Reviewer comments are reproduced in bold and author responses are in regular typeface. All line numbers in the authors' response refer to the revised manuscript. Revised text is given in italics.

Review of "The Role of Contact Angle and Pore Width on Pore Condensation and Freezing," by R.O. David et al. 2019

Anonymous Referee #2

In their submitted manuscript, David et al. describe their attempts to utilize synthesized silica particles that have controlled surface pore widths, and alterations to those particles vis-a-vis functionalization, ´ to test the utility of pore condensation freezing as a physical parameterization for ice nucleation under certain atmospheric conditions. I am impressed by the detail and depth of the described studies and the submitted manuscript. However, I do feel that there remains room for improvement and clarity in an updated manuscript before acceptance for full publication in ACP. In particular, I suggest the authors strive to more clearly delineate where and when they think they can interpret their results purely on the basis of physical changes and where/when their understanding might be limited by the changes to the physical chemistry of the systems they are probing. In its current form the manuscript seeks to explain results primarily using physical parameters (i.e., size, geometry, bulk contact angle, etc.), but given the system (pore) sizes of exploration it might be that molecular-scale effects begin to play a role that cannot be dismissed.

Below I present an itemized list of thoughts and comments as I came to them in the text, which I hope put things into context. Following these comments I will return to some general items.

We thank the reviewer for the positive recommendation and for raising several points that we now address individually below and in the revised manuscript to make the paper clearer.

Itemized Scientific and Editorial Comments:

Specific Suggestions by Page and Line Number (page, line):

- **(1,29) remove parentheses around (T)**

Removed, thank you

- **(1,30) I think it advisable to keep a more general definition of heterogeneous nucleation, like that of Vali et al. 1 . There he just says "nucleation aided by the presence of a foreign substance". "ice active site" could mean many things and does not necessarily give as general an impression.**

We have reformulated to be consistent with the description provided by Vali et al. (2015*): At T > HFT ice formation takes place heterogeneously and is aided by the presence of a foreign substance (Fletcher, 1969; Kaufmann et al., 2017; Kiselev et al., 2017; Vali et al., 2015), which lowers the energy barrier required for the homogeneous nucleation of ice.*

- **(2,7) The explanation of PCF that is when water "which can exist below water saturation in narrow pores" is confusing to me. Is it not that the local saturation condition is altered? In fact the pore water is at saturation, and this is why it condenses, it is simply not at bulk water saturation.**

That is correct, as such we have revised the sentence to read: "*PCF occurs when liquid water, which can exist in narrow pores, cracks, cavities or capillaries (hereafter referred to as pores) below ambient water saturation, freezes*." on page 2 lines 8-9.

• (2, 12) I think that either "concave" should be curved, or the sign convention defined. I believe the equation is general up to the choice of sign convention for the radius of curvature.

For clarity we have now reformulated the sentence to state "*negative or concave curvature*", on page 2, line 9.

• (2, Eq. 2) To me Eq. 2 represents an amalgamation of the critical cluster size of an ice germ in the vapor and liquid phases. Given the discussion I understand the critical radius for an ice germ in the liquid phase should be presented, and this is what is indicated by σiw I believe. Typically Si would represent a pressure ratio pv/pi , that is the actual vapor pressure divided by the equilibrium vapor pressure over ice. In the presented case should the pressure ratio not refer to the pressure across the interface of the ice germ in liquid, i.e., pw/pi? See Marcolli 2 Eq. 11.

Thank you for pointing this out, indeed the ratio should be that of water and ice. The equation has been updated accordingly. The description of Eq. 2 now reads: "*where* σ_{iw} *is the interfacial energy between the ice and water interface,* v_{ice} *is the approximate volume of bulk ice, and* $\frac{p_w}{p_i}$ *is the ratio of the equilibrium vapour pressures over water and ice*." on page 2 lines 29-30.

• (3,3) The assumption of t = 0.38 nm from Schrieber et al., 2001 might warrant an additional comment. It is important to note that this value will certainly be temperature dependent, and that given the changes in surface functional group it might vary between tested samples. Given that as is it represents a sizable fraction of the pore diameter such potential changes might be important. Fig.7 from Bartels-Rausch et al. 3 is a good summary of the variation over a range of temperatures that such a surface layer can take on – at the free surface, with respect to theory, measurements, and modeling. That said the pore surface is something different, beginning as a liquid/solid(pore material) interface, and when ice forms any 'quasi-liquid' layer would be a function of the specific intermolecular interactions of that system. It might be more simple to state that the expected thickness of such a layer at such temperatures would likely be of order 1 molecular layer (≈ 2.5A for water).

