

**THE IMPORTANCE OF USING MULTIPLE ANALYSES TECHNIQUES
TO DETERMINE THE PHYSICAL CONDITION OF THE WATERLOGGED WOOD
NEAR THE CORRODED PARTS**

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(RECEIVED APRIL 2021)

ABSTRACT

In this study, three samples taken from the planks of the Yenikapı 29 shipwreck were analysed. Firstly, the maximum water content (MWC) and basic density values of the samples were calculated. MWC of the IK13-1 was 164% and the MWC of the SK6-1 was 87%. Because of these low MWC values, samples could be classified as non-degraded. When the SEM images of these two samples were examined, it was understood that the IK13-1 sample was non-degraded but the SK6-1 sample was penetrated with corrosion product and could not be classified as non-degraded. With these analyses, the XRF method was used to measure the iron amounts of the samples. The iron amount of the SK6-1 was 32.3% and the corrosion accumulation in this sample was also proved with XRF. In order to avoid incorrect results, multiple analysis techniques should be used for determining the physical condition of the waterlogged wood near the corroded parts.

KEYWORDS: Basic density, maximum water content, SEM, waterlogged wood, Yenikapı shipwrecks.

INTRODUCTION

During burial under water and/or wet land for many years, the wood transforms into a waterlogged form. Due to this transformation, the physical and chemical properties of the wood change. At first sight, the excavated waterlogged woods seem to be in a good condition because the pores of the wood are saturated with water. This sight can commonly be misleading and the wood in waterlogged form might as well be degraded. The age of the wood, species of the wood, intended purpose of the wood, the ambient conditions under which it is buried and the time it spends under these conditions affect the degree of the degradation of the waterlogged wood. In addition, the chemical and biological degradations of the wood cause the changes in the physical condition of the wood. Therefore, the determination of physical and chemical condition of the waterlogged wood is crucial for

understanding the degradation process of the waterlogged wood. In addition, the waterlogged wood is classified according to its degradation degree. A classification system, which relates maximum water content (MWC), is commonly used (Broda et al. 2015a,b, Koman and Feher 2015, Christensen 1970, De Jong 1977, Fix 2015, Grattan 1987, Hamilton 1996, Hedges 1990, Florian 1990, Kaye and Cole-Hamilton 1998, McConnachie et al. 2008, Dollarhide 2019). In addition, several techniques are used in order to determine the physical condition of the waterlogged wood. For instance, physical resistance monitoring techniques such as pin test, Pilodyn, Sibbert drill, and electrical resistance monitoring, wood-water amount related analyses such as MWC, basic density (BD), loss of wood substance, and visual analyses techniques such as optical microscope and electron microscope are used in order to determine the physical condition of the waterlogged wood (Grattan 2000, Ogilvie 2000, Rodgers 2004, Jensen and Gregory 2006, Gregory et al. 2007, Capretti et al. 2008, Heritage 2010, Macchioni et al. 2012, Pizzo et al. 2010, Babinski et al. 2014, Oron et al. 2016, Kılıç and Kılıç 2018, Romagnoli et al. 2018, Han et al. 2020). Furthermore, a combination of these techniques can be applied.

37 shipwrecks, which are located in Istanbul, are considered as the world's largest medieval shipwreck collection. The shipwrecks were excavated by Directorate of Istanbul Archaeological Museums and conservation studies of the 31 of these shipwrecks have been carried out by the Istanbul University Yenikapı Shipwrecks Project (Kocabaş 2015). The members of the project undertook the conservation process of the shipwrecks. One of the important steps of these conservation processes was the determination of the physical condition of the wooden parts of the shipwrecks. The MWC and the BD values of the waterlogged woods were calculated in order to understand the physical condition of the waterlogged woods. The studies, which were conducted on Yenikapı shipwrecks, showed that there was an inverse proportion between MWC and BD values of the waterlogged wood and MWC values of the highly degraded woods were higher (Kılıç and Kılıç 2019). On the other hand, inorganic residues, which penetrated into the structure of the woods, could cause failure in these values (Kılıç 2017). For instance, corrosion products such as corroded nails could penetrate into woods. The basic principle of the MWC analysis is calculating the amount of the water inside the woods. Highly degraded woods have more gaps inside the wood. Normally, these gaps of the waterlogged wood are filled with water and the amount of the water inside the wood indicate the physical degradation level of the waterlogged wood. The water can be removed easily with heat and the MWC value can be calculated using differences between the weights of the wood. On the other hand, iron corrosion products are stable and cannot be removed with heat. Therefore, the weight of the wood, before and after having been put in an oven, was similar. Corrosion products which have penetrated the gaps in the woods cause failures in the MWC and the BD calculations. In general, the wooden parts which were penetrated with corrosion products can be identified easily by naked eye observation. Therefore, the results could be ignored easily (Kılıç and Kılıç 2019). On the other hand, corrosion product can only penetrate the core of the wood. In this situation the parts of the woods contaminated with iron corrosion products cannot be identified easily. In order to determine iron corroded areas, SEM can be used as a monitoring technique. SEM is a useful tool for the investigation of the surface appearance of waterlogged wood (Charola and Koestler 2006). SEM images with high resolution can provide a determination

