

Bidirectional reflectance of laboratory cometary analogues to interpret the spectrophotometric properties of the nucleus of comet 67P/Churyumov-Gerasimenko

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Abstract

In this work we provide measurements of the bidirectional reflectance in the VIS-NIR spectral range for a selection of laboratory samples to deduce parameters such as their visible spectrum, phase curve, hemispheric albedo and phase reddening. The aim is to simulate the reflectance of the comet nucleus in order to aid the interpretation of data from the surface of comet 67P/Churyumov-Gerasimenko (67P) acquired by the Rosetta OSIRIS imaging system. We produced a broad variety of well characterised and reproducible samples containing water ice, carbonaceous compounds and complex organic molecules. We were able to reproduce the individual global spectrophotometric parameters (albedo, spectrum, phase reddening, phase curve) of 67P by using mixtures of activated charcoal, tholins and water ice. However, no mixture was able to fit all parameters simultaneously. The samples with highest porosities best match the phase curve of 67P. Spectral considerations from our experiments show that the parti-

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cle size of the darkening agent on the cometary surface should have a lower limit of a few hundred nanometres to several micrometres and cannot be dominated by particles in the range of some tens of nanometres. Furthermore, our findings indicate that the bright ice patches observed by OSIRIS and VIRTIS should be relatively dust free at small scale.

Keywords: Photometry, water ice, organics, comets

1. Introduction

After its ten-year interplanetary journey, ESAs Rosetta spacecraft arrived at comet 67P/Churyumov-Gerasimenko (67P) in August 2014. Since then the nucleus has been scrutinised and mapped regularly by the Optical, Spectroscopic,
5 and Infrared Remote Imaging System (OSIRIS) (Keller et al., 2007) consisting of a Narrow Angle Camera (NAC) and a Wide Angle Camera (WAC). First imaging and spectroscopy results from the OSIRIS and VIRTIS¹ instruments showed unexpected surface morphology and diversity (Sierks et al., 2015; Thomas et al., 2015; Capaccioni et al., 2015).

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A detailed analysis of OSIRIS data of the spectrophotometric properties of 67Ps nucleus was carried out by Fornasier et al. (2015), finding a strong opposition peak on the phase function, a red spectral behaviour in the UV to NIR spectral range without absorption bands and a strong phase reddening. According to the spectral slopes they define three groups (blue, moderately red,
15 and red) of terrains across the nucleus. The blue terrains mainly lie in the neck region, while the two lobes are more red.

To link these spectrophotometric properties to physical surface characteristics such as chemical composition, grain sizes, porosity and texture, a profound
20 knowledge of analogues is vital. Since the resolution of OSIRIS is limited to the

¹Visible and Infrared Thermal Imaging Spectrometer, Coradini et al., 2007

centimetres to metres range, depending on the spacecraft-nucleus distance, it will never be possible to draw any direct conclusion about surface properties on the millimetre- or submillimetre- scale from this dataset. Other instruments on Rosetta and Philae can however provide information on the physical and chemical properties of the surface at smaller scales, either directly (CIVA and ROLIS images at resolutions down to 0.8 mm/px; [Bibring et al., 2007](#); [Mottola et al., 2007](#); [Schröder et al., 2017](#)) or indirectly, from the inversion of remote-sensing data (MIRO, [Gulkis et al., 2007](#); VIRTIS, [Coradini et al., 2007](#)).

A common finding of Rosetta is the absence of extended surface regions of exposed water ice, in general agreement with previous observations of cometary nuclei such as 9P/Tempel 1 ([Sunshine et al., 2006](#)) or 103P/Hartley 2 ([A'Hearn et al., 2011](#); [Protopapa et al., 2014](#)). A notable exception are two bright 1500 square metre wide patches observed in the Anhur/Bes regions ([Fornasier et al., 2016](#)). The global surface is mainly covered by carbon-bearing, dehydrated, refractory and organic-rich material ([Capaccioni et al., 2015](#)). Nevertheless OSIRIS's high-resolution images revealed over one hundred metre-sized bright spots dispersed over the comets surface, mainly in low insolation areas, that were hypothesized to be ice-rich features ([Pommerol et al., 2015b](#); [Deshapriya et al., 2016](#)). The presence of water ice on two large debris falls in the Imhotep region has indeed been confirmed by the Visible Infrared and Thermal Imaging Spectrometer (VIRTIS), with a spatial resolution of 15 – 30 m ([Filacchione et al., 2016](#)). Spectral fitting and inversion indicates crystalline pure water ice grains with a bimodal grain size distribution with peaks at 33 – 72 μm and 1.4 – 2.6 mm. The occurrence of millimetre-sized particles can be explained by growth by vapour diffusion in ice-rich layers or by sintering. This particle size is significantly larger than the 1 μm grains (and agglomerates of them) that were reported in the coma of comet 103P/ Hartley 2 ([Sunshine et al., 2011](#)). Further comparisons of the OSIRIS and VIRTIS datasets have led to the conclusion that most of the bright features observed by OSIRIS were indeed exposures of dirty water ice ([Barucci et al., 2016](#)) of indeterminate the grain size.

In addition to chemical composition the morphological properties of ejected
 55 non-volatile grains were analysed by the optical microscope of the Cometary
 Secondary Ion Mass Analyzer (COSIMA, [Kissel et al., 2007](#)) with a spatial
 resolution of 14 μm . Larger grains exceeding 50 μm are fluffy and most likely
 agglomerates ([Schulz et al., 2015](#)). [Hilchenbach et al. \(2016\)](#) report on particle
 sizes ranging from 15 μm to 225 μm with a differential size distribution fitted
 60 with a power law exponent of -3.1, meaning a large number of small particles.

Measurements by the Grain Impact Analyzer and Dust Accumulator (GI-
 ADA, [Colangeli et al., 2007](#)) revealed porous aggregates (0.2 to 2.5 mm) of
 submicron particles ([Della Corte et al., 2015](#)). [Fulle et al. \(2015\)](#) report on a
 65 second family of compact particles in the size range of 0.03 to 1 mm. Neverthe-
 less post-landing images at highest resolution taken by ROLIS did not reveal
 any particles ([Schröder et al., 2017](#)), suggesting a surface consisting of small par-
 ticles without agglomerates formation at this site. Preliminary analyses of the
 Micro-Imaging Dust Analysis System (MIDAS, [Riedler et al., 2007](#)) reveal only
 70 very few small (sub-micron) particles, but several particles much larger than
 expected ([Bentley et al., 2015](#)). It is not clear whether these results represent
 the dust composition on the surface of the comet or if they are biased in terms of
 chemical composition, size and shapes as all data is collected in the coma of 67P.

