Reviewer comments have been reproduced in bold and author responses in regular typeface.

Referee report on "Ice nucleation properties of K-feldspar polymorphs and plagioclase feldspars" by André Welti et al. The paper "Ice nucleation properties of K-feldspar polymorphs and plagioclase feldspars" by A. Welti, U. Lohmann, and Z. Kanji continues the series of experimental studies aimed at understanding the ice nucleating (IN) properties of the feldspar component of atmospheric mineral dust (MD). In particular, the role of potassium-rich feldspar and its modifications requires a thorough characterization due to its abundance in the atmosphere and the easiness it triggers ice nucleation in supercooled water or supersaturated water vapor.

Apart from reporting the results of freezing experiments, the manuscript suggests that some qualitative correlations between the feldspar genesis and its IN activity could be established. However, as many factors (pressure, temperature of crystallization, cooling rate, presence of volatile fluxes, shearing stress, variations in bulk composition of magmatic fluid, etc.) can influence the structure of the K-feldspar in igneous rocks, the authors do not attempt specifying a mechanism responsible for the strong variation of IN activity of feldspar. Even so, establishing such correlations could provide a useful tool to predict what kind of feldspar might me expected to be a better IN particle. From this point of view, the correlation between median freezing temperature and Rb/Sr ratio appears to be especially promising (more on that below). I therefore support the publication of the manuscript but would like to attract authors' attention to the following issues:

1. The manuscript reports size resolved immersion freezing experiments on 9 new types of feldspar using a single-particle continuous flow diffusion chamber (CFDC), and thus presents a large body of new data that could be used for parameterization of IN activity of feldspar-containing MD aerosols in cloud models. However, the data is not presented in terms of ice nucleating active site (INAS) density, which became standard for reporting the IN properties of aerosol particles over the past years. Although the possibility of such analysis is indicated (end of section 4.1) and obviously has been conducted for amazonite sample (Ickes et al. 2017), it was not extended to other samples and sizes in this manuscript. This is particularly unfortunate, as the experimental conditions (using mobility-selected single feldspar particles as INPs and well-defined thermodynamic conditions in the CFDC) are ideally suited for performing the INAS density-based analysis. This would substantially simplify the comparison of the results with the numerous data of recent IN experiments on feldspar. I strongly recommend including this analysis into the revised version of the manuscript. Additionally, the size distributions of aerosol particles used in the experiments have to be added to the manuscript (as a supplement) or made available for anyone who would be interested to calculate the INAS densities.

We added the figure below as Appendix A in the revised manuscript showing INAS densities. Compared to the experimental data shown in Fig. 3 of the manuscript, INAS density (n_s) scales the frozen fraction (FF) by the particle surface area according to:

$$n_s = \frac{ln(1-FF)}{A}$$

Where A denotes the geometric surface area, calculated as $A = 4\pi\left(\frac{\mathrm{d}}{2}\right)^2$, with d being the selected mobility diameter. Because measurements are conducted with quasi monodisperse particles, INAS densities can easily be calculated without the need for individual size distributions.

