Ice formation via deposition nucleation on mineral dust and organics: dependence of onset relative humidity on total particulate surface area

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Received 19 December 2007
Accepted for publication 6 March 2008
Published 30 May 2008
Online at stacks.iop.org/ERL/3/025004

Abstract
We present ice nucleation results for Arizona test dust, kaolinite, montmorillonite, silica, silica coated with a hydrophobic octyl chain, Gascoyne leonardite (a humic material), and Aldrich humic acid (sodium salt). The focus was on deposition mode nucleation below water saturation at 233 K. Particles were deposited onto a hydrophobic cold stage by atomization of a slurry/solution and exposed to a constant partial pressure of water vapor. By lowering the temperature of the stage, the relative humidity with respect to ice (RHᵢ) was gradually increased until ice nucleation was observed using digital photography. Different numbers of particles were deposited onto the cold stage by varying the atomization solution concentration and deposition time. For the same total particulate surface area, mineral dust particles nucleated ice at lower supersaturations than all other materials. The most hydrophobic materials, i.e. Gascoyne leonardite and octyl silica, were the least active. For our limit of detection of one ice crystal, the ice onset RHᵢ values were dependent on the total surface area of the particulates, indicating that no unique threshold RHᵢ for ice nucleation prevails.

Keywords: ice nucleation, deposition nucleation, mineral dust, organic particulates

1. Introduction

Cloud–aerosol interactions still provide some of the greatest uncertainties in predictions of climate change (IPCC 2007). In part, this results from an incomplete understanding of the processes of cloud formation, particularly those giving rise to mixed phase and ice clouds. The two basic mechanisms of ice formation in the atmosphere are homogeneous and heterogeneous nucleation. The former usually occurs at high supersaturations and low temperatures, as are encountered at times in the upper troposphere. However, it has been known that heterogeneous mechanisms prevail as well, given the relatively high temperatures and low supersaturations at which ice can form (Pruppacher and Klett 1997).

Recent field studies have confirmed the importance of heterogeneous nucleation emphasizing, for example, the role of mineral dust particles as ice nuclei (IN). Taking just one example, Prenni et al (2007) examined ice formation processes in cumulus clouds in Florida during CRYSTAL-FACE and found that the highest concentrations of ice crystals occurred on the same date as the highest concentrations of heterogeneous IN when ice crystal residuals were tested for nucleation in a continuous flow diffusion chamber (CFDC) while flying through Saharan aerosol layers (DeMott et al 2003b). By taking CFDC measurements of anvil particle residuals they concluded that the high ice crystal numbers were due to intake of Saharan dust by the clouds.

Knowing that heterogeneous nucleation occurs in the field, the challenge to the laboratory community is to identify
the materials that act as good IN and to express the rates of nucleation in a form that can be usefully employed in cloud-climate models. In that regard, early studies have identified both mineral dust materials and organics as possible candidates. For example, Higuchi and Fukuta (1966) and Roberts and Hallett (1968) showed that the montmorillonite and kaolinite clays are good IN at temperatures much higher than required for homogeneous nucleation. And Parungo and Lodge (1965) tested a series of organics, i.e. substituted phenols and benzoic acids, for ice formation abilities in the immersion and deposition mode. It was found that there was a free energy relationship between molecular structure and nucleating ability.

More recent studies take advantage of new experimental techniques and of our improved knowledge of particulate types that may be present in the atmosphere. For example, differential scanning calorimetry has been used to show that solutions containing immersed solid oxalic, adipic, succinic, phthalic and fumaric acids were found to be poor IN. By contrast, the dihydrate of oxalic acid was found to be a quite efficient (Zobrist et al 2006). This was in partial agreement with a study carried out by Parsons et al (2004) which showed that some dicarboxylic acids are inefficient IN. It is also consistent with the findings that another soluble solid, ammonium sulfate (AS), can also act as a heterogeneous IN (Zuberi et al 2002, Abbatt et al 2006, Shilling et al 2006). Other organics are also active at warmer temperatures between $-10$ and $-17^\circ C$. In particular, Ochshorn and Cantrell (2006) found that long-chain alcohols promote ice nucleation by stabilizing ice-like clusters with their hydrophilic tails. In the case of mineral dusts, a number of studies have shown different dust types to be efficient IN in the deposition mode at temperatures below 240 K. For example, Asian dust studied by Archuleta et al (2005) and Field et al (2006), Arizona test dust (a reference dust material) studied by Knopf and Koop (2006) and Möhler et al (2006), kaolinite studied by Dymarska et al (2006), and Owens Lake dust (a significant dust source in the western hemisphere) studied by Koehler et al (2007) were all found to be good IN, forming ice at RHs well below those required for homogeneous freezing requirements.

