eyes and lips, but avoided adding colour to the skin. The most valued colour in the Roman palette was purple, which represented members of the upper classes and gods.[11]

Gottfried Semper, a German architect, art critic, and professor of architecture, argued in the mid-nineteenth century that "the white marble never remained naked, not even the parts intended to appear white; but the layer of colour by which they were covered was rendered more or less transparent, to enable the white colour of the marble to appear through it."[12]

Antique sculptures had been believed to have been pure white marble until the nineteenth century, when, due to increased interest in archaeological excavations, sculptures with traces of original polychromy were discovered.[13] Despite frequent debates during the nineteenth and twentieth centuries concerning the existence of polychromy on antique sculptures, as confirmed by myriad archaeological evidence, it was hard to dispel the preconception of the whiteness of antique stone sculptures.[14] The reason for such difficulties in acceptance of polychromy on stone could lie in many records from the Renaissance and early nineteenth century that described white, monochromatic ancient sculptures, and attributed colouring to other, barbaric cultures.[15] Although for centuries it was believed that Roman stone sculptures had been carved without a finishing coat of layer, the development of science has allowed for simpler and more detailed analyses of sculptures and the discovery of polychromy in consequence.[16] Numerous collaborations between artists and scientists have resulted in the production of many reconstructions of polychromy in ancient works, such as the "Treu Head",[17] the Parthenon in Athens[18], the statue of the archer "Paris".[19] Unfortunately, such joint efforts are still a rarity; remnants of polychromy on stone reliefs and polychromatic sculptures are seldom thoroughly attended to, and such works are rarely recorded as polychromatic.

Early restoration and cleaning methods, as well as trends in restoration, substantially reduced the preserved amounts of the original polychromy,[20]. Fortunately, significant evidence

10 Richter, Hall 1944, pp. 235–236.
11 Gage 1999, p. 16.
12 Semper 1851, p. 245.
13 Brinkmann 2008, pp. 18, 21.
14 Simmons Stager 2012, p. 16.
15 Combs 2012, p. 26.
16 Kiilerich 2016, p. 1.
17 Verri, Opper, Lazzarini 2014, p. 154.
18 Simmons Stager 2012, pp. 122–123.
19 Brinkmann, Wünsche 2007, p. 9.
20 People have always cared about their cultural heritage, but the

skih veličina i diskolorirani.[21] Mnoštvo tehnika istraživanja, kao što su mikroskopsko ispitivanje i ultraljubičasta fotografija,[22] te različite vrste kemijskih analiza materijala mogu pružiti vrijednu informaciju o izvornom izgledu nekog djela, kao i o njegovu podrijetlu.[23] No da bi za budućnost bilo tragova za analizu, nerijetko je nužna konsolidacija ostataka polikromiranog sloja na određenim fragmentima. Prilikom konzerviranja-restauriranja umjetnina od kamena zahvat konsolidacije obavlja se samo onda kada je to uistinu prijeko potrebno, kako bi se izbjegla kontaminacija polikromije s konsolidantom; katkad je to, međutim, jedini način sprječavanja degradacije materijala koja se nezaustavljivo događa tijekom vremena.[24]

Danas kemija, osobito analitička kemija, igra ključnu ulogu u određivanju sastava i svojstava materijala, u istraživanju drevnih tehnika izrade te određivanju uzroka i mehanizma degradacije. Nadalje, analitička kemija razvija i procjenjuje djelotvornost restauratorskih materijala i metoda.[25] Jedna od možda najčešće korištenih metoda instrumentalne analize je FT-IR spektroskopija. Odlučili smo koristiti se njome zbog brzine same analize i velike baze podataka analiziranih pigmenata, koji su nam služili za komparaciju rezultata analiza.

Infracrvena spektroskopija (IR) tehnika je analize materijala koja se koristi dulje od 70 godina, a FT-IR (*Fourier Transform Infrared Spectroscopy*) tehnika je koja se gotovo isključivo koristi u suvremenoj IR spektroskopiji. Analiza FT-IR-om djelomično je invazivna metoda analize (ovisno o načinu pripreme uzorka), utoliko što zahtijeva da se uzorak odvoji od predmeta, ali tijekom analize uzorak se ne uništi te ga je moguće pohraniti. U ovom radu korištena je tehnika FT-IR spektroskopije metodom izrade KBr pločice. IR zračenje prolazi kroz uzorak, pri čemu dolazi do apsorpcije dijela spektra, a dobivene informacije obrađuju se računalom.[26] Spektar koji je dobiven predstavlja molekulsku apsorpciju, stvarajući molekulski „otisak prstiju" tvari u uzorku, koji se zatim uspoređuje sa spektrom poznatog spoja.[27] Vrijeme potrebno da se analizira jedan uzorak, svedeno je na samo nekoliko sekunda.[28]

21 Abbe 2007.

22 Metodu detekcije polikromije na kamenu razvio je Vinzenz Brinkmann, koristeći se mikroskopom, bočnim svjetlom i UV svjetlom, fluorescentnim svjetlom i refleksijom. Mikroskop otkriva sitne tragove polikromije i nijansu boje oksidiranih i prljavštinom prikrivenih tragova pigmenta. Bočno svjetlo otkriva male urezane linije ili sloj polikromije na površini kamena. UV svjetlo, flourescentno i reflektirajuće svjetlo osvijetlit će polikromiju koja se slabo vidi golim okom, dok će na ultraljubičastoj fotografiji trag biti oštriji, te je moguće vidjeti tragove polikromije koja je jednom bila prisutna. Combs 2012, str. 32–33.