of the accumulation of the iron corrosion products inside the gaps of the wood. In addition, XRF analyses were done in order to determine inorganic compounds in the woods. The Yenikapı Shipwrecks Project handled hundreds of waterlogged wood pieces from dozens of shipwrecks, so the simplicity and the cost of the analysis techniques are very fundamental. On the other hand, there are important points to be considered in order to make these simple calculations correctly. Samples taken near the corroded parts of the woods have low MWC values due to the iron compounds in the samples. Therefore, the aim of this study is the determination of deviations in the samples which have low MWC values via SEM images.

MATERIAL AND METHODS

The study was done on the samples of YK (Yenikapı) 29 which is one of the 37 Yenikapı shipwrecks. YK 29 is a merchantman, and it is dated to the 8–9th century based on stratigraphy (Fig. 1). The shipwreck's extant length is 7.90 m and width 4.5 m. The planks of the ship were nailed to the frames with iron nails (Kocabaş 2015).

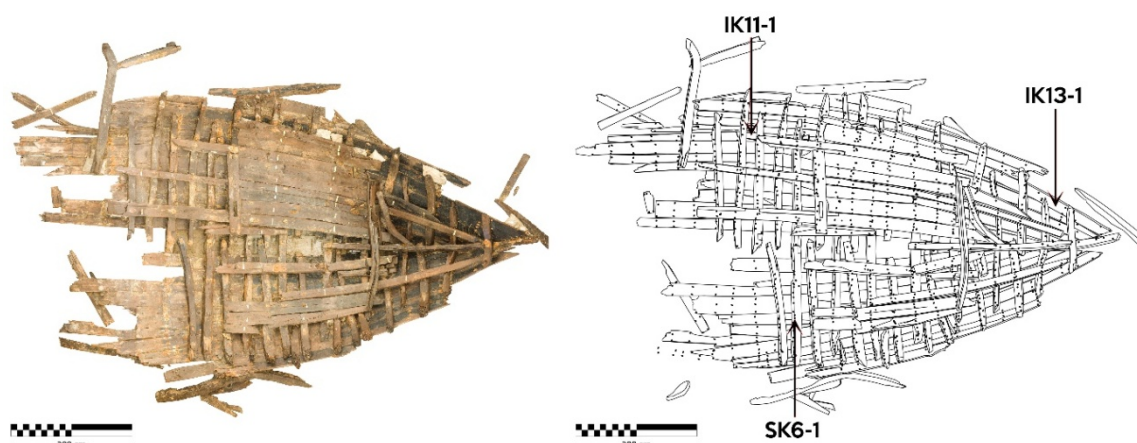


Fig. 1: Photo-mosaic image of YK 29 shipwreck (left), representation of the sample taken points on the drawing of YK 29 shipwreck (right) (IU Yenikapı shipwrecks project archive).

Samples were taken from 3 different planks (IK11-1, IK13-1, and SK6-1) which were in the desalination tanks at the IU Yenikapı Shipwrecks Research Laboratory. In order to calculate MWC and BD values, small pieces, not less than 1 g, were taken (Fig. 1). Before the measurement, the dirt on the surface of the samples was cleaned. These waterlogged samples were weighed in air and in water. Then, samples were oven-dried to a constant weight and finally weighed again. An analytical balance with an accuracy up to 0.0001 g was used for weighing (Kılıç and Kılıç 2019). The following equations were used in order to calculate MWC and BD values (Babinski et al. 2014).

$$\text{MWC} = 100 \times (m_w - m_d) / m_d \quad (1)$$

$$\text{BD} = m_d / V_w \quad (2)$$

where: MWC - maximum water content (%), BD - basic density ($\text{g}\cdot\text{cm}^{-3}$), m_w - mass of waterlogged sample (g), m_d - mass of oven-dry sample (g), and V_w - volume of waterlogged sample (cm^3).

