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The potential of radiocarbon analysis for the detection of art forgeries

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ABSTRACT

Art objects form an essential part of cultural heritage and are appreciated for their artistic values. However, the observed investment in art and capacity for high monetary returns encourages counterfeiting of art objects. The art market's lack of transparency and traditional confidential protocols amplifies the problem. Radiocarbon analysis provides a tool to detect anachronistic materials. Measurement of bomb peak radiocarbon, which was observed in the atmosphere during the last 70 years, can provide clear evidence of post-1950 material. Here we briefly introduce the method and discuss its application in detecting forgeries. Three accelerator mass spectrometry AMS laboratories performed a ^{14}C dating inter-comparison study on the material used in art. Results obtained on modern cotton paper, two antique sheets of paper, one parchment, and one textile demonstrate the radiocarbon dating capacity to date the material accurately. The excellent agreement between laboratories is crucial for the broader application of this scientific tool in forensic studies and court cases.

1. Introduction

Art is an integral part of human history and is valued chiefly for its aesthetic qualities. A portion of fine art, such as paintings and sculptures, is open to the public and can be appreciated in museums and art galleries worldwide. However, fine art is also a commodity and is considered an attractive investment [1]. Due to the unique role of art, the involvement of connoisseurs and collectors, and its monetary values, fine art is the target of counterfeit. The forgers might be just individuals who might have different motives, with financial returns prevailing. For example, in the early 20th century, Han van Megeeren forged Vermeer to deceive the connoisseur but he also made considerable profits [2]. As the Knoedler art gallery case illustrates, a more organized scheme cannot be excluded [3] and tracking down forgers and their networks might be difficult due to the lack of transparency characteristic of the art market [4]. Nevertheless, awareness is growing among potential buyers who are seeking advice in art research.

Radiocarbon analysis is a powerful method to detect anachronistic materials in fine art. The technique relies on the fact that the material used by artists is plant or animal-based and registers a radiocarbon signature of harvest time or death. It is important to note that radiocarbon dating does not provide temporal information on the time of artwork creation. Moreover, it cannot detect material recycling, such as the reuse of parchment or canvas.

The radioactive isotope of carbon (^{14}C) is produced in the atmosphere by the interaction of cosmic rays with the atmosphere. Only one ^{14}C atom is present for trillion atoms of stable carbon ^{12}C. However, they too are incorporated the plants, shells, corals, and any carbon-bearing material commonly present in nature [5]. The decay of ^{14}C ($T_{1/2}$ = 5700 ± 30 years) makes it an excellent clock to be used for the past 55,000 years. Radiocarbon dating of artwork using the technique developed by Libby in 1949 [6] was problematic because it required grams of organic matter to count the beta decay of ^{14}C. However, after 1977 separation of isotopic masses and detection of low concentrations of ^{14}C (^{14}C /^{12}C on the level of 10^{-12} to 10^{-15}) using Accelerator Mass Spectrometry (AMS) became possible [7]. The technique allowed the analysis of samples containing as little as one milligram of carbon, i.e., a few milligrams of canvas or wood.

The development revolutionized the field of radiocarbon applications and provided an opportunity to date objects of art. One of the main challenges in ^{14}C dating of artworks is the contamination of samples with old (^{14}C free) carbon due to conservation. It often uses products made of fossil carbon, which is million years old; therefore, all ^{14}C had enough time to decay. Despite being destructive, AMS radiocarbon dating can most effectively detect material formed after