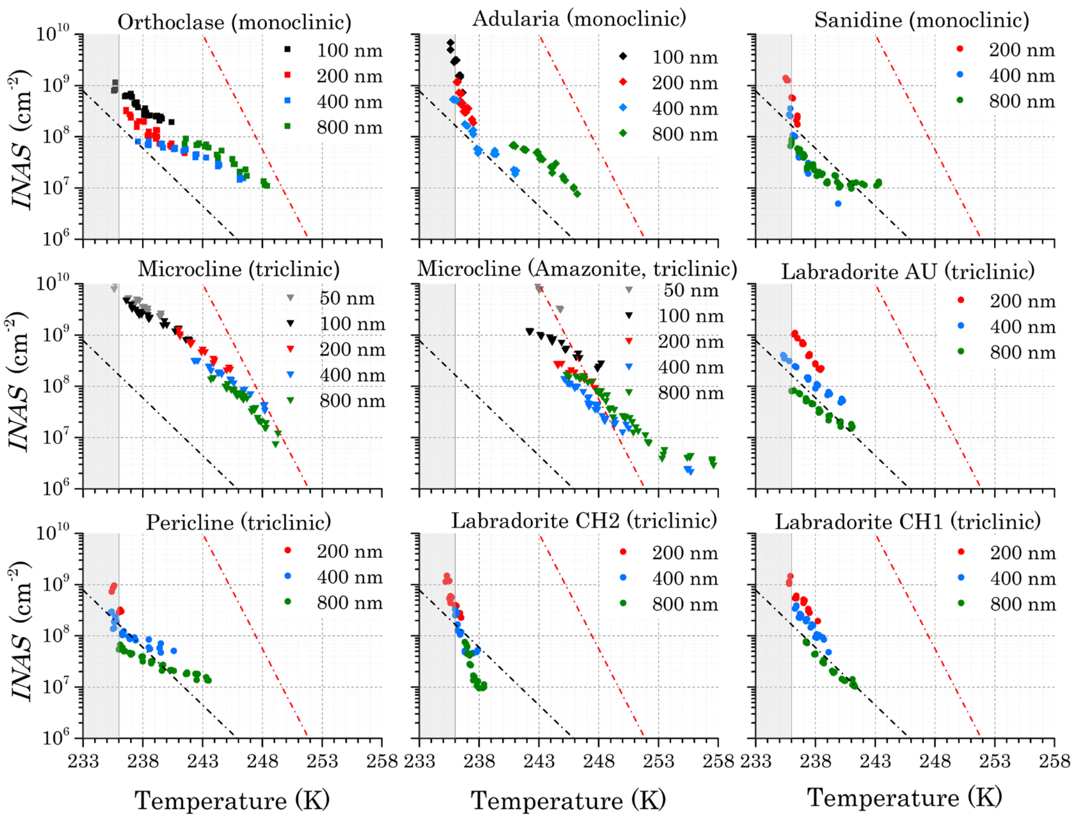


Figure A1 (in revised manuscript): Ice nucleation sites per surface area (INAS) of 9 feldspar samples as a function of temperature and size corresponding to observed frozen fractions as shown in Figure 3. The upper (80-100%) and lower (0-20%) frozen fractions have been omitted to exclude saturation errors from the detection.

2. That the IN properties of all feldspars are size dependent, is convincingly shown by the figure 5. It is also apparent that the T50 values for the smallest particle sizes in each curve lie in the homogeneous freezing range, implying that the T50 values have been calculated for all droplets, frozen both heterogeneously and homogeneously. This doesn't make sense to me. What is the purpose of reporting the median freezing temperature for particles that did not induce freezing of supercooled droplets? Was it not possible to correct the frozen fraction for homogeneously frozen droplets? Again, this is a very strong argument for reporting the INAS densities instead of median freezing temperatures.

In cases when homogeneous and heterogeneous nucleation contribute to ice formation, it is beyond the experimental capacity to accurately determine the corresponding fractions. When the measured T_{50} lies within homogeneous freezing temperatures, all that can be deduced with confidence is that the corresponding sample is not an efficient ice nucleator in the immersion mode at temperatures above homogenous freezing temperature. In response to the Reviewer request, we instead removed the T_{50} data from Figure 5 if a frozen fraction of 50% was not reached above the homogeneous freezing temperature. As requested by the Reviewer, INAS densities have now been included in the appendix (see comment above). It can be seen from these plots that normalizing frozen fractions by the particle surface area in the homogeneous nucleation regime, does not produce overlapping (except for amazonite), but rather parallel displaced INAS densities. This is an artifact of the *INAS* scaling, treating homogeneous freezing that is not dependent on the size of immersed particles, the same as heterogeneous freezing.

