

Dating the Shroud using Wide Angle X-Ray Scattering

Interview with Liberato de Caro

Earlier this year, we witnessed an extraordinary amount of news media coverage of some recent research carried out by a team of scientists from the Institute of Crystallography in Bari led by Liberato de Caro. This team developed an innovative new method of dating linen fabric using Wide Angle X-Ray Scattering (WAXS) and in 2022 had used this technique to determine the age of fragments of material from the Shroud. This test concluded that the fabric of the Shroud dated from the first century, a result which supports claims that it is the same cloth that was used to cover the body of Jesus Christ in the tomb.



Liberato de Caro

Could you tell us a little about the type of work typically performed by you and your team at the Institute of Crystallography in Bari?

Personally, I have been working for more than 30 years mainly on techniques for investigating matter and materials on the atomic scale with X-rays and electron microscopy, but I have also been involved in many other research topics. The Institute of the National Research Council, where I work, is the Institute of Crystallography. Crystallography is a science that uses the order of crystals to determine the structure of even the most complex molecules. For many years, I also worked actively in the field of Crystallography. In particular, in Bari, Italy, the Institute of Crystallography has a laboratory called XMI-LAB which is at the forefront internationally for the study of materials using various techniques including Wide Angle X-ray Scattering (WAXS), one of the most widely used atomic scale investigation techniques in the world [1]. Given the presence of this internationally leading-edge instrumentation, the other colleagues, co-authors of the X-ray dating of the Shroud of Turin specimen, among other skills, are highly experienced in the WAXS technique.

What gave you the idea that it may be possible to calculate the age of linen material from the spectra produced by WAXS?

The WAXS technique is useful for determining the structure and order level of materials on the atomic scale. The cellulose that makes up flax fibres consists of chains in which the same molecule (polymer) is repeated. With the passage of centuries, the average length of these polymer chains decreases. X-rays, penetrating into the matter, can give us information about the average length of the polymer chains of the analysed sample. By comparing the length of the polymer chains of cellulose from contemporary linen samples, it is possible to determine how old the sample under examination is.

What tests did you perform to confirm that this method of dating linen was capable of giving accurate measurements of the true age?

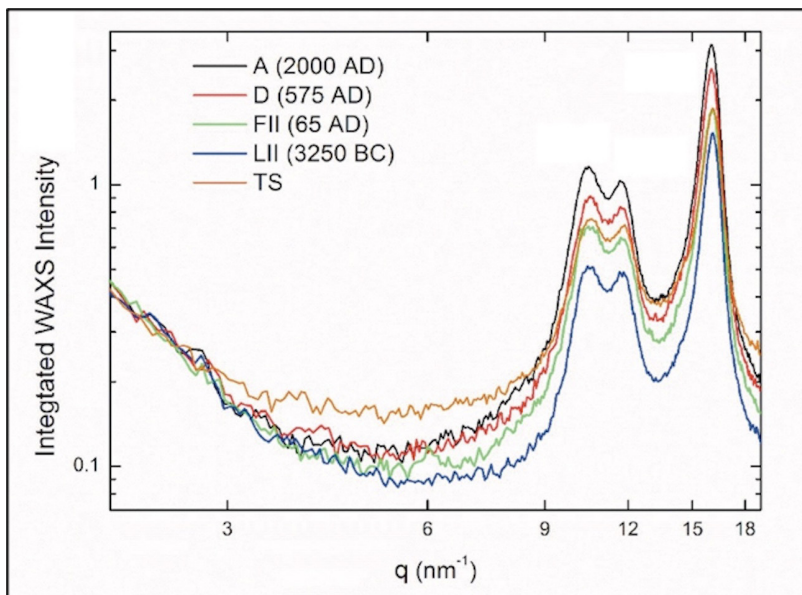
A series of ancient linen samples from different periods, already dated with carbon 14, were dated with the WAXS technique, which enabled the production of a calibration curve for the new dating technique. The comparison of the dates obtained by WAXS and carbon 14 allowed us to verify that the new dating technique provided the true age of the archaeological artefact analysed [2].

The Shroud was scorched and nearly destroyed in the 1532 fire in Chambéry and some people have questioned whether the temperature and humidity conditions produced by that fire would have affected the WAXS measurements for the Shroud, causing it to give a result which is much older than its true age. How did you determine what effect, if any, these conditions would have had on the Shroud's age measurement?

The Shroud suffered damage from this fire in some localised regions but most of the fabric wasn't badly damaged, otherwise the body image of the Man it wrapped would no longer be visible. In order for exposure to high temperatures to increase the natural ageing of linen, it is also necessary to maintain a constant relative humidity, since laboratory tests show that if the relative humidity drops to zero, the reaction speed of cellulose depolymerisation decreases to zero. This effectively blocks any increase in ageing of the cellulose.

During a fire, the relative humidity of the environment in which the fire occurs drops to zero. So exposure for a time to a temperature which is higher than the ambient temperature but also at zero relative humidity does not cause any increase in the natural ageing of the flax cellulose. We verified this in the laboratory by subjecting a flax sample to a temperature of 200 °C for half-an-hour in an oven. As the relative humidity went to zero, no change was seen in the WAXS measurements before and after subjecting the sample to this high temperature, confirming that there would have been no increase in the natural ageing of the Shroud linen due to the Chambéry fire, with the exception of those parts of the fabric that were damaged. In fact, a large part of the sheet would have been subjected to a temperature of about 200°C during the fire, since at 310°C linen burns. If the whole sheet had reached a temperature of 310°C then the Shroud of Turin would no longer exist, it would have been totally burnt.

These laboratory tests confirmed that the fire did not contribute to the natural ageing of the linen of the Shroud. Moreover, the most significant data obtained was that the WAXS measurements on the Turin Shroud sample were fully compatible with those of a sample taken from the Masada Fortress in Israel, which we know from Roman historical accounts to be two thousand years old.



