Qualitative investigation on VOC-emissions from spruce (*Picea abies*) and larch (*Larix decidua*) loose bark and bark panels

Günther Kain1, Daniel Stratev2, Eugenia Tudor1,3,4, Bernhard Lienbacher1, Martin Weigl2, Marius-Catalin Barbu1,3, Alexander Petutschnigg1,5

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Abstract

Tree bark is an interesting source for various building and furniture materials. It holds a high number of volatile components—resulting in emissions whose effects on building residents have to be considered. Spruce and larch barks were dried using different methods and prepared as loose particles and panels. The VOC (volatile organic compound) emissions after 3 and 14 days were measured in a small chamber using gas chromatography and coupled mass spectroscopy. The influence of bark type, drying method, hot pressing and time on the emissions was quantified. The total volatile organic compounds (TVOC) emissions from spruce bark were higher than those from larch bark. High-temperature treatment and time significantly decrease TVOC emissions from the investigated barks. Terpenes, aldehydes and acids were analysed in the emitting gases. The high temperatures whilst pressing panels are problematic due to furfural constitution. Its emissions and partly those of 2-octenal and acetic acid are relevant for present and upcoming evaluation schemes. Aldehydes are the critical substance when using bark in the interior and must be controlled in product development.

1 Introduction

One aim in material sciences is to find new materials, another to derive innovative utilisation opportunities for existing resources. Mainly used for energetic purposes (Feng et al. 2013), tree bark could be an alternative resource for building insulation boards (Kain et al. 2018), loose blow-in insulation (Kain et al. 2013), pallet blocks (Heinzmann and Barbu 2013), coating material in laminated flooring (Tudor et al. 2018) or particleboards (Nemli and Colakoglu 2005). There is also work focusing on the partial replacement of wood particles (Wang et al. 2015) and wood fibres (Xing et al. 2007) by bark particles or fibres.

Indoor air quality is a key parameter for residential and office buildings. Volatile components contained in building elements are critically discussed for some time in this respect. The amount of very volatile, volatile and semi-volatile organic components (VVOC, VOC, SVOC) emitting from building products is subject of national and international measurement standards and regulations (EN 16516 2018; EN ISO 10580 2010; EN 12460 1-5 2016; ISO 16000-9 2006; ISO 16000-6 2011; JIS A 1460 2001; JIS A 1901 2003; JIS A 5905 2003; JIS A 5908 2003; CARB-ACTM 2007). The European Union has proposed an evaluation framework for the characterisation and evaluation of indoor emissions from construction products, although this is not enacted yet and lacks limit values (European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013).

The exposure to VOC can cause allergy symptoms, eye irritation, headache and insomnia (Jensen et al. 2001; Salthammer et al. 2010; Liu and Zhu 2014; Kim et al. 2015). The effect of VOC-emissions from wood-based building materials on air quality is discussed controversially in the...
literature. Whilst some authors state that they are negligible (Nyrud et al. 2012; Fürhapper 2017), others argue that emissions from wood-based construction materials are a significant factor for indoor air quality (Harb et al. 2018) and that temperature has a great effect in this respect (Jiang et al. 2017). Heat treatment whilst drying and hot-pressing was shown to be a means to influence the amount of VOC emissions from wood-based composites, like for example reducing primary and increasing short-term secondary emissions (Makowski and Ohlmeyer 2006; Jiang et al. 2017).

Wood is primarily composed of cellulose, hemicelluloses and lignin but also contains minerals, resin acids, oligosaccharides, fatty acids etc., as well as volatile and semi-volatile organic substances (primary emissions). The VOC emissions of softwood-based (and thermally treated) materials are mostly terpenoids (mono-, sesqui-terpenes etc.) and aldehydes. With materials based on deciduous tree species, organic acids and aldehydes are more pronounced (Risholm-Sundman et al. 1998; Pohleven et al. 2019). Aldehydes, for example pentanal, hexanal, furfural, benzaldehyde and carboxylic acids, belong to the secondary emissions, which are formed because of oxidation or hydrolysis of other wood constituents (fatty acids, hemicelluloses etc.). Other volatile substances like formaldehyde or isocyanates (typical adhesive components) are introduced during the production process of wood composites and their emission mechanisms are more complex.