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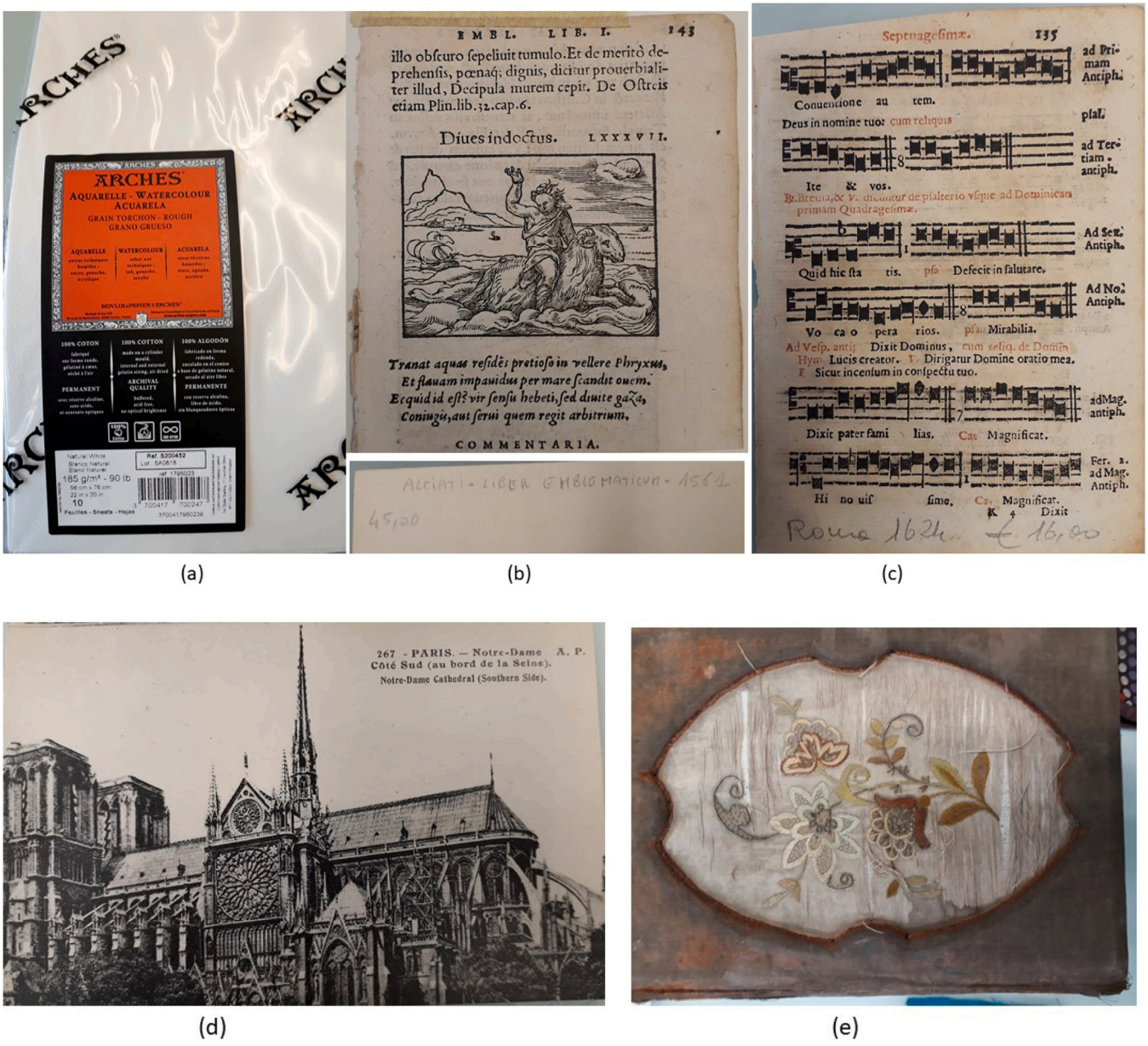

Fig. 1. Objects sampled for inter-comparison AMS analysis (a) Arches® paper, (b) antique paper '1561 CE', (c) antique paper 'Rome1624 CE' (d) old postcard 'Notre-Dame de Paris' (e) textile decoration of the old box.

1950 CE (CE - Common Era). In 1952 the first nuclear tests of the hydrogen bomb performed in the stratosphere led to the artificial production of ^{14}C in the atmosphere. After the Partial Nuclear Test Ban Treaty in 1963, the high levels (nearly double the natural) began to decline, partly due to redistribution between carbon reservoirs, and in the last few decades due to the overwhelming effect of fossil fuel combustion (Suess effect) [8,9]. Currently, the atmospheric ^{14}C concentration is near the natural levels at non-urban locations, i.e., remote regions free of emissions of fossil fuels [10].