WAXS profiles produced by the Turin Shroud (TS) and four test samples. The intensities of the signal peaks to the right of the profile produced by the Shroud sample are almost identical to those produced by sample FII, a first century linen fabric from an archaeological site in Masada. This shows that the two samples are of a similar age. From X-ray Dating of a Turin Shroud's Linen Sample by Liberato De Caro et al., Heritage 2022, 5, 860–870.

This direct comparison of the WAXS measurements obtained from the Shroud sample and that of the Masada sample allows us to conclude with certainty that the Shroud of Turin is two thousand years old, because this coincidence in the age of the two archaeological finds, both dated two thousand years old, would be truly strange if one of the two had suffered an acceleration in the natural ageing of the linen due to the fire. This would mean that we would have to conclude that a linen sheet produced during medieval times had undergone accelerated ageing of the cellulose due to the Chambery fire, and that by sheer chance this increase gave a result which was precisely two thousand years old. This is the equivalent of having a partly-filled, non-transparent bottle and somehow managing to blindly guess exactly how much more liquid to add to the bottle to fill it exactly to the brim. Such a remote possibility can certainly be dismissed given its improbability of coming true. So the WAXS measurements allow us to conclude that the Shroud is with certainty two thousand years old.

There is evidence that the radiocarbon dating result was significantly affected by some form of contamination. Is it possible that the Shroud material that you tested could have been contaminated and if so, would this have caused an incorrect result?

Following the legal action of Tristan Casabianca, the raw data of the measurements carried out by the three laboratories that dated the Shroud with the radiocarbon method were finally released in 2019, thirty years after the publication of the Shroud's radiocarbon dating in 1989. This raw data, published for the first time in 2019 in the paper of Casabianca and co-workers shows that the C-14 measurements have undergone a "statistical make-up" to reduce the differences between the dating results of the three laboratories [3]. This result, in turn, indicates a bias introduced in the radiocarbon dating to mask the effects of contamination, which is evident because the age of the Shroud decreases moving from the outermost sample with respect to the cloth's border to the innermost one. Conversely, X-ray dating can be reliably carried out on a sample even if it has been contaminated by C-14 from more recent times, because X-ray dating depends on a completely different property of the material, the structural order of cellulose.

This technique is very new. Are there any developments and further research that you would like to see in order to improve the precision and reliability of the WAXS dating method?

It would be possible to obtain a more accurate calibration curve by using the WAXS technique to date more samples taken from linen fabrics of known age from different eras than were available for our research in 2019. This applies also to radiocarbon dating, where the calibration curve is periodically recalculated and improved based on new data. As further research, it would be possible to use the WAXS technique to date other relics that tradition associates with Jesus, such as the Sudarium of Oviedo.

Do you believe that other laboratories will eventually choose to use this innovative dating technique to measure the age of other ancient fabrics, rather than using radiocarbon dating method? What advantages does it have over radiocarbon dating?

The WAXS technique is already used by many laboratories around the world to investigate the order of matter and materials on the atomic scale. We have been working with X-rays for more than 30 years. The novelty of our research is that we have applied this well-known investigation technique to the measurement of the degradation of the cellulose of linen fibres, which occurs with the passage of centuries, resulting in a new dating technique. It can be considered as a complementary technique to radiocarbon dating which can be used when there are doubts about the results obtained from C-14 measurements. It is also a valid alternative when it is suspected that a sample may be highly contaminated with carbon from later periods, or when the standard procedure of cleaning the sample prior to radiocarbon dating may not have been effective.

In fact, as already clarified, X-ray dating is unaffected by carbon-14 contamination and unlike the radiocarbon method where the sample used for dating is destroyed, it is not destructive. Therefore, X-ray dating can be repeated several times, even by different laboratories, using the same sample. This is a great advantage over radiocarbon dating.

Another major advantage of X-ray dating applies to the dating of ancient textiles. These are approximately 50 per cent empty space which can become filled with air and dirt, making them difficult to clean and so are the least suitable materials for radiocarbon dating. It is therefore possible that this new WAXS-based dating technique could in future become a viable alternative to the radiocarbon method for the dating of ancient textiles.

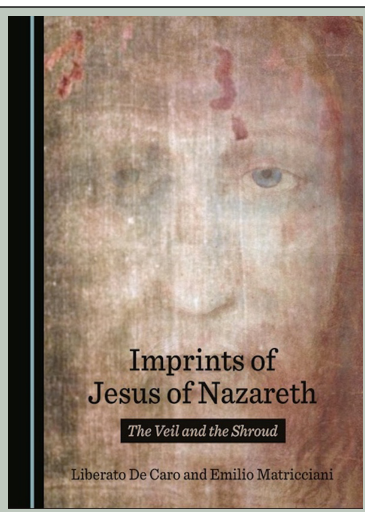
Finally, how surprised were you by the widespread news media coverage given to your 2022 WAXS dating of the Shroud in recent weeks?

There is a perfectly apt adjective which is often used to describe what sometimes happens in the media: 'viral'. Just as a virus can lie dormant for a while and then explode into an exponential growth of contagion, so some media reports can suddenly have an exponential growth in media interest. In fact, just as a virus, in order to spread rapidly among the world's population, must break out of its isolation and enter a global circuit in which many people of different nationalities interact with each other, so a piece of news must reach a few particular websites, seen by many users all over the world, such as those in English, in order to then be able to spread rapidly throughout the world.

References

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Liberato de Caro's book
*Imprints of Jesus of Nazareth:
 The Veil and the Shroud*
 covers the latest studies of
 the Shroud and
 the Veil of Manoppello.
 It is available from Cambridge
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