75 To better understand and interpret the observational data and findings it
 is crucial to comprehend the physical processes involved. A useful approach
 is to conduct representative laboratory simulations in order to quantify these
 processes and provide a starting point for numerical models. Until now only
 a few laboratory studies investigated the optical properties and surface evolu-
 80 tion of cometary analog materials using ice/non-volatile mixtures. The most
 extensive and comprehensive campaign was the KOSI (KOMETEN-SIMULATIONS)
 experiments performed at the DLR space simulator in Köln from 1987 to 1993
 ([Gruen et al., 1991](#); [Gruen et al., 1993](#); [Seiferlin et al., 1995](#)). A brief review can

be found in [Sears et al. \(1999\)](#). These experiments focuses on the surface evo-
85 lution and sublimation processes under simulated space conditions pursuant to
findings of ESA’s Giotto mission to comet 1P/Halley. Although 11 experimental
runs were performed, there was no systematic approach for mixing constituents.
For the KOSI 3, 4 and 6 experiments, [Oehler and Neukum \(1991\)](#) measured the
reflectance spectra at two wavelengths in the visible and in the NIR (0.36 μm
90 and 2.5 μm) before and after the sublimation experiments under three different
illumination angles.

[Stephens and Gustafson \(1991\)](#) measured the bidirectional reflectance of
ice/carbon, ice/silica and ice/carbon/silica mixtures from the UV to NIR spec-
95 tral range. But only a single mixing ratio was analysed and the main focus lay
on the formation of a surface mantle. The formation and evolution of such man-
tles from icy samples containing minerals and organic compound was recently
investigated by [Poch et al. \(2016a\)](#) and [Poch et al. \(2016b\)](#). It was found that
that different mixing types, coatings or inclusions, influence the spectral and
100 morphological evolution of the samples.

The major limitations of previous optical experimental studies to simulate
cometary nuclei are the spectral coverage and the sample reproducibility. In the
case of [Stephens and Gustafson \(1991\)](#) and [Oehler and Neukum \(1991\)](#) only two
105 wavelengths were analysed. In this work we will show an extensive spectropho-
tometric study covering the whole VIS-NIR spectral range using six different
filter bands to be compared with OSIRIS data. We provide a systematic ap-
proach in sample composition as well as in particle sizes, using standardised
protocols to ensure reproducibility.

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The aim of this study is to see how closely we can match the spectrophoto-
metric properties of 67P described by [Fornasier et al. \(2015\)](#) by combining the
three types of materials: carbonaceous compounds (darkening agent), complex
organics (spectral reddening agent) and water ice (spectrally neutral agent),

115 thought to be major constituents of the cometary surface. For each category
of material we have selected particularly well characterised samples and mixed
them in systematic combinations. The samples were produced with a standard-
ised protocol to reach maximal control on the mixing ratio and the reproducibil-
ity. The systematics of such a study are useful for understanding the contribu-
120 tions of individual properties to the global spectrophotometric behaviour.

For all samples the bidirectional reflectance in the VIS-NIR spectral range
was measured using the PHIRE-2 goniometer. From these measurements albe-
dos and phase reddening were derived. Complementary results focusing on the
125 surface evolution processes under simulated space conditions will be reported in
a follow-up publication.

2. Methods

2.1. Instrument

All measurements presented in this work were acquired using the PHIRE-2
130 radio-goniometer. Details about the instrument and its calibration can be found
in [Pommerol et al. \(2011\)](#) and [Jost et al. \(2016\)](#). Only the relevant specifica-
tions are summarised here. The goniometer is located inside a freezer, operated
at $-35\text{ }^{\circ}\text{C}$ for ice-bearing samples. Ice-free samples were measured at room
temperature. A collimated light source is equipped with six bandpass filters
135 (centred at 450/550/650/750/905/1064 nm) and illuminates the samples with
an incidence angle in the range of 0° to 60° . The scattered light is measured
with a silicon photovoltaic sensor on another mobile arm, reaching from -80°
to $+80^{\circ}$ emission angle. The angle between incidence and emission directions
(constant azimuth) is called the phase angle α .

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The use of bandpass filters precludes highly resolved spectral data. But since
cometary nuclei display featureless spectra in the visible spectral range, without
any sharp absorption features, six values in the VIS-NIR range are enough to

PHIRE-2		OSIRIS		
Wavelength	Bandwidth	NAC Filter	Wavelength	Bandwidth
(nm)	(nm)		(nm)	(nm)
450±10	70±30	F84	480.7	74.9
550±10	70±30	F83	535.7	62.4
650±10	70±30	F82	649.2	84.5
750±10	70±30	F88	743.7	64.1
905±3.5	25±3.5	F41	882.1	65.9
1064±3.5	25±3.5	F71	989.3	38.2

Table 1: Table listing the PHIRE-2 filters and the corresponding OSIRIS NAC filters with the associated central wavelength and bandwidth.

represent an overall trend in terms of magnitude and slope.

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Although the filters used on board OSIRIS are not identical to the ones used in our laboratory study (see [Table 1](#)), the results should be qualitatively comparable and we note that our filters have similar or lower bandwidths than the OSIRIS filters.

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PHIRE-2 can be equipped with either a mirror head or a beam splitting system ([Jost et al., 2016](#)). The advantage of the beamsplitter is the possibility of measuring at very low phase angles (down to 0°) whereas the use of the mirror head results in vignetting of the incident beam for phase angles lower than 4°, preventing any measurement in this configuration. On the other hand, the use of the beamsplitter reduces the signal by a factor 4. Although this is not a problem for bright samples such as those studied by [Jost et al. \(2016\)](#), this causes issues in the case of very dark samples as used in this study. Hence the majority of samples in this study was acquired using the mirror setup.

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160 2.2. Physical units

2.2.1. Reflectance factor

Throughout this manuscript, the measured light is expressed in terms of reflectance factor (REFF) defined as the ratio between the brightness of the sample measured in a particular geometry and the one of a hypothetical Lambertian diffuser (a perfectly diffuse surface which scatters isotropically) observed
165 in the same geometry. The reflectance factor REFF (Hapke, 1993) is related to radiance I [$\text{W m}^{-2} \text{sr}^{-1}$] (scattered light) and irradiance F [W/m^2] (incoming light) as $REFF = \pi * I / (F * \cos(i))$, where i is the incidence angle of the light source.

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2.2.2. Hemispherical albedo (Directional-hemispherical reflectance)

The integrated hemispherical albedo A_H is an import photometric property as it is controlling the energetic budget and therefore the surface temperature. We have thus derived values of A_H by integrating our phase curves over a range of emission angles from 0° to 90° for a vertical illumination. The term $\cos(e) \cdot \sin(e)$ has the effect that missing values at opposition and at 90° emission angle can be neglected. In case of 2D data (principal plane only) and assuming azimuthal symmetry the hemispherical albedo calculates as follows (see Eq. 10.18 in Hapke (1993)):

$$A_H = 2 \cdot \int_0^{\pi/2} REFF(i, e, a,) \cdot \cos(e) \cdot \sin(e) de \quad (1)$$

2.3. Phase reddening

Many solar system objects display surface colours that are known to be dependent on the observation geometry. Often, an increase of the spectral
175 slope with increasing solar phase angle is observed in the visible wavelength range. This phenomenon is often referred to as phase reddening. The opposite behaviour, a decrease of the spectral slope with phase angle, is called phase bluing. Reddening is widely observed in our solar system such as e.g. on the

Moon (Gehrels et al., 1964; McCord, 1969) on asteroids (Taylor et al., 1971;
 180 Clark et al., 2002; Abe et al., 2006; Magrin et al., 2012) or on comets (Fornasier
 et al., 2015). Bluing is rarely observed (Rosenbush et al., 2009; Lantz et al.,
 2015). Schröder et al. (2014) investigated laboratory observations of granular
 surfaces prepared from different rock types and attempted to reproduce the re-
 sults by simulation. They showed that microscopic roughness highly influences
 185 the spectral slope and its dependence on illumination/measurement geometry.