23 Brinkmann, Koch-Brinkmann 2010. str. 115; Abbe 2011, str. 18.

24 Miliša 2013, str. 228.

25 Mazzeo, Prati, Roda 2011, str. 2885.

26 Stuart 2004, str. 18.

27 Skoog, West, Holler 1999. str. 569.

28 Introduction 2001, str. 2–4.

of the original appearance has been retained in some works, albeit in microtraces. Residues of polychromy are difficult to identify and reconstruct with certainty because they are often microscopic and discoloured.[21] Many research techniques, such as microscopic examination and ultraviolet photography,[22] and various types of chemical analyses of material can provide valuable information about the original appearance of works, as well as their origin.[23] However, in order for there to be traces for future analyses, it is often necessary to consolidate the remains of the polychrome layer on certain fragments. When conserving-restoring stone works of art, the consolidation procedure is carried out only when it is absolutely necessary, in order to avoid contamination of the polychromy with the consolidant. Notwithstanding, sometimes this is the only way to prevent material degradation which occurs inevitably over time.[24]

Nowadays, chemistry, especially analytical chemistry, plays a crucial role in identifying the composition and properties of material, in researching ancient techniques, and in determining the causes and mechanisms of degradation. Furthermore, analytical chemistry develops and evaluates the effectiveness of restoration materials and methods.[25] One of the arguably most commonly used methods of instrumental analysis is FT-IR spectroscopy. We decided to use it because of the speed of the analysis itself and the extensive database of analysed pigments, which served us to compare the results of the analyses.

Infrared spectroscopy (IR) is a material analysis technique which has been used for more than 70 years, while FT-IR (*Fourier Transform Infrared Spectroscopy*) is a technique used almost exclusively in modern IR spectroscopy. FT-IR analysis is a partially invasive method (depending on the method of sample

attitude towards its protection varied in different periods. The nineteenth century was the time of romantic notions of cultural heritage, and the objective of the restoration was to reinstate the presumed original forms of buildings. The idea of ancient polychrome sculptures was not readily accepted in the 19th century, resulting in the destruction of polychromy on a large number of ancient sculptures. Marasović 1983, pp. 33–40; Vokić 2007, pp. 152–156.

21 Abbe 2007.

22 The method of polychromy identification on stone was developed by Vinzenz Brinkmann, using a microscope, oblique light and UV light, fluorescent light and reflection. The microscope detects minute traces of polychromy and the colour shades of oxidised and dirt-covered traces of pigment. The oblique light reveals small incised lines or layers of polychromy on the stone surface. The UV light, fluorescent and reflective light illuminate polychromy which is poorly visible to the naked eye, while ultraviolet photography provides sharper trace, making it possible to see traces of polychromy that existed in the past. Combs 2012, pp. 32–33.

23 Brinkmann, Koch-Brinkmann 2010. p. 115; Abbe 2011, p. 18.

24 Miliša 2013, p. 228.

25 Mazzeo, Prati, Roda 2011, p. 2885.

Opis analiziranih uzoraka

Prikupljeno je pet uzoraka pigmenata sa četiri kamene skulpture,[29] od čega su samo dva uzorka bila prikladna za izradu mikropresjeka i FT-IR-a.[30] Sve četiri kamene skulpture danas se čuvaju u Arheološkome muzeju u Splitu. Dva uzorka crvene boje u obliku praha uzeta su s dvije glave ženskih likova skulptura pronađenih na području Salone te su evidentirani kao uzorak 1 i uzorak 2. Uzorak 1 uzet je s područja bočne lijeve strane punđe mramorne glave božice Venere[31] (sl. 1), datirane u 1. stoljeće (inv. br. AMS 38902), dok je uzorak 2 uzet s cvjetnog vijenca na vrhu glave sa skulpture koja prikazuje božicu Floru[32] (sl. 2), a datira se u razdoblje od 2. do 4. st. (inv. br. AMS C-38).

Uzorak 3 i uzorak 4 uzeti su s kamene stele s prikazom tri ljudske figure[33] (sl. 3) (inv. br. AMS D-410), jedan crvenosmeđe boje u obliku praha, a drugi crne boje u obliku ljuskica. Oba uzorka bila su na istoj figuri, na području desnog oka, i jedini su tragovi polikromije na toj steli. Stela je izrađena od vapnenca između trećega i četvrtog desetljeća 1. stoljeća. Uzorak 5 je jarkocrvene boje, a skinut je s kamenog okvira vrata hrama ili mauzoleja[34] (pentelički mramor) (sl. 4) iz 4. stoljeća pr. Kr. (inv. br. AMS 38040)[35].

Kratak opis metode rada

Analiza polikromije sastojala se od dvije faze. Prva faza odvijala se *in situ*, a uključivala je vizualni pregled kamenih skulptura te su nakon uočavanja tragova polikromije napravljene mikroskopske snimke površine[36] (sl. 5). Po završetku mikroskopskog snimanja uzorak polikromije uzorkovan je čistim skalpelom,

29 Predmet ovog istraživanja (tema ovog članka) nije povijesnoumjetnička i stilska analiza spomenutih skulptura, nego kemijski sastav ostataka izvorne polikromije na njima. No da bismo bolje razumjeli kako je propadao bojani sloj, znanja iz arhiva itekako su korisna.

30 Riječ je o uzorcima veličine ispod 500 mikrona, koji su bili premali za pripremu mikropresjeka jer je njima zaista nemoguće rukovati.

31 Skulptura s koje je uzet uzorak 1 (inv. br. AMS 38902), „glava božice s ostacima boje, Praksitelovih značajki, Salona; mramor; sredina 1. st.", objavljena je u: Cambi 2005, str. 60, sl. 82.

32 Skulptura s koje je uzet uzorak 2 (inv. br. AMS C-38), „glava božice s cvjetnim aranžmanom Praksitelovih značajki, Salona; mramor; posljednja četvrtina 1. st.", objavljena je u: Cambi 2005, str. 70, sl. 98, Cambi 2002, str. 115–174, sl. 38.