Indeed, the width of the quasi-liquid layer on flat surfaces varies with temperature and with surface functional groups, and the same is the case for the quasi-liquid layer within pores. Yet, the exact width of the quasi-liquid layer is uncertain and a parameterization as a function of temperature out of scope. Schreiber et al. (2001) determined a value of t = 0.38 nm by fitting measured melting point depressions to a modified Gibbs-Thomson equation. Since the temperature range (223 - 263 K) and pore diameters (3 – 12 nm) investigated in Schreiber et al, (2001) are the same as in this study, t = 0.38 nm seems appropriate for the experiments carried out in this study. Furthermore, the assumption of t = 0.38 nm leads to a good agreement with our own results as can be seen from the DSC measurements and with melting point depressions compiled from literature in Marcolli (2014). We have now added: *"The width of the quasi-liquid layer has been shown to depend on temperature and surface chemistry but the exact thickness of the layer varies greatly between different observational techniques and MD studies (Bartels-Rausch et al., 2014). Nevertheless, the thickness of the quasi-liquid layer can be parameterized by fitting the measured melting point depressions of ice in pores to a modified version of the Gibbs-Thomson equation and has been shown to* *vary between 1 and 2 monolayers thick for the pore diameters and across the temperature range investigated in this study* (Findenegg et al., 2008; Jähnert et al., 2008; Marcolli, 2014; Schreiber et al., 2001; Wang et al., 2019)*. When accounting for the quasi-liquid layer thickness, assumed as t = 0.38 nm (Schreiber et al., 2001), the diameter of a pore capable of hosting ice (D_p) can be expressed as:…*" to the discussion on page 3 lines 1-9.

As an additional comment related to this and Eq. (3), is that any 'quasi-liquid' layer thickness t would be an equilibrium phenomenon, and it is not self-evident that it should be considered as a limit to the volume available for nucleation, which is fundamentally non-equilibrium. In fact the heterogeneity at the pore surface might help to initiate nucleation even if the relaxed, low-energy state of the system would prefer disorder.

Remember taken from the point of view of pore ice the layer closest to the pore wall may be disordered or 'quasi-liquid' but taken from the viewpoint of pore water the surface layer may exhibit enhanced order. That might stimulate nucleation in analogy to what is noted (22, 25-27) in the 9.0M2 particles which the authors say contain ice nucleation sites even at higher temperatures.

The water in the quasi-liquid layer is not available for incorporation into the critical ice embryo as it is bound to the pore surface (i.e. due to the interaction with the pore surface, it is at a lower chemical potential than the bulk of the pore water). Since the interaction with the pore wall depends on the functionalization of the pore surface, the thickness and the structure of the quasi-liquid layer indeed depends on the pore surface properties and it also may be structured in a way suitable for ice formation, i.e. it may act as an ice-nucleating surface inducing heterogeneous ice nucleation. Within the framework of CNT, this is described by amending the Gibbs free energy barrier of nucleation with a term that depends on the contact angle between the ice embryo and the pore surface, implying a spherical-cap-shaped embryo instead of a sphere. However, heterogeneous ice nucleation would still be limited by the width of the pore and therefore, the pore diameter would still need to be wide enough for bulk ice to fit inside the pore. For the pore diameters where heterogeneous freezing can occur without being impeded by the critical pore diameters (9.1H2 and 9.0M2) the ordering/disordering of the quasi-liquid layer may be responsible for the observed freezing. We discuss this on page 25 starting on line 10.

- **(3, 5-10) I find the discussion of water saturation in and out of the pore confusing, also see my comment on Eq. 2 above. My basic understanding is that within the pore everything is occurring in condensed liquid?**

We have reformulated the paragraph for clarity and it now reads: "*Based on CNT, the ice growing out of the pore needs to be supercritical with respect to the vapour phase. The energy barrier for nucleation in the vapour phase is significantly higher than that in water. This increase in energy barrier comes from the need to replace σ_{iw} with the interfacial energy between ice and vapour (σ_{iv}) in Eq. 2, which is approximately a factor of 4.8 larger than σ_{iw} at 236 K (Cooper, 1974; Ickes et al., 2015; Ketcham and Hobbs, 1969). Additionally, as the ice growing out of the pore experiences an environment that is subsaturated with respect to water, $\frac{p_w}{p_i}$ in Eq. 2 must be replaced by the ice saturation ratio (S_i), which is smaller than $\frac{p_w}{p_i}$. Therefore, the critical radius for ice growth out of the pore is much larger than that of the critical radius in the pore necessitating a substantial increase in the ice saturation ratio (S_i) for ice to be able to grow out of a pore…*" on page 3 lines 12-19.