To be consistent when comparing our results with those of Fornasier et al.
 (2015); Fornasier et al. (2016), we express the spectral slope in units of % per
 100 nm using the same formula (Eq. (2)). Positive values indicate red slopes
 190 and negative values blue slopes. At a given phase angle,

$$S[\%/100nm] = \frac{(R_{905} - R_{550})}{R_{550} \times (905nm - 550nm)} \times 10^4 \quad (2)$$

, when the slope S increases as a function of phase angle we call it phase
 reddening. The 355 nm wide wavelength range over which the spectral slope is
 calculated is very similar to the one of 347 nm from Fornasier et al. (2015).

195 2.4. Samples

2.4.1. Ice particle production

For this study we used two types of spherical water ice particles. The
 “coarse” type has a mean diameter of 67 μm with a standard deviation of 31 μm
 (as measured by a cryo-SEM), the “fine” type is about ten times smaller with a
 200 diameter of $4.54 \pm 2.54 \mu\text{m}$. For additional details about the production setups
 and procedures, see Yoldi et al. (2015) and Jost et al. (2016). An overview of
 all sample materials used in this study is provided in Table 2.

2.4.2. Tholins

We used tholins as analogues of the complex organic matter, because they exhibit a strong red spectral slope from 0.2 to 1.0 μm , allowing us to vary the redness and albedo of our samples by mixing it with darker and spectrally different carbonaceous compounds. The word “tholins” was first used by [Sagan and Khare \(1979\)](#) to describe the complex macromolecular organics formed after the irradiation of a gas mixture composed of simple molecules, displaying a orange-brownish colour. The yellowish to dark organic residues obtained after the irradiation of simple ices have also been named tholins or “ice tholins” (see e.g. [McDonald et al., 1996](#)). The tholins used in this work were previously utilised by [Poch et al. \(2016a\)](#) and [Poch et al. \(2016b\)](#). The material was produced in a plasma of 95 % N_2 and 5 % CH_4 , using the PAM-PRE setup ([Sciamma-O’Brien et al., 2010](#); [Szopa et al., 2006](#)). Tholins are made of a complex mixture of molecular and macromolecular material composed of aliphatic hydrocarbons, nitriles, imines, amines or N-bearing aromatic compounds ([Bernard et al., 2006](#); [Carrasco et al., 2009](#); [Pernot et al., 2010](#); [Quirico et al., 2008](#); [Gautier et al., 2014](#); [Gautier et al., 2016](#)). The complex organic molecules are arranged in high porosity spherical grains with a mean diameter of 315 ± 185 nm ([Carrasco et al., 2009](#)). Their optical indices have been characterised by [Mahjoub et al. \(2012\)](#) and the reflectance spectra by [de Bergh et al. \(2008\)](#).

2.4.3. Carbon powders

We used carbon powders as analogues of the darkening agent(s) of cometary surfaces. Amorphous carbon inherited from interstellar dust could represent up to 10 % of cometary materials ([Wooden, 2008](#)) and could partly contribute to the low albedo of cometary nuclei. However such a high amount of pure carbon in cometary refractory material is questionable, since by date only complex organic materials rather than pure carbons have been found in natural extraterrestrial samples like porous interplanetary dust particles (IDP) or STARDUST samples (e.g. [Sandford et al., 2006](#); [Brownlee, 2014](#)).

Carbon black

The carbon black used in this work was purchased from Alfa-Aesar (product
235 3972). It is the solid residue obtained by acetylene combustion and consists of
amorphous carbon shaped as spherical particles with an average diameter of
42 nm. The structure of the individual particles cannot be resolved in Fig. 1a.

Activated charcoal

The activated charcoal used in this study was purchased from Sigma-Aldrich
240 (Product C3345). This granular carbon has been prepared from wood particles
which have been impregnated with an unspecified chemical agent and pyrolysed
to produce very porous particles of pure carbon. The particles are smaller
than 75 μm according to the manufacturer. Our own microscope image analysis
resulted in a mean diameter of 17.6 μm with a standard deviation of 11.3 μm .
245 The particle shape is irregular with sharp edges (see Fig. 1b). There is very
little formation of agglomerates.

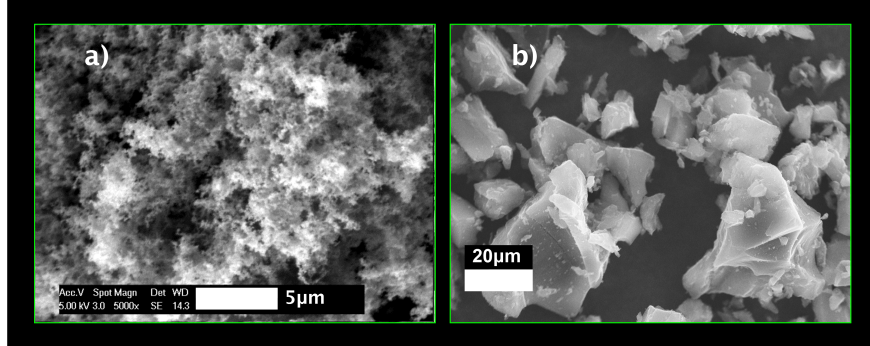


Figure 1: Environmental scanning electron microscope images of carbon black (a) and activated charcoal (b) acquired at Institute of Geography at University of Bern.

2.5. Mixing types

We developed two different mixing procedures for producing two types of laboratory analogues:

	Particle size	Shape	Bulk density	Porosity
Fine grained ice	$4.54 \pm 2.54 \text{ }\mu\text{m}$	spherical	$0.11 - 0.19 \text{ g/cm}^3$	$79 - 88 \%$
Coarse grained ice	$67 \pm 31 \text{ }\mu\text{m}$	spherical	$0.4 - 0.5 \text{ g/cm}^3$	$46 - 56 \%$
Tholins*	$315 \pm 185 \text{ nm}$	spherical	$0.077 \pm 0.008 \text{ g/cm}^3$	$94.6 \pm 0.6 \%$
Carbon black	42 nm	spherical	$0.08 - 0.12 \text{ g/cm}^3$	$93 - 95 \%$
Activated charcoal	$17.6 \pm 11.3 \text{ }\mu\text{m}$	spherical	0.31 g/cm^3	81%

* [Brouet et al. \(2016\)](#)

Table 2: Sample material characteristics

1. Intra-mixture: tholins and carbon particles are mixed in water prior to the freezing process. The non-volatiles are present as inclusions within the icy grains. This mixture type is only applicable for the coarse ice grains. The suspension is prepared by ultrasonication, performed with a Hielscher 200Ht ultrasonic unit equipped with a 7 mm diameter sonotrode. The duration of ultrasonication was limited to about 1 min to keep the suspension temperature below 30 °C, to avoid thermal changes.
2. Inter-mixture: non-volatiles are mixed intimately with the pure water ice particles at the grain level. This process works for both the coarse and the fine particles. The mixture was prepared in an aluminium bottle cooled to liquid nitrogen temperature, by adding freshly produced ice and precooled non-volatiles. The bottle was then agitated using a Vortex-Genie2 mixer at full speed for about 1 min, interrupted for re-cooling the bottle in liquid nitrogen.