33 Skulptura s koje je uzet uzorak 3 i 4 (inv. br. AMS D-410), „ulomak arhitektonske stele, Salona, sjeveroistočna nekropola; vapnenac; 3. – 4. desetljeće 1. st.", objavljena je u: Maršić 2009, sl. 1 – 3.

34 Okvir vrata s kojeg je uzet uzorak 5 (inv. br. AMS 38040), „oplata vrata hrama (ili mauzoleja), vjerojatno Vis (antička *Issa*); pentelički mramor; kasno 4. st. pr. Kr.", objavljeno je u: Kirigin 2008, str. 19–20, kat. br. 9.

35 Berti, Lazzarini 2017, str. 159–169.

36 Pri snimanju fotografija mikroskopom korišten je digitalni mikroskop marke *Dino-Lite Basic* AM2111 *series*, ANMO *Electronic Corporation. Made in Taiwan*.

preparation), insofar as it requires that the sample be removed from the object. However, the sample is not destroyed during the analysis and can be stored. In this research, we used FT-IR spectroscopy with the KBr disk technique. The IR radiation passes through the sample, whereby part of the spectrum is absorbed, and the obtained data is processed by a computer.[26] The resulting spectrum represents molecular absorption, creating a molecular fingerprint of the substance in the sample, which is then compared with the spectrum of a known compound.[27] The time required to analyse one sample is reduced to just a few seconds.[28]

Description of analysed samples

Five samples of pigments were collected from four stone sculptures,[29] of which only two samples were suitable for microsectioning and FT-IR.[30] All four stone sculptures are kept in the Archaeological Museum in Split. Two samples of the red colour in powder form were taken from two female stone heads discovered in the area Salona area and recorded as sample 1 and sample 2. Sample 1 was collected from the left side of the bun on the marble head of the goddess Venus[31] (Fig. 1), dated to the 1st century (Inv. No. AMS 38902), while sample 2 was taken from the wreath of flowers on the top of the head from a sculpture depicting the goddess Flora[32] (Fig. 2), dated to the period from the second to the fourth century (Inv. No. AMS C-38).

Sample 3 and sample 4 were taken from a stone stele showing three human figures[33] (Fig. 3) (Inv. No. AMS D-410),

26 Stuart 2004, p. 18.

27 Skoog, West, Holler 1999. p. 569.

28 Introduction 2001, pp. 2–4.

29 The subject of this research (the topic of this article) is not the historical-artistic and stylistic analysis of the mentioned sculptures, but rather the chemical composition of the remains of original polychromy on them. Notwithstanding, the knowledge from the archives is quite useful for a better understanding of the process of deterioration of the coloured layer.

30 These were samples smaller than 500 microns, which were too small for microsectioning because they were literally impossible to handle.

31 The sculpture from which sample 1 was taken (Inv. No. AMS 38902), "head of a goddess with remnants of colour, characteristics of the style of Praxiteles, Salona; marble; mid-first century", published in: Cambi 2005, p. 60, Fig. 82.

32 The sculpture from which sample 2 was taken (Inv. No. AMS C-38), "head of a goddess with a floral arrangement, characteristics of the style of Praxiteles, Salona; marble; last quarter of the first century", published in: Cambi 2005, p. 70, Fig. 98, Cambi 2002, pp. 115-174, Fig. 38.

33 The sculpture from which samples 3 and 4 were taken (Inv. No. AMS D-410), "fragment of an architectural stele, Salona, north-eastern necropolis; limestone; third–fourth decade of the 1st century", published in: Maršić 2009, Figs. 1–3.

Slika 1.
Prednja i bočna strana skulpture božice Venere; mjesto uzorkovanja označeno je crvenom kružnicom. Arheološki muzej u Splitu, inv. br. AMS 38902 (foto: Miona Miliša)

Figure 1.
Front and side of the sculpture of the goddess Venus; the sampling point is marked with a red circle. Archaeological Museum in Split, Inv. No. AMS 38902 (photograph: Miona Miliša)

Slika 2.
Prednja i bočna strana skulpture božice Flore; mjesto uzorkovanja označeno je crvenom kružnicom. Arheološki muzej u Splitu, AMS C-38 (foto: Miona Miliša)

Figure 2.
Front and side of the sculpture of the goddess Flora; the sampling point is marked with a red circle. Archaeological Museum in Split, AMS C-38 (photograph: Miona Miliša)

Slika 3.
Prednja strana stele s prikazom tri ljudske figure; na desnoj strani slike vide se ostaci boje na području lijevog oka s kojeg su uzorkovani uzorci 3 i 4. Arheološki muzej u Splitu, AMS D-410 (foto: Miona Miliša)

Figure 3.
Front of the stele depicting three human figures; on the right: remnants of colour can be seen in the area of the left eye from which samples 3 and 4 were taken. Archaeological Museum in Split, AMS D-410 (photograph: Miona Miliša)

pri čemu se nastojalo sastrugati samo pigment. Kod uzoraka u obliku ljuskica, uzetih s kamene stele sa tri ljudske figure, bilo je nemoguće uzeti samo sloj polikromije, zbog iznimno tankog obojanog sloja. U uzorcima je osim sloja polikromije bila i velika količina kamene podloge, koja je po sastavu $CaCO_3$. Druga faza odvijala se u Laboratoriju za prirodoznanstvena istraži-

one reddish-brown in powder form, and the other black in the form o flakes. Both samples originate from the area of the right eye of the same figure, and are the only traces of polychromy on that stele. The stele was made of limestone between the third and fourth decades of the first century. Sample 5 is bright red, and was taken from the stone frame of