Consequently, the future living organisms and material produced from plant and animal tissue will have apparent ^{14}C ages corresponding to the pre-1950 CE times. The two anthropogenic effects add to natural changes observed in levels of atmospheric ^{14}C. The natural variability is due to changes in the ^{14}C production rate and changes in the global carbon cycle. The variability of ^{14}C observed during 55,000 years and its effect on the radiocarbon ages can be compensated by calibration [11]. The calibration curve compiles measurements of ^{14}C content in independently dated archives such as tree rings [12].

Application of the bomb peak ^{14}C analysis to detect forgeries was recognized early on by the antiquities trade, which developed a practice of screening the artifacts by commissioning ^{14}C analysis from research laboratories [13]. However, currently, more than 45 radiocarbon laboratories follow guidelines published on the web page of the Radiocarbon journal. The guidelines require information on the dated objects due diligence to prevent radiocarbon dating for illicit antiquity trade [14].

Like the antiquity trade, the fine arts market lacks transparency, but it has different mechanisms for art authentication. Often, connoisseur expertize and non-destructive analysis are the first choices in art authentication. Radiocarbon dating is employed when no conclusive answers can be found. The use of bomb peak ^{14}C analysis to detect forgeries gained momentum after copies of well-known artists showed its post-1950 signature [15,16]. However, some

Table 1
Results of ^{14}C AMS analysis from three laboratories (DeA-Debrecen, LTL-Lecce, ETH Zurich).

Sample#	Object	Material	AMS lab code	$F^{14}C$	± 1σ	^{14}C age (BP)	± 1σ
1	Arches /AS1	paper	DeA-19196	1.0215	0.0039	-171	31
			LTL19155A	1.0184	0.0045	-146	35
			ETH-95362	1.0140	0.0028	-111	23
		χ^2-Test: df= 2 T = 2.5(5% 6.0)		**1.0170**	**0.0020**	**-136**	**17**
2	Arches/AS2	paper	DeA-19197	1.0232	0.0039	-184	31
			LTL19156A	1.0140	0.0046	-112	36
			ETH-95363	1.0170	0.0028	-132	22
		χ^2-Test: df= 2 T = 2.7(5% 6.0)		**1.0181**	**0.0020**	**-142**	**17**
1&2	**AS1&AS2**	**χ^2-Test: df= 5 T = 5.3(5% 11.1)**		**1.0175**	**0.001**	**-139**	**12**
3	1561 CE	paper	DeA-19198	0.9672	0.0037	268	31
			LTL19158A	0.9661	0.0047	277	39
			ETH-95364	0.9600	0.0027	326	23
		χ^2-Test: df= 2 T = 2.7(5% 6.0)		**0.9631**	**0.0020**	**300**	**17**
4	Rome1624 CE	paper	DeA-19199	0.9561	0.0037	360	31
			LTL19157A	0.9574	0.0038	350	32
			ETH-95365	0.9520	0.0027	393	23
		χ^2-Test: df= 2 T = 1.4(5% 6.0)		**0.9544**	**0.0020**	**374**	**16**
5	Textile	textile	DeA-19232	0.9823	0.0043	143	35
			LTL19161A	0.9794	0.0049	167	40
			ETH-95366	0.9830	0.0027	139	22
		χ^2-Test: df= 2 T = 0.4(5% 6.0)		**0.9822**	**0.0020**	**145**	**17**
6	Postcard	paper	DeA-19200	0.9865	0.0038	109	31
			LTL19160A	0.9790	0.0032	170	26
			ETH-95367	0.9810	0.0028	151	23
		χ^2-Test: df= 2 T = 2.3(5% 6.0)		**0.9816**	**0.0020**	**148**	**16**
7	Parchment	parchment	DeA-19233	0.9815	0.0043	150	35
			LTL19159A	0.9833	0.0049	135	40
			ETH-95369	0.9810	0.0027	151	22
		χ^2-Test: df= 2 T = 0.1(5% 6.0)		**0.9815**	**0.002**	**148**	**17**

recycling of painting's support: canvas, or wood is feasible and practiced even before the advent of ^{14}C dating. Such cases might still be detected with the help of dating the binder used in paint [17], as proposed by Keisch and Miller [18] and shown by Hendriks et al. [19].