3. Another major (critical) comment is that the reported correlations between crystalline structure, trace element composition, and IN activity are not sufficiently supported by statistics. The authors state that IN efficiency is correlated with the degree of ordering of Si and Al atoms in the feldspar framework, implying that there must be a mechanism responsible for this correlation. This mechanism is not discussed in the manuscript, nor is the suggested correlation expressed in any quantitative manner (although such quantity can be derived, see for example, (Smith 1970, Brown and Parsons 1989). I doubt that such correlation could be of practical use unless a mechanistic explanation is suggested or a quantitative correlation analysis is performed.

In the initial manuscript, we already explain that we cannot give a reason for the correlation of crystal structures and the ice nucleation activity of feldspars, since the crystal structures do not match that of ice. We agree the explanations given in the manuscript are qualitative (not supported by statistics) as pointed out by Reviewer 2. However, in the reference given by the reviewer (Smith, 1970), the author also confirms that in theory a quantitative description of order-disorder of crystal structure is possible but this has not been done quantitatively as it poses additional complexity and challenges. As such even in Brown and Parsons (1989), the second reference suggested by the reviewer, only a qualitative description is suggested. Quantifying the degree of order in feldspar samples would likely be a full study of its own and much beyond the scope of this work.

The intention of discussing crystalline structure and trace elemental composition is to highlight the possibility that several feldspar properties are connected (e.g. by the conditions of rock formation), and more detailed investigations are needed to disentangle their importance for the ice nucleation activity and the mechanism. Because the lack of a mechanistic understanding we prefer not to provide statistical correlations that could be misunderstood as parametrizations and hope this study to serve as a stepping stone for future studies aiming to establish a quantitative understanding.

Nevertheless, we suggest that it is possible to identify properties which can be used as qualitative tracers (not causes) for the ice nucleation activity. Focus is therefore placed on the most practical tracer, which we think is the trace elemental composition, for which qualitative agreement is shown in Figures 7 and 8.

3. With respect to the trace elements composition, the number of investigated samples is definitely too low to draw any particular conclusion. I agree that for the presented choice of samples, the correlation between the IN activity and the ratio of Rb/Sr content is striking. On the other hand, the partitioning of trace elements takes place as feldspar is cooling and forming separate phases, so that Rb replaces K and Sr replaces Ca; thus the Rb/Sr ratio is indicative for the position of feldspar on the ternary phase diagram (An-Ab-Or) and the absolute concentrations of trace elements depends on the cooling/crystallization process and abundance of trace elements in particular magmatic fluid (Parsons et al. 2008). As a consequence, the high Rb content is expected in low microcline and the observed correlation just reflects the correlation between the IN activity and microstructure of feldspar. It is still very interesting that the most IN active sample has also the highest Rb content, an order of magnitude higher than the second-best microcline. I think this fact deserves to be highlighted in the manuscript, even if no mechanistic explanation can be offered at the moment. With the amount of experimental data on IN properties of feldspars that recently became available, it would be relatively easy to improve the statistical analysis and to show if the connection of IN activity and Rb content is not just a coincidence. I would really like to see such analysis in the manuscript or in the follow-up work.

We fully agree with the reviewer that we should highlight better that a replacement of K by Rb results in a lower microcline polymorph, yet a higher Rb/Sr ratio, thus a higher ice nucleation activity suggesting that observed correlation reflects a potential relationship between ice nucleation activity and feldspar microstructure. We also agree, with more samples than those used here, a systematic analysis of available data in light of the findings in this manuscript could be a promising follow-up study. Following the Reviewer recommendation, we added:

Page 10 line 31-page 11 line 3:"The Rb/Sr ratio is indicative for the position of feldspar on the ternary phase diagram (Figure 1) and the absolute concentrations of trace elements depends on the cooling/crystallization process and abundance of trace elements in particular magmatic fluid (Parsons et al., 2009). The replacement of K by Rb results in a lower microcline polymorph, and a higher Rb/Sr ratio, correlating to higher ice nucleation activity. This suggests that the observed correlation reflects a possible relationship between ice nucleation activity and feldspar microstructure."