Slika 4.
Prednja strana okvira vrata hrama ili mauzoleja; na desnoj strani fotografije je detalj reljefa s mjesta uzorkovanja. Arheološki muzej u Splitu, AMS 38040 (foto: Miona Miliša)

Figure 4.
Front of the door frame of a temple or mausoleum; on the right: a detail of the relief with the sampling point is shown. Archaeological Museum in Split, AMS 38040 (photograph: Miona Miliša)

vanja u konzervaciji-restauraciji na Umjetničkoj akademiji u Splitu te je uključivala analizu uzoraka FT-IR spektroskopom i izradu mikropresjeka obojanog sloja uzoraka 4 i 5 u epoksidnoj smoli.[37]

FT-IR spektroskopija provedena je na spektrometru *Shimadzu* FT-IR 8400S. Uzorak pigmenta pomiješan je s kalijevim bromidom (KBr), usitnjen u tarioniku te je prešanjem izrađena tanka pločica, tzv. pastila. Prije analize svakog uzorka snimljen je pozadinski spektar. Spektri pigmenta snimani su u rasponu od 4000 do 400 cm^{-1} te su uspoređeni s bazom standarda spektara pigmenata.[38]

Rezultati analiza

Crvena boja s glave božice Venere, evidentirana kao uzorak 1, uzeta je u obliku praha, te je analizirana FT-IR spektroskopijom. Mikrosnimke površine traga polikromije pokazale su crveno-narančasti bojani sloj na kamenu. Površina skulpture ne sadrži tragove nečistoća ili patine, što upućuje na zaključak da

37 Voditelj Laboratorija za prirodoznanstvena istraživanja u konzervaciji-restauraciji na Umjetničkoj akademiji u Splitu je izv. prof. dr. sc. Ivica Ljubenkov, pročelnik Odjela za kemiju Prirodoslovno-matematičkog fakulteta u Splitu.

38 Baza standarda spektara pigmenata nalazi se u Laboratoriju (vidjeti prethodnu bilješku). Snimljeno je 32 spektra svakog uzorka u rezoluciji od 4 cm^{-1}.

the door of the temple or mausoleum[34] (Pentelic marble) (Fig. 4) from the fourth century BC (Inv. No. AMS 38040)[35].

Brief description of the method

The polychromy analysis consisted of two phases. Phase one took place *in situ*, and included a visual inspection of the stone sculptures. Having detected traces of polychromy, microscopic images of the surface were made[36] (Fig. 5). Upon completion of microscopic imaging, a polychromy sample was sampled with a clean scalpel, wherein it was attempted to scrape off the pigment only. In the case of flake samples, taken from the stone stele with three human figures, it was impossible to withdraw the layer of polychromy only, since the layer of colour was extremely thin. In addition to the polychromy layer, the samples also contained a quantity of stone substrate, $CaCO_3$ in terms of composition. Phase two was carried out in the Laboratory for Natural Science Research in Conservation-Restoration at the Academy of Arts in Split and included analysis of samples by FT-IR spectroscopy and microsectioning of the coloured layer of samples 4 and 5 in epoxy resin.[37]

FT-IR spectroscopy was performed on a *Shimadzu* FT-IR 8400S spectrometer. The pigment sample was mixed with potassium bromide (KBr), ground in a mortar and pressed to form a thin plate, i.e. pellet. A background spectrum was recorded before analysing each sample. The pigment spectra were recorded in the range from 4000 to 400 cm^{-1} and compared with a database of spectral standards.[38]

Results of the analyses

The red colour from the head of the goddess Venus, recorded as sample 1, was taken in powder form and analysed by FT-IR spectroscopy. Micrographs of the polychromy trace surface showed a red-orange layer of colour on the stone. The surface of the sculpture does not contain traces of dirt or patina, which suggests that the sculpture was cleaned in the past.[39] From

34 The door frame from which sample 5 was taken (Inv. No. AMS 38040), "door frame of a temple (or mausoleum), probably Vis (ancient *Issa*); Pentelic marble; late fourth century BC", published in: Kirigin 2008, pp. 19–20, Cat. No. 9.

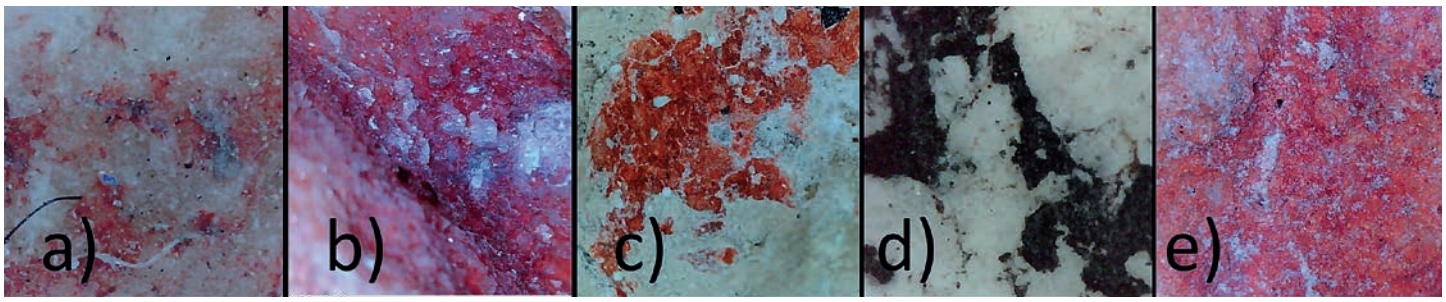
35 Berti, Lazzarini 2017, pp. 159–169.

36 The photographs were taken using the digital microscope *Dino-Lite Basic* AM2111 *series*, ANMO *Electronic Corporation. Made in Taiwan*.