The deposition of sample material into the sample holder was done by sieving (800 μm mesh size) to avoid big agglomerates and to produce homogeneous samples of controlled bulk density. There is no compression - the piled material is cut with a spatula and a thin layer of material is sieved on top to obtain a random surface orientation.

3. Laboratory results

270 In this section we will present experimental results of 3 kinds of mixtures:
1 - carbon-tholin mixtures; 2 - ice-tholin mixtures and 3 - carbon-ice mixtures.
The ternary case of ice-carbon-tholin mixtures will be treated in our follow up
work.

275 All datasets shown in this work are publicly available on the Data Analysis
Center for Exoplanets (DACE) platform ([https://dace.unige.ch/lossy/
samplesearch/index](https://dace.unige.ch/lossy/samplesearch/index)) where they can be freely visualized and downloaded.

3.1. Reflectance spectra and albedo

3.1.1. Dry dust mixtures

280 Despite the fact that carbon black and activated charcoal have the same
chemical composition and a similar amorphous structure, their spectral be-
haviour is different (Fig. 2a). Activated charcoal displays a red spectral slope
while carbon black is spectrally blue.

285 Even with a strong reddening agent, such as tholins, the spectrum remains
blue beyond 650 nm, because of the blue spectrum of the nanometre-size car-
bon black (see Fig. 2b). With such a mixture it is not possible to represent the
cometary spectrum, which monotonically increases in the 400 – 1100 nm range.
This could indicate a lower limit to the darkening agent particle size and would
290 exclude particles with a size of a few tens of nanometres, which is consistent
with compact particle sizes as observed by GIADA from Fulle et al. (2015) of
30 μm to 1 mm. The detection of “submicron particles” by Della Corte et al.
(2015) and Fulle et al. (2015) is quite vague in its definition and not specified
in detail.

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To reproduce the spectrum of 67P and specifically the red slope, we combined
different fractions of tholins from 10 to 50 wt. % with activated charcoal. We

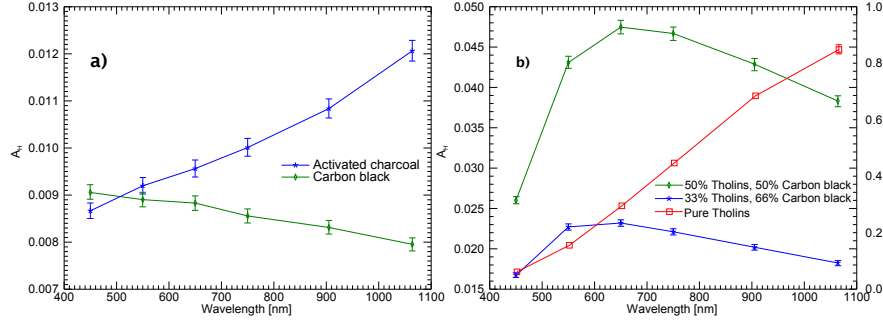


Figure 2: (a) Visible reflectance spectrum of carbon black and activated charcoal. The hemispherical albedo A_H is calculated for each filter band by integration of the phase curve at $i = 0^\circ$. While carbon black with particle sizes smaller than the wavelength has a blue spectral slope, the activated charcoal with particle size much larger than the observed wavelengths has a red slope. (b) Reflectance spectra of carbon black mixed with tholins. Blue, 1:1 mixing ratio, green, 2 wt. parts CB 1 part tholins. The spectrum of pure tholins is overlaid with the y-scale on the right side. The mixture of carbon black (blue slope) with tholins (red slope) results in arch-shaped spectral with a maximum at around 650 nm.

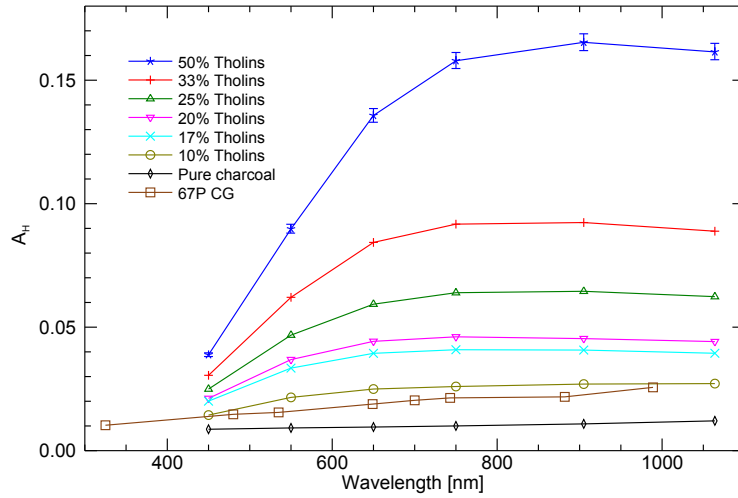


Figure 3: Visible reflectance spectra in units of hemispherical albedo A_H from charcoal mixed with tholins in different ratios. Both constituents have an intrinsic red spectral slope (see Fig. 1). The spectrum of 67P is overplotted. A mixture of charcoal with 10 wt. % of tholins has the highest coincidence with the spectrum of 67P, whereas the slope of the cometary curve is more monotonic increasing. Our mixtures are always concave shaped.

first analysed the mixture as a dry powder without any ice. The values of 67P
are calculated by using Hapke parameters fitted to disk-averaged reflectance
300 from Fornasier et al. (2015, Tab. 3). All spectra in Fig. 3 of the charcoal-
tholins mixtures show a prominent red slope in the 450 nm to 750 nm range,
but are almost flat or even slightly blue towards NIR (where tholins become
transparent). The best agreement with the cometary spectrum was reached
with a mixture of 90 % charcoal and 10 % tholins. This lab spectrum an the
305 one of the surface of the nucleus intersect at around 450 nm and 1000 nm, but
the lab spectrum is less monotonic and displays a concave curvature (being
brighter than the cometary surface in the entire 450 – 1000 nm range).

3.1.2. Ice-tholin mixtures

To study the influence of ice particle size and mixing type on the spectral
310 red slope, we mixed pure water ice with tholins. These mixtures are not repre-
sentative for comets in terms of brightness. In Fig. 4 as expected a higher tholin
content (1 % vs 0.1 %) decreases the albedo in the visible range, but has nearly
no effect at 1064 nm (and even already at 905 nm for fine-grained ice). The
different mixing type (inter-mixture vs. intra-mixture, see Section 2.5) plays al-
315 most no role and differences between the two 0.1 % Tholin samples (with coarse
ice grains) may be explained by the very sensitive preparation process, where
already minor losses in vessels and tubes can play a significant role. The spec-
tral variations caused by the difference in ice particle size are more prominent.
Small ice particles have a higher surface area per unit mass, therefore the rel-
320 ative area coated with tholin particles is smaller, which generally increases the
visible albedo. The spectral slopes for coarse and fine-grained ice mixed with
1 % tholins do not differ significantly, despite an offset in albedo of about 0.2 in
the VIS range. It is unexpected that at 450 nm and 1064 nm the albedo of pure
tholins cannot be distinguished from a mixture of coarse grained ice particles
325 and 1 % tholins.