Page 12, line 20-23: "More ice nucleation active samples show higher Rb/Sr ratios, e.g., the most active microcline sample has an order of magnitude higher Rb/Sr ratio than the second-best microcline. Therefore, Rb/Sr ratios could serve as tracer for highly ice nucleation active feldspar particles or even help to differentiate sources of feldspars acting as INP at specific temperatures."

4. I am also confused by the discussion of Pb content and its correlation with the median freezing temperature. The authors state that "The best linear correlation between a single compound and the ice nucleation activity based on T50, was found for the Pb content in the feldspar samples (Figure 7).", but obviously the Pb content in microcline (better IN) is lower than in orthoclase (weaker IN). The quality of the correlation as expressed by Pearson's r-coefficient is apparently influenced by the low Pb content in the plagioclase and high-temperature disordered alkali feldspar, which has been formed before any trace elements partitioning could take place. I think the plagioclase feldspars should be excluded from the correlation analysis. If you do that, the correlation between T50 and Rb/Sr content ratio would be even better than for Pb and the T50 as a function of Rb/Sr would be also monotonically rising.

On a side note, if Pb is normalized by Nd content, the correlation between T50 and Pb/Nd ratio becomes very clear, with T50 as a function of Pb/Nd staying constant for plagioclases and then monotonically rising from the level of adularia and sanidine towards amazonite. This is just to illustrate that such correlations can be constructed very easily and most of them are probably meaningless, unless a larger set of samples is analyzed or an underlying physical and chemical mechanism is suggested. I encourage the authors to do so.

The decision to compare T_{50} to Pb, Rb and Sr is based on the random forest analysis which identified these three as the most promising predictors. The *r*-coefficients in the text, give the correlation between these elements and not to the T_{50} as the Reviewer states, as such we will clarify the statement so that a potential reader does not mis-understand the correlation. Furthermore, we share the view of the Reviewer that the correlation of Pb content and T_{50} is biased by the low content in the plagioclase feldspars, the limited number of samples and potential sub sequential uptake. It is because of Pb is suggested by the random forest analysis that we discuss it and then argue to discard it as a predictor. We thank the Reviewer for pointing out the possibility to create a monotonic correlation between T_{50} and Pb/Nd. However, in contrast to Rb/Sr, we did not find any literature suggesting an interpretation of this ratio.

To clarify we now write:
Page 10, line 24-25 "Based on these arguments and that the correlation of Pb content to T_{50} is not monotonic (e.g., lower Pb content in microcline than orthoclase, Figure 7) we focus on other predictors suggested by the Random Forest analysis.

Page 11 line 3-5: "We note that a monotonic correlation between T_{50} and a Pb/Nd ratio can also be constructed, but no interpretation of the implication of such a ratio could be found in the literature and neither was Nd suggested as a predictor by the Random Forest analysis."

5. With respect to the potential predictability of IN activity based on the relative concentrations of trace elements, I have to point out that the measurements reported here have been conducted exclusively with the single-crystal samples. In case of real-world atmospheric mineral dust, the majority of feldspar would be coming from phenocrysts (inclusions of feldspar in e.g. granite matrix), where the trace elements content could be very different. Again, the idea of using partitioning of trace elements as a predictor for IN efficacy is very attractive and promising, but must be explored deeper and supported by extensive dataset.

We already discuss this limitation of the study in regard to atmospheric aging in the initial manuscript page 10 lines 20-24 (page 11 lines 26-30 in revised manuscript) but we clarify this further prompted by the Reviewer comment and added the following to the revised manuscript:

Page 11, line 31-32: **"**Additionally, a majority of airborne feldspar particles could be coming from phenocrysts (inclusions of feldspar in e.g. granite matrix), where the trace element content could be different."

Specific remarks (ordered according to page and line number, citations given in italic):
1-14. The sentence "Ice nucleation is most efficient on the crystallographic ordered, triclinic K-feldspar species microcline, while the intermediate and disordered, monoclinic K-feldspar polymorphs orthoclase and sanidine nucleate ice at lower temperatures." strongly implies a causality between the degree of ordering and IN efficacy. Since such causality is not supported by the experimental data of the manuscript, I suggest that the sentence should be reformulated.