37 The manager of the Laboratory for Natural Science Research in Conservation-Restoration at the Academy of Arts in Split is Ivica Ljubenkov, PhD, Assoc. Prof., Head of the Department of Chemistry, Faculty of Natural Sciences and Mathematics in Split.

38 The database of spectral standards is located in the Laboratory (see the previous footnote). Thirty-two spectra per sample were recorded at a resolution of 4 cm^{-1}.

39 Most of the ancient sculptures exhibited in the interior of the building were cleaned several times in the past, most recently in the



Slika 5.
Mikroskopske snimke uzoraka: a) crveni pigment sa skulpture božice Venere; b) crveni pigment sa skulpture božice Flore; c) crveno-smeđi pigment sa stele; d) crni pigment sa stele; e) crveni pigment s okvira vrata hrama ili mauzoleja (analizirala Josipa Marić na Umjetničkoj akademiji u Splitu)

Figure 5.
Microscopic images of samples: a) red pigment from the sculpture of the goddess Venus; b) red pigment from the sculpture of the goddess Flora; c) red-brown pigment from the stele; d) black pigment from the stele; e) red pigment from the door frame of a temple or mausoleum (analysed by Josipa Marić at the Academy of Arts in Split)

je skulptura nekad u prošlosti čišćena.[39] Od vremena nastanka antičke skulpture, u njezinom izvornom bojanom izdanju do našeg vremena, sama kamena umjetnina zasigurno je bila izložena raznim utjecajima u različitim okolišima. Tragovi vremena ostavili su svoje ožiljke u obliku raznih mehaničkih oštećenja, erozije površine zbog utjecaja atmosferilija i sl. Sve to je izazvalo propadanje izvorne boje, a na koncu i velike promjene u razdoblju od prvotnog izgleda kamene epiderme do onoga što vidimo danas. FT-IR analizom nije se uspjelo doći do identiteta pigmenta zato što određene tvari ne pokazuju apsorpciju u rasponu od 4000 do 400 cm^{-1} (sl. 6). Neki crveni pigmenti, kao cinober, apsorbiraju se u području ispod 400 cm^{-1}.

Uzorak 2 uzorak je crvene boje s glave božice Flore. Uzorak je uzet iz udubina između cvjetova na obruču, jer je tu ostalo najviše sačuvanog pigmenta.[40] Mikrosnimke su pokazale intenzivno crveni bojani sloj, s točkastim primjesama plavog pigmenta te površinske nečistoće kamena. FT-IR analizom došlo se do zaključka da je riječ o pigmentu na bazi željeznog oksida.

Usporedbom sa spektrima pigmenata na bazi željeznih oksida iz baze podataka Laboratorija za prirodoznanstvena istraživanja u konzervaciji-restauraciji, pri Odsjeku konzervacije i restauracije Umjetničke akademije Sveučilišta u Splitu, apsorpcijske vrpce pri 1099 i 1024 cm^{-1} i intenzitet signala ukazuju na manju količinu željeznih oksida. Usporedbom sa spektrima iz baze podataka jasno je vidljivo da pigment nije pečen, zbog odsutnosti signala u području 650 – 500 cm^{-1} (sl. 7).

Uspoređivanjem spektara uzoraka 1 i 2 ustanovilo se kako je riječ o istom pigmentu.

Crna boja uzeta s kamene stele s tri ljudske figure eviden-

the time of its creation, in its original coloured configuration, the ancient stone work of art must have been exposed to different influences in various environments. Time has taken its toll in the form of various mechanical damage, surface erosion due to weathering, etc. All this caused the decay of the original colour, and ultimately major changes from the period of the original appearance of the stone epidermis to what one can see today. FT-IR analysis did not reveal pigment identity because certain substances do not show absorption in the range of 4000 to 400 cm^{-1} (Fig. 6). Some red pigments, such as vermilion, are absorbed below 400 cm^{-1}.

Sample 2 is a specimen of the red colour from the head of the goddess Flora. The sample was taken from the hollows between the flowers on the band, because this is where most of the preserved pigment remained.[40] Micrographs showed a layer of intense red colour, with spotty tinges of blue pigment and surface impurities of the stone. FT-IR analysis concluded that it was a pigment based on iron oxide.

In comparison with the spectra of pigments based on iron oxides from the database of the Laboratory for Natural Science Research in Conservation-Restoration of the Conservation-Restoration Department at the Academy of Arts in Split, University of Split, the absorption bands at 1099 and 1024 cm^{-1} and the signal intensity indicate a small amount of iron oxides. The comparison with the spectra from the database clearly shows that the pigment is not fired, due to the absence of signal in the range of 650–500 cm^{-1} (Fig. 7).

By comparing the spectra of samples 1 and 2, they were found to be the same pigment.

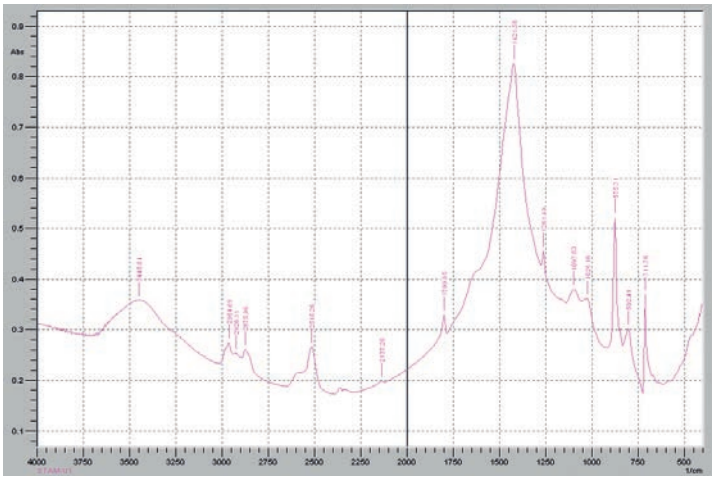
The black colour taken from the stone stele with three

39 Većina skulptura antičkog postava izložena u interijeru zgrade bila je čišćena u nekoliko navrata u prošlosti, zadnji put krajem prošlog stoljeća (1990-ih godina). Ovim putem zahvaljujemo kolegi Ivi Donelliju, koji je radio u restauratorskoj radionici Arheološkog muzeja u Splitu (od 1993. do 2005. godine), na pruženim informacijama.