With these past studies in mind, we focus in this work on the role of deposition nucleation under the largely unexplored cold conditions of the mid and upper troposphere, where cirrus clouds form. We are particularly interested in comparing the relative efficiencies of a range of mineral dust types and organics using the same experimental technique that is sensitive to the nucleation of a single ice crystal from a large total number of particles. This work extends earlier measurements of Kanji and Abbatt (2006) that showed efficient deposition nucleation onto a number of pure minerals as well as Saharan dust. Particularly interesting was the dependence of the ice onset RH$_I$ on the number of Saharan dust particles being examined, i.e. demonstrating that there is no unique threshold at which nucleation occurs. In this work, we continue to address mineral dust samples by extending the work to the dependence of the ice onset RH$_I$ on surface area for Arizona test dust, montmorillonite and silica. One question we seek to answer is whether the findings for Saharan dust are general or not. As general motivation, dust and its components are prime candidates for study given that dust is often associated with ice particles and has been shown to be an active IN in the cirrus regime (e.g. DeMott et al (2003a)).

A second focus of the paper is on organics. In particular, the residence time of crustal material can be on the order of several weeks (Prospero 1999). And so, it is likely that physical and chemical processing will produce coatings of various types on the dust. Ice formation on dust that is immersed in AS has been investigated before (Zuberi et al 2002, Hung et al 2003), however the role of organic coatings has not. In this regard, we have also studied the effects of placing a highly hydrophobic material on a silica surface. In later work, we will study how oxidation of this coating may affect IN abilities. With the wide range of atmospheric organics, we have also focussed on humic materials given that they may be lofted as particulates and because they act in some way as a model of humic-like substances in the atmosphere (Grabar and Rudich 2006). Lastly, we have studied oxalic dihydrate acid given the findings of Zobrist et al (2006) described above.

The results of the study indicate that a wide range of materials can act as deposition mode IN with a consistent trend of increasing activity from mineral dusts to hygroscopic solids to hydrophobic materials. Also, we confirm the findings from Kanji and Abbatt (2006) that the ice onset RH$_I$ for a specific particulate type is dependent on the number of particles being studied, i.e. their total surface area.

2. Experimental methods

2.1. Experimental approach

Ice nucleation measurements were obtained using the flow system shown in figure 1. The detailed instrument design and performance characteristics are described in Kanji and Abbatt (2006). Briefly, aerosol particles are deposited onto a Teflon-coated, copper cold stage that is housed in a vacuum chamber and is in thermal contact with a re-circulating chiller to allow temperature control. The Teflon-coated stage is the coldest part of the system and is exposed to a constant partial pressure of water while its temperature is reduced, to increase RH$_I$. The entire Teflon surface of the cold stage is under observation by a digital camera (figure 2) that captures ice formation as soon as an ice crystal forms (minimum detectable size 100 μm). The system is sensitive to one ice crystal and the RH$_I$ at which ice
is first observed is defined as the onset RH$_i$. The RH$_i$ changes by 1% every 6 s as the stage cools. Upon observation of an ice formation event, the experiment is concluded. In this study the measurements are conducted at 233 K to mimic the cirrus regime. The error associated with the reported RH$_i$ is ±2%, arising largely from the uncertainty in temperature (Kanji and Abbatt 2006).

