NEW METHOD OF FIXING SNOW SAMPLES IN THE FIELD FOR X-RAY CT IMAGEING USING 1-BROMODODECANE

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ABSTRACT: Snow is characterised by a three-dimensional network structure comprising a multitude of ice particles, exhibiting a diverse range of shapes and intricate interconnections. This three-dimensional network structure is a principal factor influencing the transport mechanisms of heat and water vapour, as well as the mechanical characteristics of snow. It is therefore essential to gain an understanding of the three-dimensional structure of snow in order to comprehend the physical properties of snow. X-ray computed tomography (X-ray CT) is an invaluable technique for examining the microstructure of snow. However, when investigating natural snowpacks, it is essential to safeguard the snow samples from metamorphosis and microstructure disruption during their transfer from the field to the laboratory. In order to achieve this, a number of methods have been proposed for the purpose of filling the voids of the snow sample with a liquid. The most commonly used method is the utilisation of diethyl phthalate as the filling liquid. In this case, the ice particles are sublimated in a high vacuum after the sample has been fixed with diethyl phthalate. The remaining negative structures (replicas) must then be imaged when imaging with X-ray CT.

We propose a novel method utilising 1-bromododecane ($C_{12}H_{25}Br$) as the filling liquid. $C_{12}H_{25}Br$ can be stored in a conventional freezer, given that its melting point is -9.5 ℃. Furthermore, the distinct X-ray absorption difference between $C_{12}H_{25}Br$ and ice allows for the differentiation of ice particles and $C_{12}H_{25}Br$ in X-ray CT images. Consequently, direct measurement of ice particles is feasible without the necessity of replication, in contrast to the approach employed with diethyl phthalate. To assess the efficacy of the proposed method, fresh snow fixed with $C_{12}H_{25}Br$ was imaged after 50 days of storage in a cold room at -15 °C. The morphology of the snow crystals was found to be well preserved. Consequently, our proposed method is highly effective in transporting samples from their point of origin in the field to a laboratory setting for extended periods without compromising their integrity or causing deterioration. It is anticipated that this approach will facilitate accelerated investigations into the microstructure of natural snow.

KEYWORDS: snow sample fixing, X-CT imaging, snow microstructure

1. INTRODUCTION

Snow is a porous material comprising a network of ice particles held together by sintering and a continuous interstitial space. The microstructure of snow thus determines many of its properties, including mechanical strength, thermal conductivity, surface chemistry and albedo. For numerical simulation of these properties, it is essential to know the microstructural properties of snow at the sub-millimetre level. However, the resolutions of visual observations in the field are insufficient.

The utilisation of X-ray computed tomography (X-ray CT) has been put forth as a methodology for quantifying the microstructure of snow (Brzoska et al., 1999; Coleou et al, 2001; Flin et al., 2003; Schneebeli, 2004; Schneebeli and Sokratov, 2004), A number of studies have employed this approach (e.g., Kaempfer and Schneebeli, 2007; Löwe et al., 2011; Pinzer et al., 2012; Calonne et al., 2012). Nevertheless, the assessment of snow microstructure by X-ray CT has been confined to samples procured in the vicinity of the laboratory where X-ray CT is employed. This is due to the fact that prolonged storage or transportation of snow samples to the laboratory may result in alterations to their microstructure, potentially due to metamorphism and/or the inherent fragility of the snow. Consequently, samples that must be transported from remote locations, such as avalanche release zones, Greenland, or Antarctica, or that

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cannot be analysed promptly, must be fixed in the field to prevent metamorphism and destruction.

A number of techniques for fixing and preserving snow samples for X-ray CT have been proposed (Flin et al., 2003; Heggli et al., 2009). This study presents a novel method for fixing and preserving snow samples using 1-bromododecane.

2. CHARACTERISTICS OF 1-BROMODODECANE

The transportation and long-term storage of snow and ice samples for magnetic resonance imaging (MRI) have already been established. In particular, this method entails the freezing and fixing of snow samples in the field, subsequent to the injection of n-Dodecane ($C_{12}H_{26}$) with a melting point of ‑9.7 °C (Ozeki et al., 2003). As ice particles cannot be directly imaged by MRI (Yamaguchi et al., 2023), the actual imaging was conducted by imaging $C_{12}H_{26}$-filled voids and further inverting the brightness and darkness of the image to obtain a three-dimensional network structure of the snow sample (Ozeki et al., 2003).