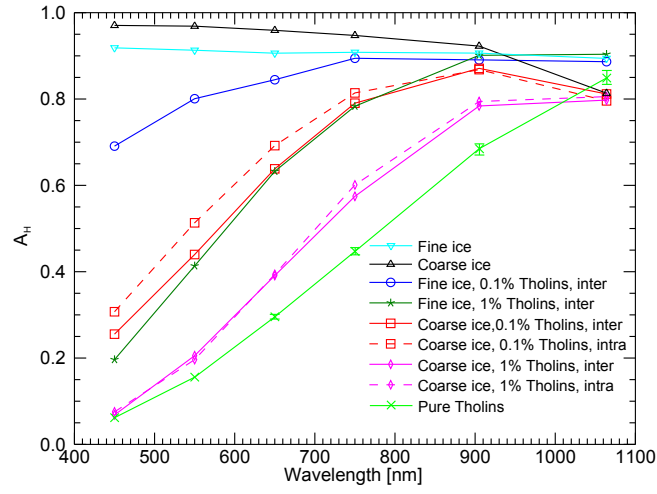


Figure 4: Hemispherical albedo of different ice-tholin mixtures as a function of wavelength. Two different mixing ratios (0.1 and 1 wt. % tholins) were measured. For the coarse grained ice both mixing types (inter + intra mixture; see [Section 2.5](#)) were applied. Curves of pure ice and pure tholins are plotted as reference. While at shorter wavelengths (< 750 nm) the tholin content is dominating the albedo, the influence vanishes completely at 1064 nm, where only the ice grain size play a role.

3.1.3. Ice-dust mixtures

To create a dirty-ice mixture with the same albedo as the surface of 67Ps nucleus at a certain wavelength, we analysed all possible combinations of different carbon contaminants and ice particle sizes at different mixing ratios. Their hemispherical albedos are plotted in Fig. 5 as a function of carbon content. The spectral reddening is neglected for this analysis and the wavelength of $\lambda = 750$ nm is selected as it lies in the middle of the analysed spectral range. As a general rule it can be stated that small ice particles need more darkening agent to become dark than coarse ice particles. Smaller carbon particles absorb more efficiently than coarse ones, because the absorbing surface per mass is bigger. In the case of coarse ice particles mixed with nanometre-sized carbon black, only 2 wt. % of contaminant is needed to reach an albedo as low as 67P. In the other extreme case (small ice particles, big charcoal grains) a 1:1 mixing ratio is needed to reach such a low albedo.

To explain the exponential decrease in brightness with contaminant level in Fig. 5, it has to be clarified that both of our mixing types (inter/intra) are intimate mixtures where the two species are mixed as homogeneously as possible at the grain size level. The opposite case is a areal mixture where large chunks of pure material are arranged on the surface (e.g. checkerboard-like). The main difference between these mixing types is the travel-path of a single photon. In an areal mixture each photon ideally “sees” only one species of material until it is scattered or absorbed. The resulting albedo of the total surface is therefore a linear combination of the individual constituents’ albedos and their surface covering fraction. A mixture of 50 % charcoal and 50 % ice would result in a hemispherical albedo of ~ 0.5 which is by magnitudes brighter than our laboratory samples. In an intimate mixture, in case of multiple scattering, each photon is very likely to be affected by both constituents within its mean free path length. This effect is most pronounced when the difference in single scattering albedo of the two materials in a binary mixture is large. This reduces

the brightness of an intimate mixture compared to a areal mixture of the same mass ratio. The surface of a cometary nucleus is more likely expected to be an intimate mixture as the grains are small and the primordial mixing took place by condensation and aggregation at small scale level (Greenberg, 1982).

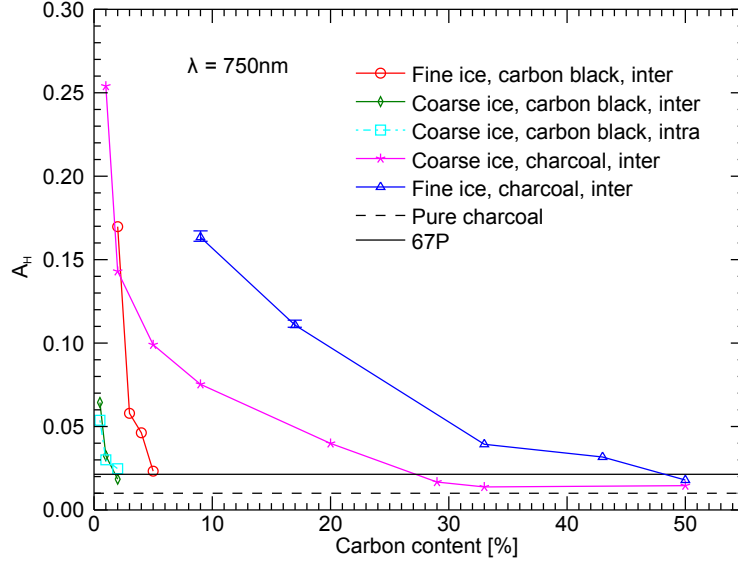


Figure 5: Hemispherical albedo of ice-carbon mixtures as function of carbon content all measured at $i = 0^\circ$ and $\lambda = 750$ nm. Albedos of pure charcoal and 67P are plotted as horizontal reference lines. All possible combinations of ice grains (fine/coarse) and carbon particles (charcoal/carbon black) are shown. Further the combination of coarse ice particles and carbon black was produced using both mixing types (inter/intra mixture), but resulted without noticeable difference in albedo. Generally spoken it can be stated that mixtures of fine grained ice are brighter than with coarse grains when using the same carbon content. Vice versa smaller carbon particles have a bigger darkening effect than bigger ones, when using the mixing ratios. Both findings can be explained by changing total surface area per mass.

360 3.2. Phase reddening

At larger heliocentric distances ($r_h = 3.6$ AU) in August 2014 the phase reddening effect of 67P was fitted as $0.104 \text{ } \%(100\text{nm})^{-1}\text{degree}^{-1}$ (Fornasier et al., 2015) having spectral slopes between 11 and 16 $\%/100\text{nm}$ at phase angles between 1.3° and 52° . During the period from April to August 2015

365 ($r_h = 1.88 - 1.26$ AU) the phase reddening effect was found to be significantly weaker ($0.041 \text{ \%}(100\text{nm})^{-1}\text{degree}^{-1}$; Fornasier et al., 2016) at phase angles between 60° and 90° . This indicates a change in physical properties of the uppermost surface layer (texture and/or composition) during this period.

370 Comparison of the spectral slopes of carbon-tholin-mixtures, as plotted in Fig. 6, reveal two main trends: whereas the mixtures of carbon-black with tholins show a blue spectral slope ($< 0\%/100 \text{ nm}$) at low to medium phase angles, they become slightly red at high phase angles ($> 80^\circ$). Mixtures of activated-charcoal with tholins are all red without exceptions, but get redder
375 towards high phase angles.

Varying the mixing ratios mainly results in vertical offsets, the steepness remaining quite constant. Further the steepness of mixtures with carbon black and charcoal seem almost identical. This might be explained by the low absolute spectral slope of carbon materials compared to tholins. The slope is mainly
380 defined by the tholins while the carbon compound/content defines the vertical offset.