The potential of the AMS ^{14}C tool to detect counterfeit is not yet fully appreciated, primarily due to the lack of data showing its effectiveness. Most art research laboratories cannot publish their results due to confidentiality agreements. Nevertheless, few published cases have demonstrated the power of the method. For example, bomb peak dating of cotton paper from authentic and alleged T'ang Haywen (1927–1991) paintings documented the counterfeit [20]. These results supported a court case against the art dealer selling forged paintings. One of the arguments raised against ^{14}C dating results was that the potential differences between laboratories might exist. However, radiocarbon laboratories perform numerous inter-comparison studies on various materials from various periods, i.e., different ^{14}C concentrations [21].

Here we present the inter-comparison results performed by three laboratories on materials typical for art objects. Samples of paper, parchment, and textiles were analyzed using the AMS ^{14}C and showed no offset between laboratories.

2. Material and methods

2.1. Samples choice and sampling

Selections of samples for this inter-comparison study were based on the availability of sufficient amounts of material. A sheet of Arches® cotton paper was purchased in 2018 for this study (Fig. 1a).

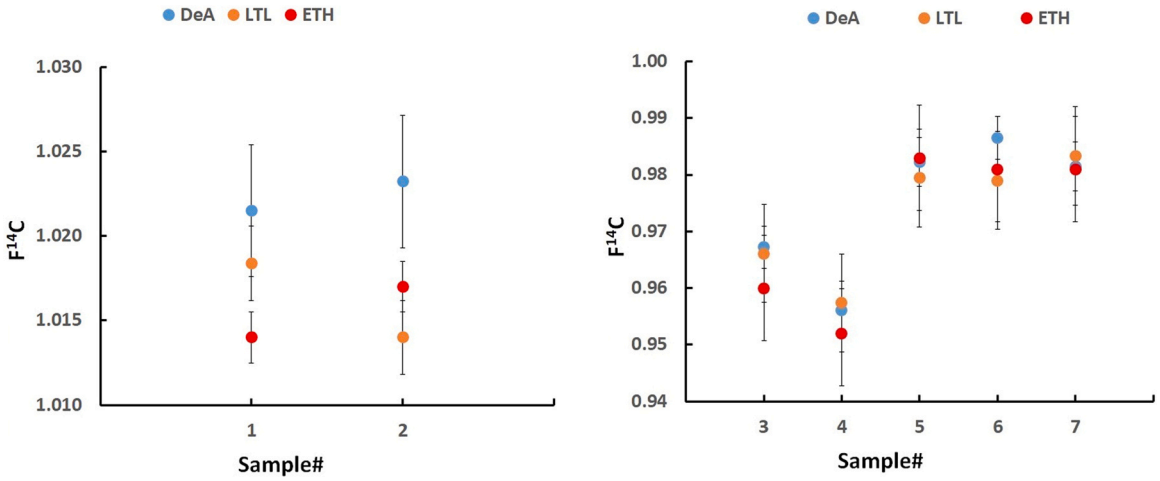

Fig. 2. Results of AMS ^{14}C analysis ($F^{14}C$ ± 1σ) obtained by AMS laboratories (DeA-Debrecen, LTL- Lecce, ETH- Zurich) for samples listed in Table 1 (a) Arches® paper (samples #1 and #2). (b) paper '1561 CE' (sample #3), paper 'Rome 1621' (sample #4), textile (sample #5), postcard (sample #6) and parchment (sample #7).