40 Burgess 1990, str. 47–52.

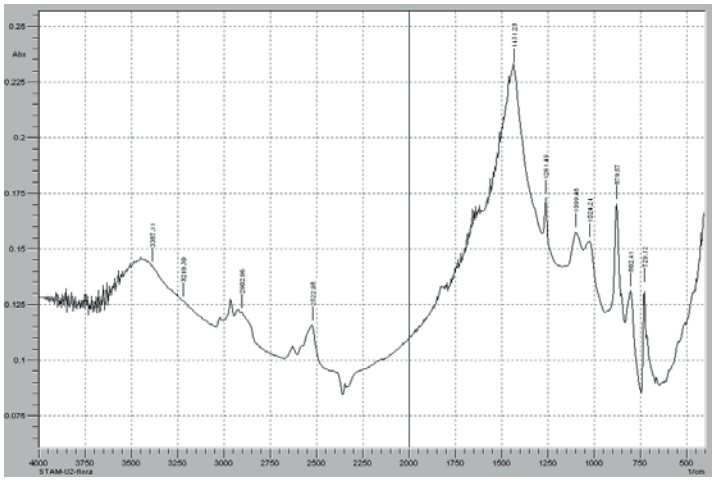
late last century (the 1990s). We would hereby like to thank our colleague Ivo Donelli, who worked in the restoration workshop of the Archaeological Museum in Split (from 1993 to 2005), for the information he provided.

40 Burgess 1990, pp. 47–52.



Slika 6.
IR spektar uzorka 1 (analizirao Ivica Ljubenkov na Umjetničkoj akademiji u Splitu)

Figure 6.
IR spectrum of sample 1 (analysed by Ivica Ljubenkov at the Academy of Arts in Split)



Slika 7.
IR spektar uzorka 2 (analizirao Ivica Ljubenkov na Umjetničkoj akademiji u Splitu)

Figure 7.
IR spectrum of sample 2 (analysed by Ivica Ljubenkov at the Academy of Arts in Split)

tirana je kao uzorak 3 te je uzeta u obliku ljuskice kojom je pripremljen mikropresjek u epoksidnoj smoli i tako se sačuvao za sljedeće generacije zbog uistinu male količine dostupnog pigmenta. Mikrosnimkom (u povećanju od 200 x) bilo je moguće uočiti samo crni bojani sloj na površini kamena bez nečistoća. Mikrosnimka presjeka prikazala je uzorak bojanog sloja zajedno s površinom kamena. Trag pigmenta vidljiv je tek na malom dijelu uzorka, zbog čega klasična analiza mikropresjeka nije bila moguća[41]. Uzrok tome jest to što je bojani sloj nanesen u iznimno tankom premazu (sl. 8a).

Crveno-smeđa boja uzeta s kamene stele s tri ljudske figure evidentirana je kao uzorak 4. Uzorak je uzet u obliku praha s lakrimalne kosti prve ljudske figure s lijeve strane. Analizom mikrosnimke primjećuje se intenzivno crvena boja bojanog sloja s mjestimičnim tamnocrvenim područjima i točkastim primjesama crne boje. FT-IR analizom uzorka spektar je pokazao veliku količinu gipsa, zbog apsorpcijskih vrpca pri 1116, 1082 i 1033 cm^{-1} koje se mogu pripisati asimetričnom svijanju i simetričnom produžavanju veza u sulfatnoj skupini (sl. 8b).

Uzorak 5 je crvena boja uzeta s okvira vrata hrama ili mauzoleja. S predmeta je preuzeta mala količina uzorka u obliku ljuskice te kao prah na polovici visine desne lateralne strane okvira. Mikrosnimke su pokazale intenzivnu tamnocrvenu boju bojanog sloja s površinskim nečistoćama (sl. 5e), dok se na mikrosnimkama presjeka (sl. 9a) može uočiti bojani sloj intenzivno crvene boje s nečistoćama (bijele čestice, vrlo vjerojatno vapnenca). FT-IR analiza pokazala je spektar gotovo istovjetan onome venecijanskog crvenog pigmenta. To potvrđuju pre-

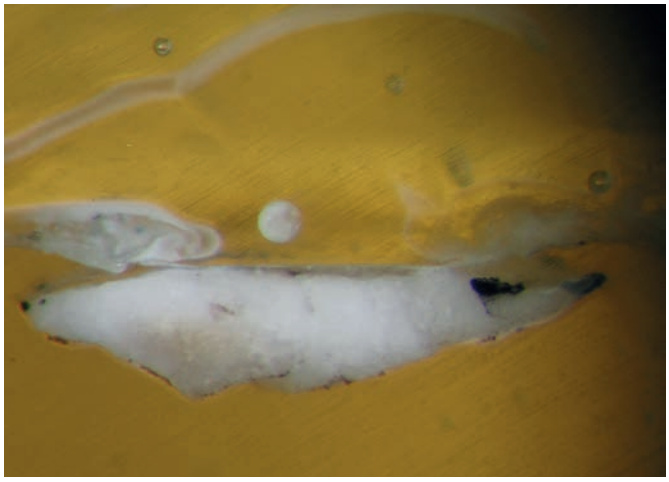
human figures was recorded as sample 3 and collected in the form of a flake for microsectioning in epoxy resin. In this way, it is preserved for future generations, given the really small amount of available pigment. The micrograph (at a magnification of 200 x) made it possible to discern only a layer of black colour on the stone surface without impurities. The cross-sectional micrograph showed a pattern of the coloured layer together with the surface of the stone. Traces of the pigment are visible only in a small part of the sample, which is why classic microsection analysis was not possible[41]. The reason for this lied in the particularly thin coat of the coloured layer (Fig. 8a).