A mixture of 33 % tholins and 67 % charcoal could well reproduce the linear
385 fit from Fornasier et al. (2015) with minor discrepancies at phase angles smaller than 20° . While the OSIRIS data indicates an almost linear relationship between phase angle and spectral slope, our measurements show a systematic deviation towards lower values at small phase angle ($\alpha < 15^\circ$). The curve acquired close to perihelion is still best represented by a mixture of 33 % tholins
390 and 67 % charcoal.

To analyse how the carbon content in ice mixtures influences the phase reddening, spectral slopes from mixtures of fine grained ice mixed with charcoal are plotted in Fig. 7a. All curves are fitted by a linear function which is overplot-
395 ted. Pure fine grained ice displays an almost flat spectrum in the $550 - 950 \text{ nm}$

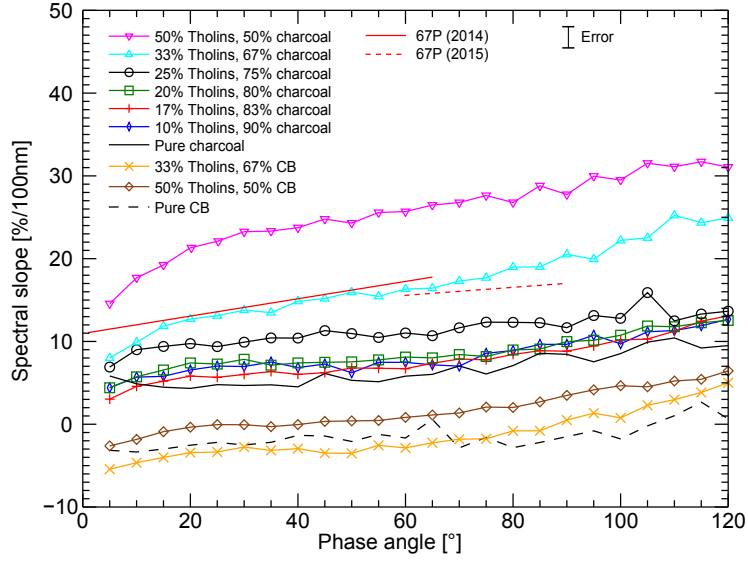


Figure 6: Spectral slopes between 550 nm and 905 nm in units of %/100 nm as a function of phase angle of different carbon–tholin mixtures. All samples were measured at $i = 60^\circ$ to achieve high phase angles. Negative values spectral slope mean a blue spectral slope (i.e. the material is brighter at 550 nm than at 905 nm). All samples show an increasing spectral slope with phase angle. However mixtures using carbon black (see corresponding spectra in Fig. 1) show a negative spectral slope at low phase angle but turn to positive values at high phase angles. Mixtures with the bigger grained charcoal are systematically red at all angular positions. The linear fit of 67P from Fornasier et al. (2015) is represented best by mixture of charcoal with 33 wt. % of tholins.

spectral range and shows nearly no increase of slope with phase angle. Mixtures with charcoal contents from 10 % to 43 % display a slightly blue spectrum (spectral slope < 0) at phase angles smaller than 20° , but turn to a red spectrum at larger angles. The 50 % mixture displays a flat spectrum at opposition, then reaches values of about 12 %/100 nm at 120° phase angle. Pure charcoal has a red spectrum at all angles and becomes redder towards high phase angles.

The slopes of the linear fits as a function of charcoal content are plotted in Fig. 7b. There seems to be a correlation between carbon-ice mixing ratio and increase of spectral slope, except for pure charcoal. This seems to be different for charcoal-tholin mixtures (Fig. 6), where the tholin content controls the vertical offset of the curves but not their steepness. This may be a physical effect in mixtures with an almost transparent constituent such as ice, which strongly influences the optical path length.

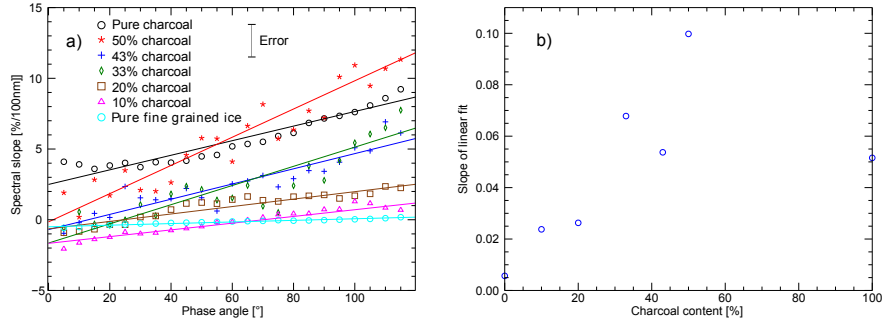


Figure 7: a) Spectral slopes between 550 nm and 905 nm in units of %/100 nm as a function of phase angle for fine grained ice mixed with charcoal. All samples were measured at $i = 60^\circ$. All curves are fitted by a linear function. Pure ice shows almost no phase reddening, while with higher carbon content the slopes are increasing. The data for pure charcoal do not fit the general trend, the spectrum is redder at low phase angles but the slope lower than for mixtures with 33/43/50 % charcoal. b) The slope of the linear fits as a function of charcoal content, the unit is percent per degree. All charcoal-ice mixtures seem to show a positive correlation of charcoal content and slope.

In Fig. 8 a variety of different ice-carbon mixtures are compared to the fitted phase curves from 67P at 743 nm (disk integrated) and 649 nm (disk resolved) (Fornasier et al., 2015; Tab 4). OSIRIS data were acquired at phase angles ranging between 1.3° and 54° . Therefore we only compare the curves for phase angles lower than 60° .

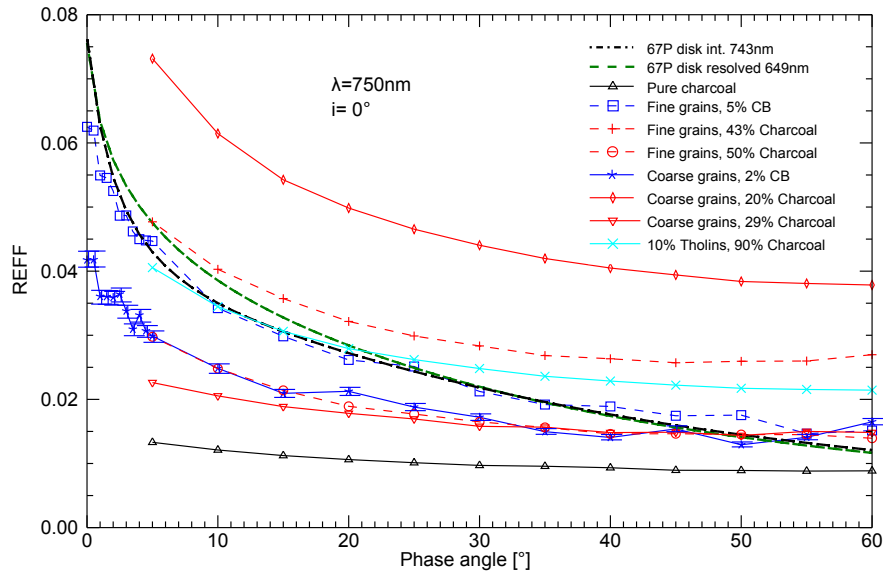


Figure 8: Phase curve of different ice-carbon mixtures acquired at $i = 0^\circ$ and $\lambda = 750$ nm. The curves of fine grained ice with 5 % carbon black and coarse grained ice with 2 % carbon black are measured with the beamsplitter setup (see Section 2.1), the rest with the mirror setup (having a minimal phase angle of 5°). The phase curves of 67P are reproduced from Hapke parameters provided in Fornasier et al. (2015). A mixture fine grained ice mixed with 5 % carbon black matches the cometary phase curves the best in terms of overall reflectance, curvature and opposition peak. Mixtures with coarse grained ice are less convex in general than mixtures with fine grained ice.