As described in detail previously (Kanji and Abbatt 2006), the operating principle of this technique is that the first ice nucleation event over the entire cold stage occurs on a single particle and that it is observable with the digital camera. In particular, the rate of growth of the crystal to the minimum detectable size is essentially instantaneous within our observational timescale. Thus, by varying the numbers of particles on the stage, we can measure the dependence of the ice onset RH$_i$ on the total surface area present. This allows us to evaluate lower limits to nucleation rates, as described later. A unique feature is that our technique is sensitive to a very low fraction of particles on the cold stage participating in ice formation on the cold stage (down to 1 particle in 10$^5$).

2.2. Sample preparation

The particulates were prepared as aqueous suspensions/solutions with 18 MΩ water, except in the case of silica. For silica and octyl-coated silica, methanol was used because it was shown (Kanji and Abbatt 2006) that water leads to clumping of silica particles on the cold stage resulting in a non-uniform distribution. The particulates were atomized from a constant output atomizer (TSI model 3076) at specific concentrations and deposition times so as to vary the total surface area of aerosols on the stage. The particles were then exposed to low RH conditions at low pressure and room temperature (RH < 1%) for 18 h to remove water. Arizona test dust (Powdered Technology Inc., 0–5 μm), kaolinite (KGa-lb, Clay Mineral Society, Source Clays Repository), oxalic acid (Aldrich Chemicals), pure silica (Lancaster Synthesis Inc., 1.5 μm), octyl-coated silica (see below for preparation), Aldrich humic acid (Aldrich Chemicals, sodium salt) and Gascoyne leonardite (International Humic Substances Society, IHSS) were all prepared in water solutions/suspensions. We anticipate that the oxalic acid will be present as the dihydrate on the cold stage before ice nucleation because the RH at which the transition from anhydrous to dihydrate occurs has been shown to be as low as RH$_w$ = 20% at 258 K and has a very weak temperature dependence (Braban et al 2003).

The octyl-coated silica was prepared using a synthesis from Usher et al (2003). Briefly, the functionalization technique is that the first ice nucleation event over the entire cold stage occurs on a single particle and that it is observable with the digital camera. In particular, the rate of growth of the crystal to the minimum detectable size is essentially instantaneous within our observational timescale. Thus, by varying the numbers of particles on the stage, we can measure the dependence of the ice onset RH$_i$ on the total surface area present. This allows us to evaluate lower limits to nucleation rates, as described later. A unique feature is that our technique is sensitive to a very low fraction of particles on the cold stage participating in ice formation on the cold stage (down to 1 particle in 10$^5$).
surface was fully functionalized or not, we note that the same procedure used by Usher et al (2003) led to a full monolayer coverage, as determined by solid state NMR data.

2.3. Surface area determination

The deposited samples were analyzed under an optical microscope for surface area measurements (Kanji and Abbatt 2006). Several optical images (e.g. figure 3) were taken to ensure a uniform distribution across the stage. The images were used to obtain a size-resolved particle count per image area. This was then applied to the entire sample to determine a total surface area assuming the particles are spherical.

3. Results

Arizona test dust (ATD) nucleates ice at relatively low RH, with RH decreasing with increasing surface area (figure 4). This is consistent with there being more nucleation sites available when the surface area is higher, i.e. the probability of nucleating ice becomes higher. As mentioned earlier, the unique nature of our experiment that allows us to see this dependence is that the criterion for establishing the onset relative humidity is that a single ice crystal forms. By increasing the total surface area on the cold stage, we increase the probability that a site that is active for the conditions of the experiment (i.e. temperature, relative humidity) will be present on the particulates. We assume that the distribution of active sites per unit surface area is uniform regardless of the size of the particulate, i.e. the same results would be seen whether a large number of small particulates or a small number of large particulates were used, provided that the total surface area were constant.

The same trend is also seen for data points for Saharan dust from Kanji and Abbatt (2006), which have been plotted against surface area for the first time, confirming the generality of this finding. However, the slopes of the two trends differ with ATD exhibiting a stronger dependence on surface area and lower overall activity. It is not clear whether this difference can be attributed to the difference in composition of the two dusts or the preparation methods before the sample is received. In both cases ice nucleation occurs well below water saturation, which is at RH$_i$ = 147% at 233 K.