In order to examine the physical condition of the samples in detail, SEM (Scanning Electron Microscope) images of the samples were taken with a FEI Quanta 450. Samples were prepared in transverse section direction (approximately 0.5 - 1 cm³) with razor blade for SEM. After these analyses, XRF analyses were done in order to determine iron amounts of the samples. XRF analyses were carried out on a Spectro xSort handheld XRF Analyzer.

RESULTS AND DISCUSSION

The MWC and the BD values of the samples are presented in Tab. 1 (Akgün 2020). The samples, which were taken from IK13-1 and SK6-1, had low MWC values and high BD values. In addition, the MWC value of the sample, which was taken from IK11-1, was higher and the BD value of the sample was lower. The wood species of the samples is cypress (*Cupressus L.*) (Akkemik 2015).

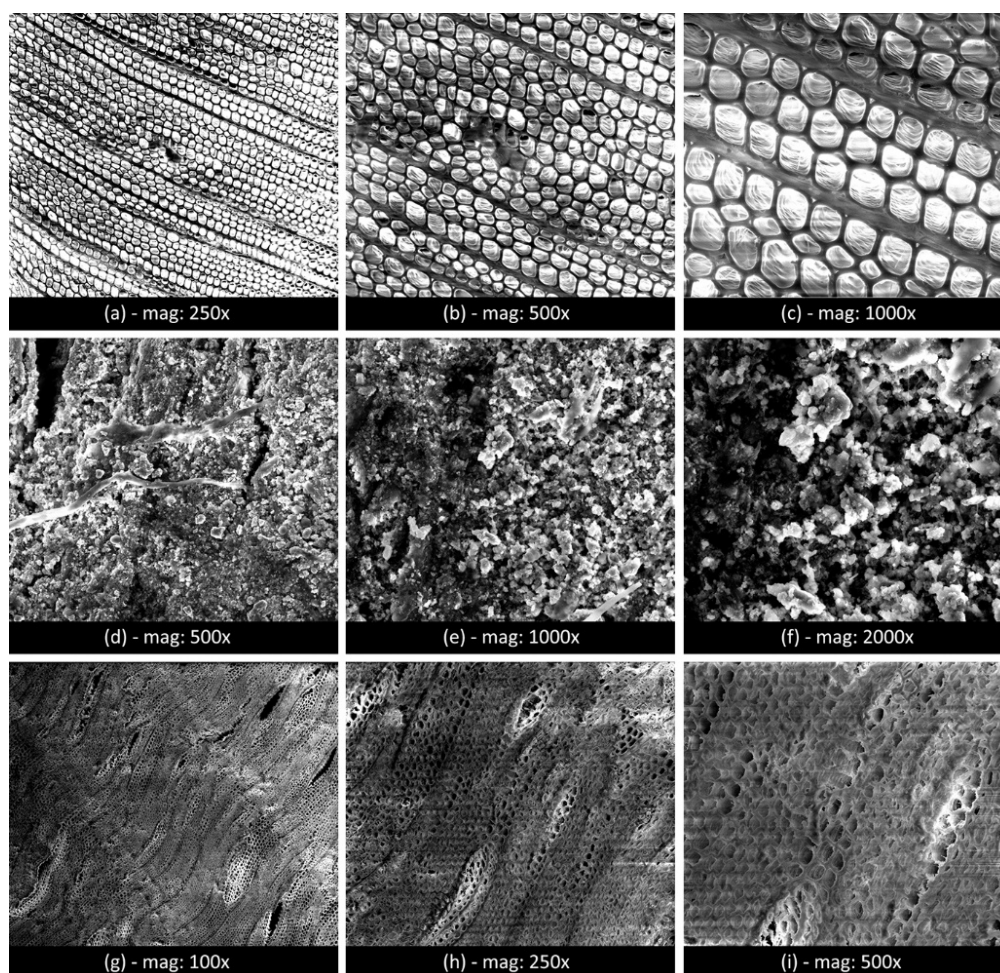


Fig. 2: SEM images of the samples. a-c: cross-section of the sample taken from IK13-1, d-f: cross-section of the sample taken from SK6-1, cross-section of the sample taken from IK13-1, g-i: cross-section of the sample taken from IK11-1.