- **(4,17) For the uninitiated a "Teflon bomb" is unfamiliar terminology.**

Thank you for pointing this out. We have now revised the terminology to state: "*a Teflon lined acid digestion vessel*" on page 4, lines 14-15

• (4,25) Here and throughout the manuscript the authors should be careful with how they refer to contact angle. "these were observed to change contact angle with ageing in air" (Note aging misspelled).

Thank you for pointing this out. We have now reformulated the sentence to state: "*We will focus on ice nucleation experiments with particles functionalized with trimethyl and hydroxyl groups rather than just calcined ones, as their contact angle with water was observed to change with aging in air (Muster et al., 2001).*" on page 5 lines 6-8.

I believe they intend to say that the water contact angle with these was observed to change after aging in air? In any case the authors should review all uses of contact angle, or use a defined 'water contact angle' throughout, to make sure the use of contact angle is correct and consistent throughout the text.

We have now revised the manuscript to clarify that contact angle refers to the water contact angle.

As a follow-up question, was the changing water contact angle with calcinated particles systematic with aging as a function of any monitored variable, like RH? If so these types of particles might offer another useful experimental system.

Unfortunately, the storage humidity was not monitored over the period during which we performed experiments with calcined particles. Therefore, it is not possible to quantify the time or conditions that led to observed changes in the water contact angle of the particles. As such, those experiments are not included in the present manuscript. Nevertheless, this may be a valid approach to investigate the impact of water contact angles on the PCF mechanism for future studies.

• (5,2) "filtered off" What is meant here?

We have clarified the text to read: "… *before the suspension was filtered and washed*…" on page 5 line 16

• (5,12) I think either BET should not appear as an acronym at first use, or perhaps it is enough to make it slightly more descriptive, for example: applying BET adsorption theory

Thanks, we have now changed the text to read: "… *and applying the Brunauer, Emmett and Teller (BET) gas adsorption theory*…" on page 6 lines 7-8

• (6,14) p/p0 could be better explained. It appears related to a comment below (13, 6), but the connection could be made more clear.

To connect p/p_0 to RH_w we have changed the text to read: "…p/p_0 *is the water saturation ratio or* $RH_w/100$." On page 7 line 2. Additionally, we have clarified the p/p_0 value used when calculating the water contact angle of the pores by stating: "*When deriving* θ, p/p_0 *is identified as the saturation ratio where the pore condensation step of the DVS measurement is the steepest*." on page 7 line 10-11.

• (7,13) Were any other sized particles bigger or smaller than 400 nm tested? Why or why not? It would be nice to rule out any effect of particle size when considering the results.

The particles were synthesized to be 400 nm in order for the observed differences in ice nucleation ability to be directly attributable to differences in pore diameters and water contact angles of the particles rather than the particle size. I. e. it was a means to eliminate one variable. The effect of particle size might be an additional parameter to be tested in future studies.

• (7,27) Contact angle again. Here it is not the pores contact angle, but I think water's contact angle with or within the pores.

In accordance with the previous comment on contact angle, we have now updated the manuscript to clearly indicate that the water contact angle with the pore or particle surface is meant.

• (13,6) See also comment above with regard to how the RH of the first sorption cycle is used. As a general comment, I would expect the hysteresis between adsorption and desorption should offer an additional verification of the pore size that is presented earlier in §3.1. It seems that the magnitude of the hysteresis, if the RH ramping is done is a quasi-steady state manner, should be directly related to the stability of the liquid in the pores. Was an effort made to use the information in this manner, or do I miss a complicating factor? Finally, have the author's considered how to propagate the uncertainty to bound the uncertainty in contact angle as derived from use with Eq. 7?

The observed hysteresis between adsorption and desorption isotherms can indeed be used to infer the pore shape and pore size as long as the pores maintain a constant water contact angle. However, the change in adsorption and desorption isotherms in the subsequent cycles indicates that the water contact angle of the pores changes with initial exposure to increasing humidity. We therefore use nitrogen adsorption to determine the pore diameters and the initial water vapour sorption cycle to infer contact angles using the pore diameters determined from nitrogen adsorption. The water uptake during the first water vapour sorption cycle is most representative of the contact angle relevant for water uptake in our ice nucleation experiments as the experiments are the first instance that the pores are exposed to high concentrations of water vapor.

• (Figure 4) The upper axes are missing a unit label. I am impressed by the agreement between the observed heat flow and the predicted critical pore diameter from the bulk physical model.

Thank you, we have now added "*nm*" to the upper axis of this figure. Indeed, it shows that the values used to constrain the interfacial tension from Murray et al., (2010) and Zobrist et al., (2007) are working well. Additionally, it corroborates that assuming a quasi-liquid layer of 0.38 nm is appropriate.