We agree with the Reviewer and in order to limit the statement we now write (Page 1 line 14):
"Amongst the investigated samples, ice nucleation is most efficient on the crystallographic ordered, triclinic K-feldspar species microcline, while the intermediate and disordered, monoclinic K-feldspar polymorphs orthoclase and sanidine nucleate ice at lower temperatures."

1-26 (Introduction). The introduction would greatly benefit from including a discussion of partitioning of the trace elements in feldspars. As mentioned above, the possible correlation between trace elements and IN activity can open a unique opportunity to classify the feldspars on a single-particle basis, for example in a laser ablation mass spectrometer.

We think it would be premature to discuss the partitioning of specific trace elements in the introduction, as the correlation of the trace elements to the ice nucleation activity is shown as a result of the current study. However, prompted by the Reviewer's suggestion, we do include a paragraph on trace elements and their importance to feldspar microtexture and characteristics, in the introduction (see page 3 lines 19-27). Additionally, we have added discussions on partitioning of the trace elements in the following places: Page 10 lines 32 to page 11 line 5 and Page 12 lines 20=23.

2-9. The degree of order (or disorder) of Al3+ and Si4+ is not something that an average atmospheric scientist would be familiar with. This sentence requires explanation.

We elaborate further on this topic on page 2 line 14-15:
"The four units are made up of one Al^{3+} and three Si^{4+} ions. In a disordered state Al^{3+} can be found in any one of the four tetrahedral sites while in an ordered state Al^{3+} occupies the same site throughout the crystal (Nesse, 2016)."

3-2. (Whale et al. 2017) did not conduct systematic study of crystalline structure in terms of disorder, although they report the fraction of orthoclase in their perthitic samples. A careful analysis of their samples could have reveal a correlation. Please elaborate on that issue.

Whale et al. 2017 do discuss order-disorder in their article. In the supplementary material they give a comprehensive summary on the order-disorder of K-feldspar and they have measured it by Raman spectroscopy for some of their samples. It is mentioned that it has been under discussion in relation to ice nucleation ability in other studies. By pointing to examples where disordered feldspar samples showed high ice nucleation activity when measured with their setup they conclude that it is not a necessary feature. However, different from the current study, their experimental setup is sensitive to much rarer particle features (ice active at much higher temperatures) than a single particle investigation, thus the conclusions might not be transferrable.

3-14. (Niedermeier et al. 2015) has reported nucleation ability of size-resolved K-feldspar sample in single-particle immersion mode and according to the manuscript, the microcline data from your study has been previously published in (Ickes et al. 2017). So the statement "for the first time" must be either removed or explained, what exactly has been done in this work for the first time.

We removed the statement (Page 3 line 29).

3-21 (Section 2). A more detailed description of the sample origin and the XRF analysis would be very helpful here. Was XRF analysis the only basis for identifying the samples or have you performed the powder XRD analysis, too? Could you speculate on how the chemical composition of single aerosol particles would be related to the chemical composition measured for bulk samples?

Composition of the samples were additionally determined by Rietveld refinement of powder XRD pattern. Note that this method is subjective to the fitting procedure to some degree. Results are given below and have been added as Appendix B in in the manuscript.

Sample	orthoclase	adularia	sanidine	microcline	plagioclase	quartz	Others
Orthoclase	70				15	5	10
Adularia		100					
Sanidine			100				
Microcline				90	10		
Microcline (Amazonite)				77	22	1	
Labradorite (AU)					59	12	29
Pericline					84	16	
Labradorite CH2					100		
Labradorite CH1					100		

Samples were provided from the geological collection of ETH Zürich except Labradorite (AU) which is a commercial sample sold in powder form as supplement for ceramics and Amazonite which was provided by a private collector. Because for all except Labradorite (AU), fine particles are obtained by grinding single stones, we expect the bulk chemical analysis to be a good approximation for the majority of particles. However, this was not investigated experimentally and certain compounds may be more abundant at a certain particle size.