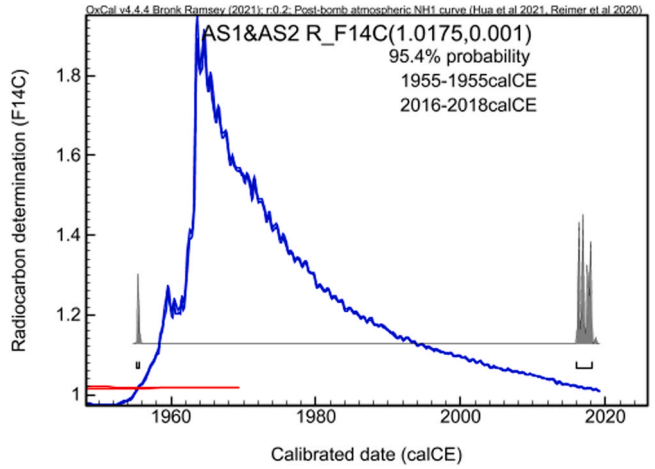

Fig. 3. Calibration using bomb^{14}C curve [10]. (a) combined analysis on Arches® paper; the purchase year was 2018.

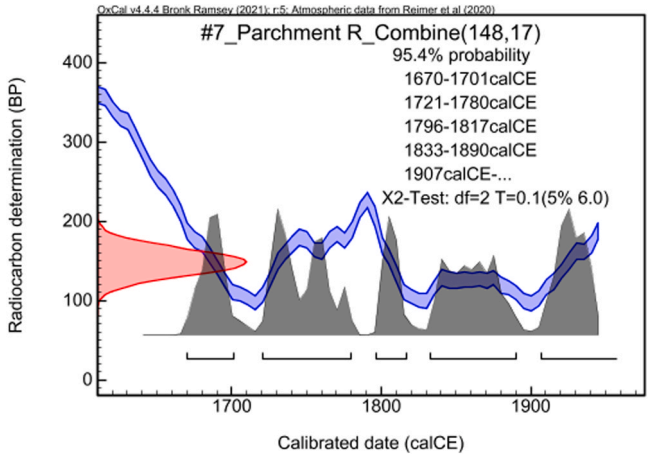

Fig. 5. Calibration of radiocarbon ages. RA ± 2σ (red) of parchment plotted against the IntCal20 calibration curve (blue) and probability density (grey) at a 95.4% confidence level.

Two pages of paper from old books were acquired at an antique shop in Florence. Each page had a year of publication year '1561 CE' (Fig. 1b) and 'Rome 1624 CE' (Fig. 1c). An old postcard (presumably 19th/20th century) of Notre-Dame de Paris cathedral (Fig. 1d) completed the selection of paper samples. In addition, an old textile sample from the cover of a box (age unknown, estimated 19th/20th century) (Fig. 1e) was selected and a piece of parchment, previously analyzed at the ETH laboratory.

Three sub-samples of selected objects were cut, weighed, and photographed before distribution to the labs: ETH-Zurich, ATOMKI-Debrecen, LECCE. The Arches® paper sheet was sampled twice at different locations. On average, each sample contained 20–40 mg of material.

2.2. Samples preparation and AMS analysis

Preparation of samples for ^{14}C AMS analysis requires sample treatment, combustion, and graphitization [5]. Chemical treatment is applied to remove contamination of samples, which introduces exogenous carbon of different ^{14}C content. The standard protocol of the ^{14}C dating applied to organic materials is called acid-base-acid (ABA) treatment. The acid removes carbonates, whereas the base step removes soluble organic contaminants such as humic acids. A bleaching step added to a modified ABA is commonly used to separate cellulose [22]. In addition, solvents can be applied to remove conservation material (varnishes, waxes, oils, consolidants).

Debrecen laboratory used BABAB (base-acid-base-acid-bleach) treatment of paper samples [23]. Textile samples were subject to additional Soxhlet extraction [24] before acid-base- acid (ABA). Parchment sample was only treated with ABA. Before the AMS analysis, the clean sample material was converted to graphite. An equivalent of 1 mg of carbon, i.e., ca. 2–3 mg of material, was vacuum sealed and combusted overnight at 550 ℃ using the sealed-tube method (MnO_2 and silver wire added) [25]. The purified CO_2 was trapped using liquid nitrogen and graphitized by the sealed-tube graphitization method [26]. The resulting graphite was pressed into Aluminium made cathodes, and the $^{14}C/^{12}C$ and $^{13}C/^{12}C$ ratios were measured using the 200 kV EnvironMICADAS [23].