First the phase curve of pure activated charcoal is extremely flat and much darker than the cometary phase curve. The mixture of coarse grained ice particles with 29 % charcoal seems to have a nearly constant offset compared to pure charcoal. Coarse ice mixed with 20 % charcoal is by more than factor of

two brighter than 29 % and the phase curve is more curved, especially at phase angles smaller than 25° . The mixture of 2 % carbon black with coarse ice is very similar to the mixture with 29 % charcoal but the slope is steeper at $\alpha < 15^\circ$, indicating a more pronounced opposition peak. A similar behaviour is observed
425 for the mixture of fine grained ice with 50 % charcoal. Fine grained ice with 5 % carbon black shows the most resemblance with the cometary phase curve over all geometries. The maximum amplitude at opposition is clearly lower than on the cometary curve but firstly the minimal phase angle in OSIRIS data is 1.3° and the opposition peak is therefore extrapolated, and secondly our instrument
430 underestimates the amplitude of the peak at exact opposition due to its limited angular resolution (see discussion in [Jost et al., 2016](#)). By normalising the phase curves at $\alpha = 5^\circ$ in [Fig. 9](#), absolute differences in reflectance disappear and variations in shape of the phase curves become more visible. The overall darkest samples (pure charcoal and coarse ice with 29 % charcoal) show the flat-
435 test phase curves whereas 67P displays the steepest curves. A steep phase curve is usually related to highly porous and/or very rough surfaces where shadowing effects play a bigger role. While large-grained ice particles samples have a bulk density of $0.4 - 0.5 \text{ g/cm}^3$, fine grained ice samples have bulk densities from 0.11 to 0.19 g/cm^3 and are much more porous.

440 4. Discussion

After having measured different mixtures of water ice and non-volatile samples, we discuss in this section their different photometric characteristics and set them into the context of the general characteristics of the surface of 67P as well as some local anomalies.

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To call two surfaces similar or identical from the point of view of spectrophotometry, different characteristics have to match:

1. The overall brightness/absorptivity, usually expressed by the hemispheric albedo (not influenced by texture)

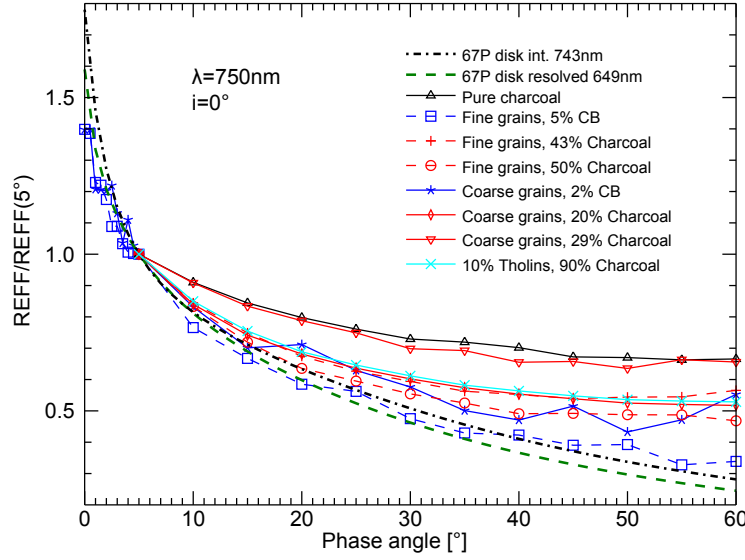


Figure 9: The same phase curves as in Fig. 7 but normalised at $\alpha = 5^\circ$

2. The normal albedo, additionally influenced by the opposition effect (roughness, porosity, grain size)
3. Reflectance spectrum, spectral slope, depth of absorption bands
4. Dependence of the spectrum on the illumination and observation geometries (spectral reddening/bluing)
5. Shape and magnitude of the bidirectional reflectance phase curve
6. Polarisation phase curve (not studied in this work)

To create an ice-bearing two component mixture that matches a given albedo is fairly easy, as long as the darkening agent is darker than the targeted value. But the resulting weight mixing ratio is not unique since the grain sizes of the two materials and the resulting ratio can vary. To infer a darkening agent content from the measured albedo at least a rough estimate of the grain sizes has to be known.

In the case of exposures of metre-sized water ice spots (Pommerol et al., 2015b) which are about ten times brighter (i.e. $A_H \approx 0.2$) than the surround-

ing cometary surface and assuming cometary ice grains in the $33 - 72 \mu\text{m}$ range (Filacchione et al., 2016) and individual dust grains smaller than $50 \mu\text{m}$ (Schulz et al., 2015) we obtain a size ratio $d_{ice}/d_{dust} > 1$. The mixture of fine-grained ice with charcoal in Fig. 4 shows a similar size ratio. An extrapolation of this
470 curve results in a charcoal content of about 5 % to reach an albedo of 0.2, The same ice grains mixed with submicron carbon black particles would need a dust percentage below 1 % to achieve an albedo of 0.2. This is a clear indication that these exposures have to be relatively new and unaltered. To have such
475 low dust content on a metre scale on a cometary surface the bright features are unlikely to be primordial (dust-ice wt. ratio >1) but have to be a result of subsurface recondensation processes, where upstreaming gas from deeper layers is cold trapped below the surface (Filacchione et al., 2016).

The normal albedo is the most difficult parameter to analyse when compar-
480 ing our laboratory measurements to remote sensing data, since it depends on a single data point (at $\alpha = 0^\circ$) on the phase curve. Hence it is more affected by systematic errors and SNR-limitations that are more prominent when using our zero-phase-angle setup. We underestimate the normal albedo in our lab data by 5 – 10 % (see discussion in Jost et al., 2016). In addition to the overall
485 brightness of a surface the normal albedo is very sensitive to the contribution of the opposition effect, caused by two different mechanisms. The Shadow Hiding Opposition Effect (SHOE; e.g. Hapke, 1986) is mainly influenced by the macroscopic surface roughness/surface texture, whereas the Coherent Backscattering Opposition Effect (CBOE; e.g. Wolf and Maret, 1985) depends on wavelength,
490 particle size, mean free path (influenced by bulk porosity and packing density) and absorption. Since our cometary analogues are relatively dark, the contribution of CBOE is expected to be low, as shown in Fornasier et al. (2015) and Feller et al. (2016). But this seems not to be generally true for mixtures of bright and dark constituents. In our phase curves (Fig. 6) the porous sample
495 with fine grained ice has significantly higher opposition amplitude than the more compact sample with large-grained ice particles. There is a clear dependence

of porosity and particle size on the opposition peak amplitude which cannot be easily explained by simple shadow hiding mechanisms.