3-27. The sample that is called "orthoclase" here should be the one closest to the ideal end-member of the alkali feldspar group (Or = $KAlSi_3O_8$). On the Figure 1, however, this sample has the same composition as amazonite with almost 20% albite. Where the name "orthoclase" came from? By the way, it is not correct to label the axes of the ternary phase diagram with fraction of K, Na, or Ca. The ternary diagram gives a sample composition in terms of weight fractions of end-members (orthoclase, albite, and anorthite), see for example (Parsons 2010). The name orthoclase is also misspelled in the legend. Please correct.

The sample was identified as orthoclase by experts and subsequent analysis (see table B1 in revised manuscript) confirmed orthoclase as the primary component. Lacking a more correct alternative, we kept the name. We have in addition added the end member names to the ternary phase diagram. We have kept the axis with fraction of K, Ca and Na since we used this analysis from the XRF to place the samples onto the ternary phase diagram as mentioned in the caption of Figure 1 already in the initial manuscript. We corrected the spelling and Figure 1 accordingly.

4-3. Table 3: What is the purpose of reporting compounds that could not be detected in any of the samples (FeO, NiO, H_2O, CO_2)? What is the difference between a "0" and N/D?

The respective compounds have been removed. "0" indicates a reliable measurement of absence of a compound, while N/D stands for not detected.

6-21:22. This kind of analysis has been published even before (Ickes et al. 2017) and should be mentioned here. See, for example (Wright et al. 2013, Niedermeier et al. 2015, Peckhaus et al. 2016) to name just a few.

We added to Page 7 line 9-10: "Parametrizations for other feldspar samples can be found in e.g., Niedermeier et al., 2015; Peckhaus et al., 2016"
Wright et al., 2013 did not report a feldspar parametrization.

7 (Section 4.2) This section should be expanded to include more thorough size dependence analysis, ideally complimented by the INAS density calculations. The T50 should be corrected to account for the homogeneous freezing of droplets containing the smallest particle sizes. Some specific question here: why would one expect linear dependence of T50? The median freezing temperature is a function of cooling rate and residence time, have you taken this into account when comparing T50 from your measurements and data from Atkinson et al., (2013)? 7-9. "The minimum size triggering immersion freezing is found to be 50 nm microcline particles". In the Sample preparation section you mention that a "substantial fraction of larger multiple charged particles… among the 50nm particles can be expected". How substantial is this fraction? Since the frozen fraction ends in the homogeneous freezing regime at 0.3 (Figure 3), is there a way to decide if the 50 nm microcline particles have been responsible for freezing at all or only the multiply charged larger particles are responsible? On the other hand, the

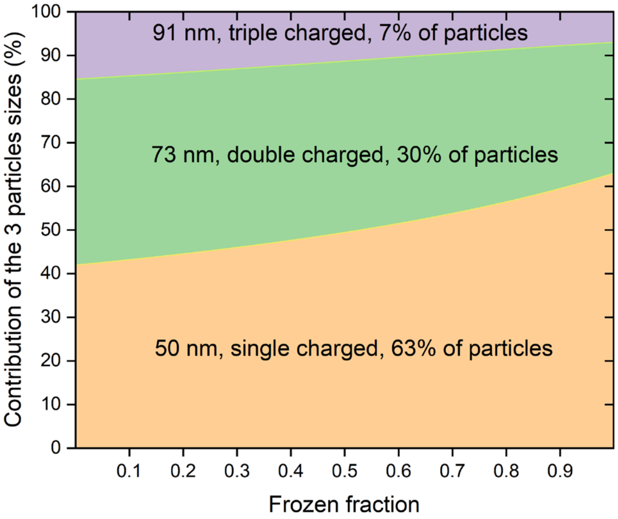
freezing curve for 50 nm amazonite particles reaches value of 0.9 suggesting that all particles have been active. Could that be that even smaller particles would be IN active?