Knopf and Koop (2006) also showed that deposition mode nucleation is the favored pathway for ice formation onto ATD below 240 K, whereas Archuleta et al (2005) showed this to be true for Asian dust. In the study by Knopf and Koop (2006) RH$_i$’s for ATD as low as 105% were observed, but it should be noted that this was for pre-activated particles. Pre-activation is a term used to describe the effect of a previous ice formation event on a particle (Higuchi and Fukuta 1966, Roberts and Hallett 1968). This effect was not observed in this work, when we performed ice nucleation on the same sample multiple times. The cause for the difference between this work and Knopf and Koop (2006) could be because between each run we warmed our sample up to room temperature and therefore dried it to a very low RH (<3%). Even though Archuleta et al (2005) saw efficient deposition mode nucleation on Asian dust and also observed that as particle size increases the RH$_i$ decreases, their RH$_i$ values are higher than seen for ATD in this study. We offer two explanations for this. First, that in this work we use polydisperse particles in the size range 0.5–5 μm whereas they used monodisperse particles and their largest size was 200 nm, which will present much lower surface areas than we have on our cold stage. Secondly, our technique is sensitive to the first ice nucleation event over the entire sample, whereas their reported values are for 1% of particles nucleating.

Silica, montmorillonite and kaolinite, components that partially make up the composition of the mineral dusts, were also studied (figure 4) and were observed to nucleate ice within the range of the Saharan dust and ATD. Montmorillonite also showed the same surface area dependency as ATD and
nucleated ice efficiently at 233 K, as recently observed by Dymarska et al (2006). It can be concluded that mineral dusts are very efficient at nucleating ice in upper tropospheric conditions, but with somewhat varying efficiencies.

To compare dust to hygroscopic aerosols in the atmosphere, we also studied ice nucleation onto oxalic acid dihydrate and Aldrich humic acid. These aerosols were found to be less efficient than mineral dusts. For example, oxalic acid dihydrate exhibited the same trend, where higher surface area gave lower RHₐ. However, it is not as good an IN as mineral dust, with higher onset RHₐ’s for the same total surface area. The work of Zobrist et al (2006) indicates that this material can act as an immersion IN and our studies confirm it also acts as a deposition IN. Aldrich humic acid, which exists as a sodium salt, was found to be less efficient than mineral dusts as well, but with comparable efficiency to oxalic acid dihydrate (see figure 4), i.e. the data point lies along the same trend line of particulate surface area versus onset RHₐ as does oxalic acid dihydrate. These results are in agreement with a data point for another hygroscopic solid, solid ammonium sulfate, published in Abbatt et al (2006). In particular, for a large total surface area of about 0.6 cm², ammonium sulfate also acted as an IN with an onset RHₐ of 107% at 224 K.

The Gascoyne leonardite, which was visually observed to be less soluble than the sodium humic salt, was less efficient at nucleating ice than the Aldrich salt and the mineral dusts. The composition of leonardite may have a role to play given that it has a higher carbon content and lower oxygen content compared to the sodium salt, as specified by their respective suppliers. In addition the leonardite sample is composed mostly of aromatic moieties (from ¹³C NMR data, as specified by IHSS).

Finally, we studied octyl-coated silica as an example of a hydrophobic material. By functionalising the silica surface with approximately a monolayer of an n-octyl hydrocarbon, we have shown that its ice nucleability can be suppressed greatly (see figure 4). For both surface areas tested, ice was observed to form only in the range of the maximum sustainable supersaturation of the Teflon-coated stage. As such, these values for RHₐ represent lower limits to RHₐ for this material given that the nucleation may have occurred on the Teflon surface. It is clear that a hydrophobic surface exposed to an incoming water molecule will require much higher supersaturations for deposition mode nucleation to take place, given its surface properties. While we do not expect that octyl-silica will be present in the atmosphere, these results highlight how ice nucleation is a process that is highly surface-specific whereby a hydrophobic monolayer is sufficient to effectively shut down ice nucleation relative to the uncoated material. These high onset RHₐ’s for such hydrophobic materials are consistent with our findings for large surface areas of n-hexane soot that also did not nucleate ice below the maximum sustainable supersaturation (Kanji and Abbatt 2006).