The red-brown colour taken from the stone stela with three human figures was recorded as sample 4. The sample was taken in powder form from the lacrimal bone of the first human figure on the left. The analysed micrograph showed an intense red colour of the painted layer with occasional dark red areas and spotty black tinges. The FT-IR analysis of the sample showed a large amount of gypsum, due to the absorption bands at 1116, 1082 and 1033 cm^{-1}, which can be attributed to asymmetric bending and symmetric stretching of bonds in the sulphate group (Fig. 8b) .

Sample 5 is a specimen of red colour taken from the door frame of a temple or mausoleum. A small amount of sample was taken in the form of a scale and as powder at half-height of the right lateral side of the frame. The micrographs revealed an intense dark red colour of the painted layer with surface impurities (Fig. 5e), while the micrographs of the cross section

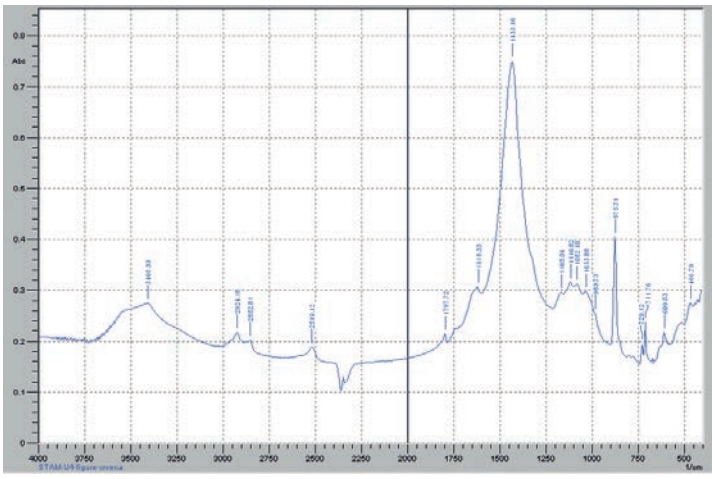
41 Na žalost, tehniku SEM/EDS nismo bili u mogućnosti koristiti.

41 Unfortunately, we were not able to use the SEM/EDS technique.



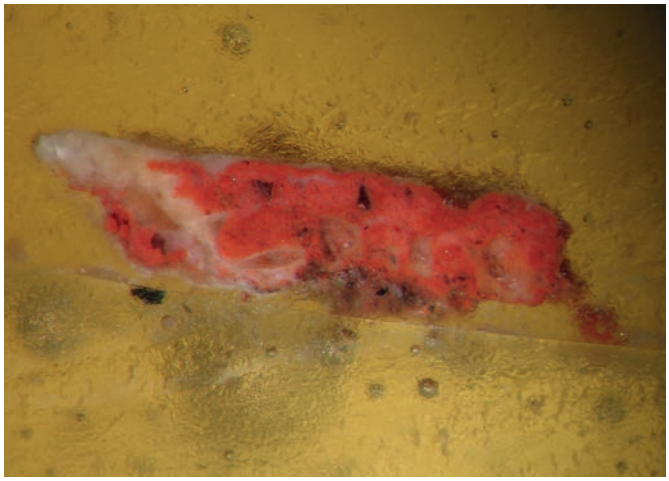
Slika 8a.
Mikrosnimka presjeka uzorka 4, u povećanju od 200 x (analizirao Ivica Ljubenkov na Umjetničkoj akademiji u Splitu)

Figure 8a.
Micrograph of cross-section of sample 4, at a magnification of 200 x (analysed by Ivica Ljubenkov at the Academy of Arts in Split)



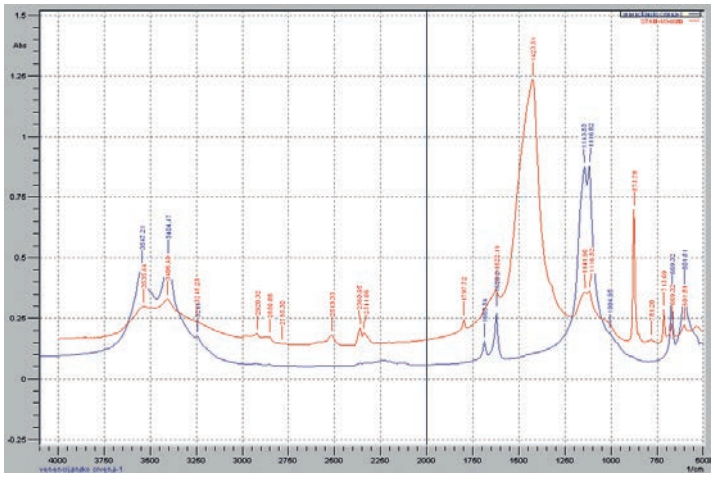
Slika 8b.
IR spektar uzorka 4 (analizirao Ivica Ljubenkov na Umjetničkoj akademiji u Splitu)

Figure 8b.
IR spectrum of sample 4 (analysed by Ivica Ljubenkov at the Academy of Arts in Split)