500 The finding that micrometre-sized activated charcoal displays a red spectral slope while nanometre-sized carbon black is spectrally blue is unexpected but can be explained by different scattering regimes. Such a behaviour is predicted by Mie and Rayleigh theory for spherical particles, as the scattering efficiency Q_s of a particle is dependent on size parameter $x = \pi * D/\lambda$ (and therefore on
505 particle size and wavelength). The maximum efficiency should lay around $x = 5$ (see e.g. [Liou, 2002](#)).

There are numerous indications that this effect is not restricted to spherical particles only, as spectral blueing is observed on a variety of dark surfaces
510 throughout the solar system which are not made of spherical particles. Whether Rayleigh scattering is a potential explanation for blue spectral signatures on solar system bodies, such as Saturnian satellites, was discussed in detail recently in [Clark et al. \(2008\)](#), [Clark et al. \(2012\)](#) or [Brown \(2014\)](#). In case of our laboratory sample material activated charcoal with particle sizes of some micrometres
515 has size parameters larger than 100 in the visible spectral range should be in a Mie scattering regime while carbon black with particle size three magnitudes smaller has size parameters below 1 and hence be in a Rayleigh regime. The theoretical size for transition between the two scattering regimes lies between $0.7 \mu\text{m}$ (at 450 nm) and $1.7 \mu\text{m}$ (at 1064 nm).

520 The geometry dependence of the spectral slope (phase reddening/bluing) is still a poorly understood phenomenon. The surfaces of most Solar System objects display an increase of spectral slope with phase angle (reddening, see [Section 2.3](#)). This effect needs to be characterised and corrected in order to
525 compare spectra of different surfaces or spectra of a surface acquired at different times and at different phase angle.

By date no physical law predicts a linear phase slope curve and explains the origin of the nonlinear deviation at small phase angles of an almost linear curve in our experimental data. A similar behaviour was found in experimental data (Gradie et al., 1980) and observations of asteroids (Clark et al., 2002). Effects of coherent backscattering mechanisms (Shkuratov et al., 1997; Shkuratov et al., 1999) can be excluded as they mainly act at $\alpha < 2^\circ$, see e.g. Poulet et al. (2002). In Schröder et al. (2014) the role of microscopic and macroscopic surface roughness as a potential cause for monotonic reddening is discussed in detail. Our laboratory samples are all prepared flat on a macroscopic scale while the surface of 67P is not flat at all.

Our measurements reveal that not only the reddening agent (tholins here) has a major influence on the spectral slope but the carbonaceous compound as well. When the carbon particles are small compared to the wavelength (Rayleigh scattering) it is very difficult to achieve a phase reddening similar to the one observed at the surface of 67P. Extreme mixing ratio of $m_{tholins} \gg m_{cb}$ would be required to mimic the observations. But in this case the overall magnitude of the spectrum would be far too high (see. Figs. 2 and 3). This sets another constrain on the minimum dust particle size on cometary surfaces of at least a few micrometres.

Further the tholins used in this study are known to be a good analogue of Titan aerosols but not necessarily representative of cometary matter. The analysis of our spectra indicates that the organic material at the surface of 67P nucleus shows a more linear red slope towards the NIR, whereas Titan tholins are almost transparent beyond 1 μm .

While the magnitude of a phase curve is mainly dominated by the albedo of the surface material, its shape depends on surface morphology parameters such as packing density/porosity or macroscopic roughness (boulders, pebbles). The macroscopic roughness does not only affect the reflectance at high phase

angle because of shadowing effects, but it also influences the shape of the oppo-
sition surge (see Fig. 8a in [Jost et al. \(2016\)](#)). A rough surface texture usually
broadens the SHOE at phase angles up to 20° . At intermediary phase angles
the curves become more linear and monotonically decreasing.

In reality the surface roughness is difficult to control and quantify on lab-
oratory samples. In this study all samples have a relatively smooth texture.
Samples composed of fine grained ice are rougher because the small ice particles
tend to form mm-sized agglomerates.

Another important parameter is the packing density or porosity which is
even harder to control than the roughness. There are two potential ways of
compacting a granular laboratory sample: a sample is compacted intentionally
by adding pressure on the material resulting in a higher bulk density, the second
way is a compaction of the top layer while preparing the surface. The second
option is much harder to quantify, it is well possible that the uppermost (pho-
tometrically relevant) millimetre of a centimetres thick sample is compacted
resulting in a vertical packing density gradient, but not showing any noticeable
change in bulk density.

The effects of porosity on the surface overall brightness as well as on the
phase curves are discussed in [Hapke \(2008\)](#) or [Shepard and Helfenstein \(2011\)](#).
In general compact surfaces appear brighter than porous ones, porous surfaces
have can have higher reflectance at low phase angles and steeper decreasing
phase curves.

In [Poch et al. \(2016b\)](#) it has been demonstrated how a highly porous ($> 90\%$)
sublimation residue of smectite phyllosilicate can change its VIS-NIR spectrum
compared to the more compact original state. In the VIS the originally red spec-
trum became blue, while in the NIR some prominent absorption features vanish.
The creation of very porous surfaces as expected in a low gravity environment

590 on comets is not directly applicable in the lab under ambient pressure conditions.

The nearly perfect match observed between the lab phase curve measured for fine grained ice mixed with 5 % carbon black and the phase curve at the surface of 67P does not mean that the surface on 67P consists of such a mixture. But
595 very pronounced opposition peak on 67P with a high amplitude induces that the uppermost layer (mm to cm) of the comet has to be very porous, since it was impossible to reproduce such a sharp peak using any of our more compact samples. The mixture of 90 % charcoal with 10 % tholins which is the best proxy for the cometary albedo and spectrum has a flatter phase curve with a
600 less pronounced opposition effect. It is very likely that a highly porous matrix of this sample material could mimic the cometary phase curve. Such a structure would be very fragile under terrestrial pressure and gravity regime. [Poch et al. \(2016a,b\)](#) could create such a structure by subliming an ice-bearing mixture of organics and minerals in a thermal vacuum, but it was not possible to transport
605 the sample to the goniometer without damaging the surface residue.

5. Conclusion

By systematic varying input parameters such as grain sizes, water ice content, addition of complex organic compounds and mixing ratios, we were able to produce a laboratory model of the individual global photometric properties such
610 as reflectance spectrum, phase curve, albedo and phase reddening of comet 67P. It was not yet possible to find a laboratory analogue that can provide a match to all cometary surface properties together. This means that at least the composition of our samples is different. Up to now we did not include yet any silicate- or iron-bearing minerals which are known to be present in interplanetary dust.
615 Our measurements with different carbon compounds support the idea that a lower limit of the mean dust particle size is in the range of some hundreds of nanometres.

Our work demonstrates, in a simple way, that the inversion of a surface
 620 composition from remote sensing data in the VIS-NIR spectral range is not
 straightforward. There are various degrees of freedom such as grain sizes, mixing
 ratios, physical structure and texture. To achieve a robust match it is vital to
 constrain at least some of the unknown properties by complementary techniques
 and instruments.

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