At ETH, laboratory samples of most art objects undergo Soxhlet treatment followed by ABA [24]. Graphitization is completed on ca. 2–3.0 mg of material, wrapped in Al cups for combustion in Elemental Analyzer and a subsequent graphitization in an AGE system [22]. The AMS analysis were completed at the 200 kV MICADAS [27].

Preparation at the Lecce laboratory involved ABA treatment. The purified sample material was then combusted using closed tubes. A mass of ca. 2–3 mg of material was vacuum-sealed in quartz tubes with CuO and silver wool and combusted at 900 °C for 4 hrs. The resulting CO_2 was cryogenically purified and graphitized at 600° with H_2 and iron powder as catalysts [28]. The resulting graphite was pressed into Aluminium made cathodes, and the $^{14}C/^{12}C$ and $^{13}C/^{12}C$ ratios were measured using 3 MV HVEE Tandetron [29,30].

2.3. Conventional Radiocarbon Ages (RA) and calibration

The measured concentration of ^{14}C measured as ^{14}C /^{12}C relative to the standard, normalized and corrected for mass fractionation

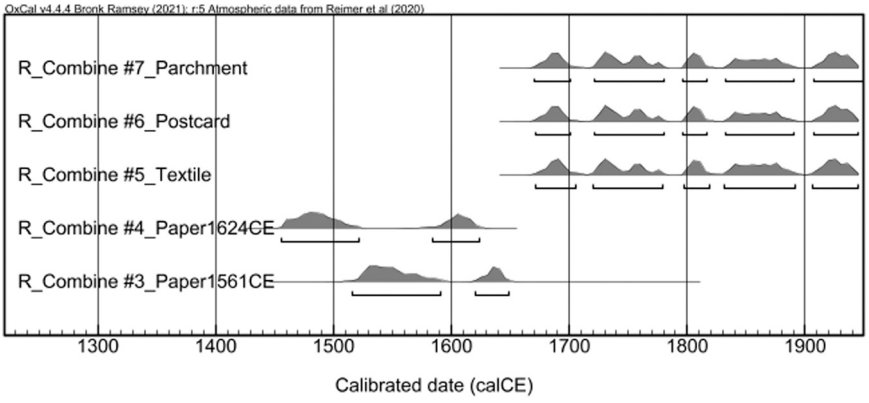

Fig. 4. Calibrated ages are plotted as probability distributions on the calendar time scale.

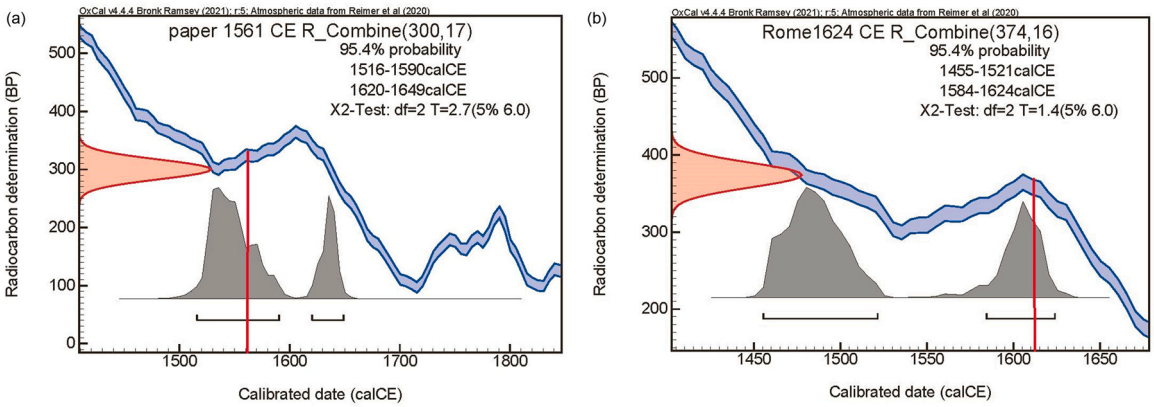

Fig. 6. Calibration of radiocarbon ages. RA ± 2σ (red) plotted against the IntCal20 calibration curve (blue) and probability density (grey) at a 95.4% confidence level. Vertical lines mark the dates written on the sheets; (a) paper with the date 1561 C (b) paper with the date 1624 CE.