4. Discussion

With respect to specific findings, our experiments with the octyl-coated silica indicate that surface functional groups determine the conditions and efficacy of deposition mode nucleation. Currently, we are not able to precisely delineate the surface properties that give rise to the varying efficiencies observed within the different types of mineral dusts. This is an area that needs to be further investigated, however it is possible that the preparation or processing of the different dusts may lead to some of the differences observed. As an example, Koehler et al (2007) found that generating Owen Lake aerosol from an aqueous suspension led to no size dependency for onset of ice nucleation, whereas this was not the case when the aerosols were generated dry. We also note that the conclusions made in this paper pertain to temperatures of 233 K and deposition freezing. At higher temperatures, it may be that other nucleation mechanisms—such as condensation freezing—will prevail.

Given that we have the dependence of the onset RHₐ on total surface areas and to put our results in a form that can predict behavior in the atmosphere, we can calculate (see figure 5) the lower limit to ice nucleation rates using the following relationship:

\[
J_{\text{lower}}(\text{events cm}^{-2} \text{s}^{-1}) = \frac{1}{(\text{SA})(t)}
\]

where the numerator is set to unity because one ice crystal is the detection limit of our system and the criterion by which we define ice onset. SA is the total surface area of particles on the stage and t is the time that the particles are exposed to a given RH, i.e. 6 s for our experiments. We refer to this as a lower limit to the nucleation rate because it may take the particles less than 6 s to form ice. The uncertainty associated in the calculation of \( J_{\text{lower}} \) is ±30%, mostly resulting from the error in surface area of the particles (±20%). Note, however, that we assume the particles are spherical. Errors associated with areas at the microscopic level above those of assuming spherical particles are not considered here.
Higher RH\textsubscript{i}'s lead to higher nucleation rates, as would be expected given that the deviation from thermodynamic equilibrium is higher. Thus, our data are consistent with the nucleation rate being not only dependent on the chemical nature of the particles but also the supersaturation with respect to ice (Bryant \textit{et al.} 1959, Mason 1960, Huffman 1973). In figure 5, nucleation rate data points from Archuleta \textit{et al.} (2005) are included which show a similar trend for Asian dust, i.e. that nucleation rate is dependent on RH\textsubscript{i}. Note that this dependence is sometimes expressed as a dependence of the onset RH\textsubscript{i} on the size of a particle. This is a valid approach if the threshold for determining ice onset is constant from one experiment to another. For example, for a specified threshold of 1 ice crystal per fixed number of particles, it would be expected that the onset RH\textsubscript{i} would be higher if smaller particles are used. This is indeed the case in the Archuleta \textit{et al.} (2005) results. We note the value of comparing nucleation rates measured with different experimental approaches, as this allows for more confidence in prediction of rates appropriate for atmospheric conditions.

The particulates tested for ice nucleation were chosen to be surrogates for aerosol materials found in the atmosphere. Using the same experimental technique for each, we found that mineral dusts were most efficient at nucleating ice followed by hygroscopic solids and then non-hygroscopic solids. It is widely recognized that a variety of aerosol types are observed externally and internally mixed in any air mass. The range of aerosol types extends from internally mixed sulfate–organic particles, to soot, mineral dust, and biogenic. As pointed out in Abbott \textit{et al.} (2006), the degree to which ice nucleates upon this complex mixture will be dependent upon both the relative surface areas of each aerosol present and their intrinsic ice-forming capabilities. The results here indicate that it is likely that mineral dusts will be the first materials that ice will form upon under the cold conditions of the mid and upper troposphere. Once these dusts have been removed by either ice nucleation or CCN processing, nucleation will then proceed via heterogeneous nucleation onto hygroscopic, soluble solids, such as crystallized organics, ammoniated sulfates or humic-like substances. Although these materials appear to nucleate ice with similar efficiencies, the overall importance of each will be determined by their relative abundances in the atmosphere.

Acknowledgments

The authors acknowledge NSERC for the support of these studies. The authors also thank Dr Dan Cziczo for helpful discussions on the importance of deposition nucleation of different aerosol types in the atmosphere.

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