Slika 9a.
Mikrosnimka presjeka uzorka 5, u povećanju od 200 x (analizirao Ivica Ljubenkov na Umjetničkoj akademiji u Splitu)

Figure 9a.
Micrograph of cross-section of sample 5, at a magnification of 200 x (analysed by Ivica Ljubenkov at the Academy of Arts in Split)



Slika 9b.
Preklopljeni IR spektar uzorka 5 i venecijanskog crvenog pigmenta (analizirao Ivica Ljubenkov na Umjetničkoj akademiji u Splitu)

Figure 9b.
Overlapping IR spectrum of sample 5 and Venetian red pigment (analysed by Ivica Ljubenkov at the Academy of Arts in Split)

klapanja apsorpcijskih vrpci pri 1141, 1116, 669 i 601 cm^{-1}. Sve navedene vrijednosti ovih vrpci su one kalcij sulfata dihidrata, odnosno gipsa. Gips je u pigmentu koji se naziva venecijanska crvena punilo, a pigmentirajuća tvar u toj mješavini je željezni oksid čije vrpce u spektru (sl. 9) nisu vidljive. Time je pokazano da se ne radi o okeru, nego o venecijanskoj crvenoj. Signali pri 1423, 873 i 713 cm^{-1} pokazuju prisutnost $CaCO_3$, koji je gotovo neizbježan kod uzimanja uzoraka s kamena (sl. 9b).

(Fig. 9a) show a layer of intense red colour with impurities (white particles, probably limestone). FT-IR analysis showed a spectrum almost identical to that of the Venetian red pigment. This was confirmed by the overlaps of the absorption bands at 1141, 1116, 669 and 601 cm^{-1}. All the above values of the tapes were those of calcium sulphate dihydrate or gypsum. Gypsum is in the pigment called Venetian red, and the pigmenting substance in this mixture is iron oxide, whose bands in the spectrum (Fig. 9) are not visible. This showed that it was not ochre, but Venetian red. The signals at 1423, 873, and 713

Zaključak

Velik broj istraživanja i objavljenih radova u posljednjih deset godina posvećen je ostacima polikromije na kamenoj skulpturi. Originalni polikromirani sloj rijetko je ostao sačuvan do današnjih dana, uglavnom se nalazi u mikroskopskim tragovima, zbog čega se često zaboravlja da su sve kamene skulpture i dekorativni arhitektonski elementi iz antike, ali i drugih razdoblja uglavnom bili bogato bojani. Polikromija je još uvijek nedovoljno istražena te ne postoji mnogo znanstvenih istraživanja na hrvatskom jeziku koja se bave problematikom polikromije. Svaka publikacija na ovu temu uvelike pridonosi razvoju znanja o polikromiji, ali i razvoju metoda restauracije i konzervacije. Problematika polikromije nije privukla osobitu pozornost povjesničara umjetnosti i muzeologa, najčešće zbog nedostatka metode detekcije boje na skulpturama i neznanja da ona uopće postoji na određenim umjetničkim djelima. Tehnika detekcije pigmenata koju je razvio Vinzenz Brinkmann 1990-ih godina, pokazala se učinkovitom i jednostavnom za izvedbu na terenu.[42] Spominjući metodu otkrivanja pigmenata, ne može se ne spomenuti degradacija polikromije koja se nezaustavljivo događa tijekom stoljeća. Polikromija pruža jedinstvenu i korisnu informaciju o djelu, ali ako se ne konsolidira, bit će izgubljena zauvijek. Nužno je sačuvati tragove polikromije za sljedeće generacije, odnosno za buduća istraživanja, kada analitičke metode budu naprednije. Nadalje, potrebno je spomenuti da metoda analize FT-IR spektroskopijom u ovom slučaju nije pružila potpune informacije: tri uzorka od pet nismo uspjeli identificirati. Analiza XRF-om korisna je i praktična, jer se bojani sloj na kamenu ne oštećuje, te bi mogla pokazati detaljniju identifikaciju analiziranih pigmenata na ovim skulpturama.

42 Brinkmann, Koch-Brinkmann 2010, str. 114–116.

cm^{-1} indicate the presence of $CaCO_3$, which is almost inevitable in stone-sampling (Fig. 9b).

Conclusion

A large number of researches and published papers in the last ten years have been dedicated to remains of polychromy on stone sculptures. The original polychrome layer has rarely been preserved to this day, and can mostly be found in microscopic traces. This is why it is often forgotten that all stone sculptures and decorative architectural elements from antiquity, as well as from other periods, were usually richly coloured. Polychromy is still insufficiently researched, and there are not many scientific studies in the Croatian language that deal with polychromy. Each publication on this topic greatly contributes to the development of knowledge about polychromy, as well as the development of methods of restoration and conservation. The matter of polychromy has not attracted much attention from art historians and museologists, most often due to the lack of methods for detecting colour on sculptures and ignorance of its very existence on certain works of art. The pigment identification technique developed by Vinzenz Brinkmann in the 1990s has proved to be effective and easy to use in the field.[42] When reference is made to the method of pigment identification, one cannot fail to mention the degradation of polychromy, which has been inevitable over the centuries. Polychromy provides unique and useful information about works of art, but if it is not consolidated, it will be lost forever. It is necessary to preserve traces of polychromy for the generations to come, i.e. for future research, when analytical methods are more advanced. Furthermore, it should be mentioned that the method of analysis by means of FT-IR spectroscopy did not provide complete information in this case: we were unable to identify three samples out of five. XRF analysis is useful and practical, because the coloured layer on the stone is not damaged, and it could provide more detailed identification of analysed pigments on such sculptures.

Prijevod** / **Translation: Denis Gracin

42 Brinkmann, Koch-Brinkmann 2010, pp. 114–116.

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