($\delta^{13}C$), is expressed as fraction $F^{14}C$ [31]. The $F^{14}C$ value is used to calculate the conventional radiocarbon ages RA as defined by Stuiver and Polach in 1977 [32].

The measured values of $F^{14}C$ provide information about when the sample material was formed; values of $F^{14}C > 1$ indicate the presence of the bomb peak (years 1954–2020).

Radiocarbon ages are calculated using the equation RA= - 8033ln ($F^{14}C$); negative RA correspond to $F^{14}C > 1$.

Calibration using IntCal20 [12] for $F^{14}C < 1$ and Bomb NH1 [10] for $F^{14}C > 1$ was applied using OxCal 4.4 [33,34].

3. Results and discussion

Results of radiocarbon analysis: $F^{14}C$ and RA values (± 1σ) are summarized in Table 1. The 1σ reported include counting statistics and corrections applied by individual laboratories [5]. The $F^{14}C$ measured by all the three laboratories for all the samples are in excellent agreement, as shown by the χ^2 Test. Fig. 2 shows all the results plotted as $F^{14}C$ with 1σ.

The weighted mean was calculated for each sample (Table 1). With the help of OxCal 4.4, these values were used for calibration to calendar ages. Despite the slight scatter of the $F^{14}C$, all the results for two samples of Arches® paper (sample #1 and #2) were combined and calibrated using the bomb ^{14}C curve [10] because the measured $F^{14}C > 1$ (Fig. 3a). The scatter might be due to differences in treatment between laboratories, indicating a need to apply modified ABA to remove contaminants (old carbon). The bomb ^{14}C curve calibration always delivers multiple intervals corresponding to measured $F^{14}C$. The year 1955 can be excluded here, and the second interval, 2016–2018, agrees with the purchase date. The potential two-year production and distribution of paper (from harvest to the store) seems feasible.

The samples of paper, parchment, and textile show $F^{14}C < 1$ and positive RA and can be calibrated using the IntCal20 calibration curve [12] (Fig. 4).

The ages of textile (#5), postcard (#6), and parchment (#7) appear to cluster around the end of the 17th to the mid-20th centuries. Such a wide range of calibrated ages, the so-called 'Stradivarius plateau' [5], is due to the changes in the atmospheric ^{14}C, partly due to natural variability and combustion of fossil carbon. In effect, all three samples appear simultaneous (Fig. 4), but that does not have to be the case. However, the multiple intervals must be considered (Fig. 5) as long as additional information about the object is not provided. For example, in the case of art objects, the date of the artist's death or the year of purchase. Such information can be included in calibration as a prior and help narrow the calendar ages ranges [5].

RA measured for antique paper samples #3 (1521 CE) and #4 (Rome1624 CE) are older and do not fall into the period of variable ^{14}C in the 17th-20th century. However, the calibration curve is complicated and forms another age plateau coinciding with Leonardo da Vinci time, called 'Vinci-plateau' [5](Fig. 6a-b). Nevertheless, both pages of antique paper are accurately dated to the declared ages, i.e., 1521 CE for sample #3 (Fig. 6a) and 1624 CE for sample #4 (Fig. 6b). Considering that the pre-industrial paper was produced from rugs, i.e., short-lived plant fiber [20], radiocarbon dating of pre-industrial paper has the potential for accurate dating as opposed to industrial, produced from wood. The wood pulp used for paper production can have a composite radiocarbon signal dependent on the age of the wood used [20].

4. Conclusions

The results of an inter-comparison performed by three laboratories on paper, textile, and parchment, i.e., the material often used as support of art, are in excellent agreement. Samples of cotton paper showed the bomb peak ^{14}C, which dates the cotton harvest to 2–3 years before purchasing the paper sheets. Calibrated radiocarbon ages of the antique sheets of the paper agree with the expected ages. Based on these results, we demonstrate that independent of the laboratory, AMS ^{14}C dating provides conclusive results such as proof of ages obtained for two antique sheets or the production of Arches® paper.

Contribution

All authors contributed equally to this study.

Declarations of interest

None.

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