• (16,6) Why is AF 0.05 chosen? Is this simply an experimental choice of the minimum AF at which nucleation can be observed? A clear explanation would be useful, especially given that in many nucleation studies of controlled materials different AF may be chosen for plotting.

Thank you for pointing this out. The *AF* of 0.05 was chosen due to the distribution in pore sizes and functional groups on the particles. Using a lower AF to represent the average freezing RH of the porous particles, may lead to the misinterpretation of the freezing results due to the presence of unique particle features that may exist on a few of the particles. We have now added the following to clarify this choice in the main text: "*An AF of 0.05 was chosen as best representing the average freezing RH of the porous particles*." starting on page 17 line 10.

• (16, 23) Although the Skrotzki paper is directed to cirrus clouds, many such uptake measurements have been undertaken over the years and are notoriously difficult to parse. Furthermore the values in the literature vary over orders of magnitude. Direct studies of molecular uptake are presented in Kong et al. 4 as well as a review and comparison with measurement and simulation studies (including the Skrotzki paper). However, perhaps for these studies it must also be considered that the changes in functional groups that are utilized also likely lead to changes in uptake coefficient. This is clear from the adosorption/desorption isotherms in Fig. 3. It has also been previously documented that even thin surface coatings can significantly affect uptake.5,6 I would recommend some reference to this body of work be included.

Since pores are closely spaced in our particles, pore openings make up a relevant fraction of the particle surface. We therefore assume that ice grows on the ice-covered part of the particle surface, for which the accommodation coefficient on ice is relevant. Molecular dynamic simulations presented in David et al. (2019), which show that ice rapidly covers the entire particle surface, further support this assumption.

We revised the discussion of the delayed freezing onset RH following a comment of reviewer #1, who pointed out that an accommodation coefficient of 0.1 is at the lower limit of experimentally determined values. We now widened up the discussion on page 18 lines 20-29 to include all potential reasons.

• (Fig. 7 caption) As in Fig. 6 caption it should be stated that points correspond to AF 0.05 condition.

Thank you for pointing this out, we have now updated the caption accordingly.

• (Fig. 8) This figure seems a bit out of place and is of limited use in the explanation here. Perhaps it could be introduced earlier in the particle characterization section and returned to here?

We have now moved to the beginning of the manuscript (new Fig.1) and introduce it on page 4 lines 6 and 20-21 by stating: "*The MCM-41 (see Fig. 1a and b) particles were synthesized following Beck et al., (1992),…" and "To obtain larger pore diameters (~9 nm), SBA-15 particles (see Fig. 1c and d) were synthesized similarly to Linton et al., (2009b) where Pluronic® P104 (1.25 g, BASF) was*…".

• (25, 26) It might be a bit strong to say that parameterizations should be based on PCF. Perhaps, should include?

We have reformulated the sentence to state: "… *parametrizations should include the PCF mechanism below the HFT*…" on page 27 lines 5-6.

• (25, 26) The final sentence links this work to understanding anthropogenic emissions, but this is really the first mention of such emissions up to this point. Are these particles particularly analogous to any known anthropogenic emission? If the link is not strong I think this point can be left out, there are of course many reasons to better understand the effects of porosity and geometry on freezing.

We have now added reference to soot particles as an example of anthropogenic emissions, which have recently been shown to nucleate ice following PCF to text as follows: *"…, such as soot, which has been shown to nucleate ice in accordance with PCF (Mahrt et al., 2018, 2020b, 2020a; Nichman et al., 2019)*,…" on page 25 lines 13-15.

Summary: I have enjoyed reading the submitted manuscript. I reiterate that I think it could benefit from an improved clarity with regard to the concrete conclusions the authors would like to posit. My understanding is that basic edifice of PCF which rests on the inverse Kelvin equation does a good job of predicting the experimental observations if some of the asserted assumptions are valid. It appears that as with many systems a complete understanding of the data would involve a much more comprehensive picture of the intermolecular interactions specific to each system. The measurements rely both on ice nucleation and crystal growth to a detectible size, thus many details related to both the ice initiation and macroscopic state are convoluted. Such complexities are intrinsic in many experiments, yet I still feel this study brings the community a step forward. However, it appears the open question remains as to how this might be utilized and tested in a messier real atmospheric aerosol scenario.

We would like to thank the reviewer for commending the efforts made in this study to disentangle the complex relationship between pore size and ability of pores to uptake water within the PCF framework. Indeed, it is our intention that this study lays the foundation for future studies to further quantify the PCF mechanism for its application in real atmosphere particles thus improving the representation in cloud models via ice nucleation parametrizations.

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