Nevertheless, when snow samples containing $C_{12}H_{26}$ are imaged using X-ray CT, the contrast difference between $C_{12}H_{26}$ and ice particles in the resulting image is minimal due to the lack of significant difference in X-ray absorption, which hinders the ability to differentiate between ice and voids filled by $C_{12}H_{26}$. Accordingly, in the present study, 1-bromododecane ($C_{12}H_{25}Br$) was employed as the filling liquid, given that it contains Br with relatively high X-ray absorption.

Table 1 Physcail Properties of $C_{12}H_{26}$ and $C_{12}H_{25}Br$

Physical Properties	$C_{12}H_{26}$	$C_{12}H_{25}Br$
Density (kg m^{-3})	752	1038
Melting point (°C)	–9.7	-9.5
Surface tension (N m^{-1})	2.0×10^{-2}	3.1×10^{-2}
Viscosity (Pa s)	1.6×10^{-3}	3.8×10^{-3}

Table 1 presents a comparative analysis of the physical properties of $C_{12}H_{26}$ and $C_{12}H_{25}Br$. The data in Table 1 indicates that $C_{12}H_{26}$ and $C_{12}H_{25}Br$ exhibit slight differences in their physical properties, yet exhibit an almost identical melting point. This suggests that $C_{12}H_{25}Br$ could be injected into samples and frozen in the field in a manner analogous to $C_{12}H_{26}$, thereby facilitating the long-term transportation and storage of the samples.

3. METHOD

3.1 *Method for fixing samle in the field*

The procedure for fixing the snow sample in the field is as follows:

1) The filling liquid ($C_{12}H_{25}Br$) should be cooled to a temperature as close as possible to its melting point, for example, in a portable freezer.

2) The snow sample, excised from the snow pit, is placed in a covered storage container that is of a sufficient size to accommodate the X-ray CT sample.

3) The $C_{12}H_{25}Br$ solution should then be slowly injected along the inner wall of the storage container using a glass rod or syringe. In the event that the temperature of the snow sample is below the melting point of $C_{12}H_{25}Br$, it is necessary to adjust the temperature to a level above the melting point before proceeding to step 3.

4) Once the voids have been filled sufficiently with $C_{12}H_{25}Br$, the storage container should be sealed with a lid.

5) The freezer should be set to a temperature below the melting point of $C_{12}H_{25}Br$, and the storage container with the snow sample should be placed inside to allow the $C_{12}H_{25}Br$ to freeze completely.

3.2 *Imagine procedure*

The X-ray CT system employed was μCT 35 (SCANCO Medical, Switzerland), with the sample size dependent on the resolution achievable. The maximum scan size encompassed a height of 120 mm and a diameter of 37.9 mm. The resolution exhibited a range of 3.5 to 72 μm.

In the event that imaging is conducted while $C_{12}H_{25}Br$ is in a frozen state, the formation of fine lines on the $C_{12}H_{25}Br$ image may occur as a consequence of the volume change of $C_{12}H_{25}Br$ in the frozen state. To circumvent this phenomenon, it is recommended that the snow sample be maintained at a temperature slightly above the melting point of $C_{12}H_{25}Br$ throughout the imaging process, ensuring that $C_{12}H_{25}Br$ remains in a liquid state. It is necessary to remove any excess $C_{12}H_{25}Br$ from the snow samples prior to imaging, as they will otherwise float in the liquid. In the event that bubbles remain in the snow sample, measures such as the addition of $C_{12}H_{25}Br$ should be implemented.

Due to the high X-ray absorption of $C_{12}H_{25}Br$, the maximum value that can be set in uCT is used. Furthermore, a greater number of images than usual were averaged (Table 2).

This study employed the Otsu binarization method (Otsu, 1979), a standard image analysis technique, to binarize the averaged X-ray CT images (representing the ice particles and $C_{12}H_{25}Br$).

Table 2: Details of the X-ray CT settings

Energy/Intensity	70kVp, 114µA, 8W
Integration Time	800 msec
Average	10

4. RESULTS AND DISCUSSION

The cryospheric environment simulator (CES) at the National Research Institute for Earth Science and Disaster Resilience is capable of generating artificial snow that closely resembles natural snow (Fig. 1a) (Abe and Kosugi, 2019). The efficacy of the novel methodology was evaluated using the aforementioned artificial snow.