A figure showing the calculated *INAS* density has been included in the appendix. Data points where homogenous freezing strongly contributes to the 50% frozen fraction have been deleted from Figure 5.

A linear dependence of T_{50} in a lin-log plot is expected for CNT based parametrizations using single contact angle or a log-normal contact angle distribution (Welti et al., 2012, Fig. 10).

We did not apply a correction for time dependence when comparing to Atkinson et al., 2013. This is not necessary as both the residence time in the current experiment (12s) and the cooling rate in the Atkinson et al., 2013 experiment (1K/min) maintain the sample at the reported temperature for a comparable duration, i.e. experimental uncertainties outweigh time dependence.

The multiple charged fraction of selected 50nm, amounts to 30% doubly charged, 73nm and 7% triple charged, 91nm particles, while 63% of particles are 50nm. . Based on the size dependence of immersion freezing and the abundance of multiple charged particles, the contribution of the 3 particle sizes at different frozen fractions can be calculated. The result is shown in the Figure below (and now shown and explained in the revised manuscript Appendix C).



We now clarify this aspect on page 5 line 21 and page 8 line 4-5.

50nm particles contribute more than 40% of ice active particles at any frozen fraction. Based on the current measurements it seems plausible that for some microcline species, particles smaller than 50nm are ice active. But it is difficult to produce such small particles.

8-25 Twinning is not just symmetrical intergrowth. In alkali feldspars twinning is interrelated with phase exsolution and Na-K exchange between phases or between feldspar and external aqueous fluids (Parsons et al. 2015).

The section was changed accordingly and the reference was added (see page 9 line 14-16 in the revised manuscript).

9-4. How thick was the Pt coating applied prior to SEM imaging? For the confinement effects to become

important the pores should be less than 10 nm in diameter, could you reach this resolution in the SEM analysis?

Sputtered films for SEM typically have a thickness range of 2–20 nm. The resolution of the SEM is 1 -1.7 nm. As no pores are detected in the SEM images, and smaller pores that could have been missed in the SEM analysis and are small enough to supress ice formation, we conclude that using the geometric surface is a better measure for the particle surface area than the BET-surface.

9-9. "The assumption that physical properties (e.g. hardness) are comparable among the tested feldspar species implies that the same degree of artificial surface features are introduced to all samples." What is the background for such assumption? Could you support it by literature data? On the page 3 line 32 I read something different: "The different polymorphs differ in physical properties (cleavage, hardness, specific weight, melting point)…" Could you clarify this point?

Feldspar do have very similar properties. As an example, their hardness is typically between 6 - 6.5 on Mohrs scale. https://en.wikipedia.org/wiki/Feldspar

We changed the sentence p.3, line 32 (now page 4 line 14) to "The different polymorphs differ in some physical properties (e.g. melting point) and are found in different rocks:…"

10-5. The discussion of kosmotropic vs. chaotropic cations is not very convincing. Why would the substitution of K+ for Rb+ increase the IN efficiency of K-rich feldspar if K+ is already kosmotropic? Or is there anything known about the degree of "kosmotropicity" for different kosmotropic cations? How many ions of Rb+ would one expect on the surface of an aerosol particle? Why some Na/Ca-rich feldspars exhibit a strong IN activity, having neither K+ nor Rb+ in their structure (like amelia albite in Whale et al., 2017)? This hypothesis should be either discussed in more detail or just omitted from the manuscript.

We clarify this discussion as suggested by the reviewer, but keep it in the manuscript as a possible second explanation, for the observed correlation between Rb/Sr and the ice nucleation activity of the feldspars (page 11 lines 3-13). To relativize the importance we added (page 11 line 13-15): "It would be necessary to quantify the concentration of cations required to influence water ordering to determine how influential Rb^+ and Sr^{2+} are for ice nucleation given their trace elemental composition.

I am looking forward to the revised version of the manuscript.

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