According to MWC and BD values of the samples, the sample taken from IK13-1 should be recognized as highly preserved due to low MWC value. The sample taken from IK11-1 should be recognized as highly degraded due to high MWC value. The MWC value of

the sample taken from SK6-1, was remarkably low for a waterlogged wood sample and the BD value was higher than the density of fresh cypress wood (the density of the fresh cypress wood is $0,45 \text{ g cm}^{-3}$) (Akgün 2020). Due to the suspicion caused by this value, it was decided to perform SEM. In order to determine the physical conditions of the samples, SEM images were taken.

When the SEM images of the sample taken from IK13-1 were examined, it was observed that the wood was highly preserved, and tracheid, ray, and tracheid tangential wall of the wood were almost intact. The SEM images of the sample showed that there was almost no degradation on the structure of wood cells (Figs. 2a-c). When SEM images of the sample taken from SK6-1 were examined, it was observed that SK6-1 was covered with iron corrosion products so the woody tissue could not be observed (Figs. 2d-f). When SEM images of the sample taken from IK11-1 were examined, the degradation of the wood cells could be observed. Especially, collapse of the wood cells could be detected (Figs. 2g-i).

After these SEM observations, XRF analyses were done in order to measure the iron amounts of the samples (Tab. 1).

Tab. 1: MWC values of the samples.

Sample	MWC (%)	BD (g cm^{-3})	Iron amounts of the samples (%)
IK11-1	441	0.20	3.7
IK13-1	164	0.45	0.4
SK6-1	87	0.71	32.3

IK and SK: Plank.

Moreover, an additional sample was taken from a part which was distant from the corroded area of the SK6-1 wood and SEM images of the sample were taken in order to have an idea about the physical condition of the SK6-1 wood (Fig. 3).

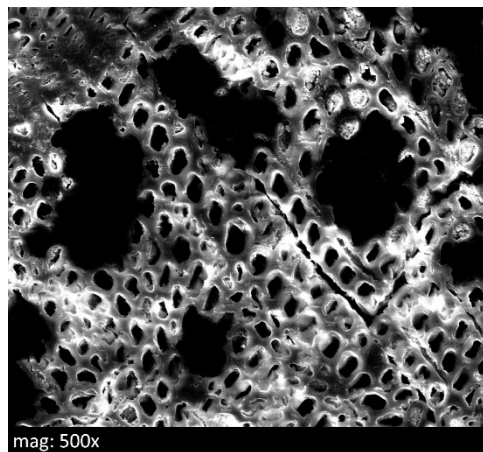


Fig. 3: SEM images of an additional sample taken from SK6-1.

When the SEM images were examined, the distortion occurring in the wood structure was observed. These images gave an idea about what might happen if the wood was not filled with corrosion products. For this reason, it is necessary to examine the woods with very low MWC values and very high BD values via microscopic methods especially via SEM.

CONCLUSIONS

The Yenikapı shipwrecks project handled hundreds of waterlogged wood pieces from dozens of shipwrecks, so the simplicity and the cost of the analysis techniques used for determination of the physical condition of the woods are very fundamental. With the help of determining the physical condition of the woods, conservation procedure of the wood can be maintained successfully. MWC and BD values can be calculated easily and economically but any contamination can cause incorrect results. In order to avoid any erroneous sampling due to the excessive number of the wooden parts, the size of the wooden parts of the shipwrecks, the sampling conditions (samples can be taken when the samples were in the desalination tank), and the sampling should be inspected well. Nevertheless, should be specifically examined with further analysis techniques such as SEM and XRF especially the wooden parts of the shipwrecks which were near the corroded iron nails. Iron corrosion products are stable against heat. In addition, the weight of the wood was similar before and after heating. Therefore, corrosion products which have penetrated the gaps in the woods cause failures in the MWC and the BD calculations. This study presented that usage of multiple analysis techniques is very beneficial for determining the physical condition of the waterlogged wood near the corroded parts.

ACKNOWLEDGEMENTS

We would like to thank Prof. Dr. Ufuk Kocabaş, Istanbul Archaeology Museums, and Yenikapı Shipwrecks Project Team for their help and support. The IU Yenikapı Shipwrecks Project has been realized with the financial support of İstanbul University Scientific Research Projects Unit (Project nos: 13986, 2294, 3907, 7381, 12765, SDK-2016-3777, SDK-2016-3776, SYL-2019-34322).

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