Initially, X-ray CT images were obtained of the artificial snow immediately following its creation (Fig. 1b). One sample was maintained in its original state, while the other was treated with $C_{12}H_{25}Br$. Both samples were subsequently stored in a refrigerated environment at a temperature of -15°C for a period of 50 days. Subsequently, X-ray CT images were obtained for both samples.

Figure 1c illustrates the outcomes of the artificial snow sample stored in a refrigerated environment at -15°C for a period of 50 days. The snow particles undergo a process of metamorphism, resulting in a rounded shape. As a consequence, the microstructure of the snow has undergone a complete transformation, losing all resemblance to the original artificial snow (Fig. 1b). In contrast, the X-ray CT image of the sample fixed with $C_{12}H_{25}Br$ (Fig. 1d) demonstrates that the snow particles maintain their original shape after 50 days. These findings suggest that the $C_{12}H_{25}Br$ fixation method is an effective approach for long-term preservation of X-ray CT specimens without metamorphism.

5. CONCLUSIONS

This study presents a novel approach utilising $C_{12}H_{25}Br$ as a filler to stabilise samples for X-ray

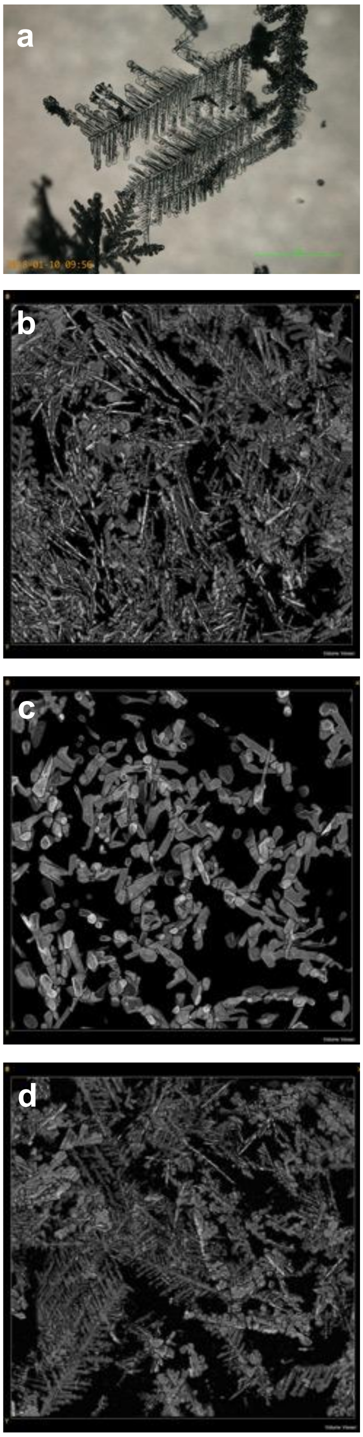

Figure 1: Panel (a) depicts a micrograph of the artificial snow immediately following its manufacture by CES. Panel (b) is an X-ray CT image of the same sample taken immediately after manufacture. Panel (c) is an X-ray image of the same sample after 50 days without $C_{12}H_{25}Br$ added. Panel (d) illustrates the same sample after 50 days with $C_{12}H_{25}Br$ fixed.

CT imaging. The method was applied to snow samples, and it was confirmed that the shape of the snow particles remained almost unchanged even after 50 days. In comparison to the conventional method (Heggli et al., 2009), which necessitates the preparation of a replica from a fixed sample, this method offers the distinct advantage of enabling direct imaging of the fixed sample by X-ray CT. Additionally, solidified 1-bromododecane is distinguishable from ice due to its opacity, and it does not exhibit polychromatic properties under polarised light, which further allows for its differentiation from ice. Moreover, it can be readily employed for microscopic X-ray Laue analysis. It is thus anticipated that this approach will not only facilitate the fixation of samples for X-ray CT, but also contribute to the physical analysis of snow cover in general.

ACKNOWLEDGEMENT

This study formed part of the project entitled ‘Research on combining risk monitoring and forecasting technologies for the mitigation of diversifying snow disasters’. Additionally, this study was supported by JSPS KAKENHI, Grant Numbers JP21H04601 (HaMaNAS PJ).

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