Tree bark contains significantly higher amounts of extracts than wood, such as lignans and neolignans (phenylpropanoids), norlignans (diphenylpentanes), flavonoids (diphenylpropanes), stilbenes (bibenzyls, phenantrenes), isoprenoids (tropolones, sesquiterpenes), tannins (condensed catechins, hydrostilbenes, hydrolysable gallotannins, ellagotannins), and other compounds such as saccharides, triglycerides, waxes, phenols and alkaloids (Sládková et al. 2015). Previous research on tree bark emissions has been focused on mercury vapour (Hanson et al. 1997), nitrogen (Schulz et al. 2001) or SO2 when burned as a fuel (Oglesby and Blosser 1980). In previous studies, the formaldehyde emissions of insulating panels made of larch (Larix decidua) bark bonded with UF, MUF and tannin, with a density of 0.5 g/cm3 were analysed. The results ranged between 0.175 and 1.850 mg HCHO/100 g for the perforator method (ÖNORM EN ISO 12460-5 2016) and between 0.010 and 0.094 mg/l for the desiccator method (JIS A 1460 2001) at a moisture content of 6.5% (Lohninger 2014). The substantially lower values, compared to reference specimens, could be explained by chemical bindings between free formaldehyde and components contained in the bark (Nemli and Colakoglu 2005).

Primary VOCs contained in bark have a fungistatic and bacteriostatic role and contribute to the protection against the attack of microorganisms, together with other non-volatile components (Salem et al. 2014). Another natural function of bark’s volatile emissions is to attract insects within plant communities (Szmigielski et al. 2012). VOCs have to be classified when bark is used in interior rooms before estimating their effect on the human organism.

In this study, a small-scaled method, described by Weigl et al. (2015), for the evaluation of volatile organic components from spruce (Picea abies) and larch (Larix decidua) bark is used. A small test chamber (1.68 l volume) was used to analyse the VOC-emissions of the two bark species. The aim of this study is to assess which VOCs emit from differently processed bark. Furthermore, the effect of the drying technique, hot-pressing process and time on VOC emissions was evaluated on a qualitative basis in order to develop hypotheses for further quantitative research on this issue.

2 Materials and methods

2.1 Testing material

Spruce (Picea abies (L.) Karst.) and larch (Larix decidua Mill.) bark particles were collected in small sawmills from Upper Austria and Salzburg. Sample taking was conducted following the rules of Kooperationsplattform Forst Holz Papier (2016) for industrial wood chips acceptance. The bark material, which was peeled from freshly felled trees and stored at a bark pile in the sawmill, was collected in April 2017 and had a moisture content (MC) of 23% in the case of larch bark, and 18% in the case of spruce bark, when taking samples.

The particles were crushed in a 4-shaft drum chipper RS 30 (Untha, Golling, Austria) and sieved in order to obtain particles with a size larger than 8 mm and smaller than 12 mm. These particles were subjected to different treatments. A mass of 1 kg of particles was prepared for each treatment. The first charge was air-dried in a climate of 20 °C/65% relative air humidity (RH) for 19 days until a MC of 17% (spruce) and 18% (larch) was reached. The second charge was chamber-dried at a temperature of 170 °C for 25 min in order to simulate an industrial drying process with high temperatures. After drying, the MC of the spruce particles accounted for 10% and 8% for the larch particles. Their loose density was 229 kg/m3 for the spruce bark and 137 kg/m3 for the larch bark. Three bark particleboards with a size of 0.33 × 0.24 × 0.025 m3 and a target density of 600 kg/m3 at the given MC were pressed in a laboratory hot press (Höfer HL OP 280 1 × 1 m2, Taiskirchen, Austria) at a press temperature of 210 °C for 5.4 minutes (press factor 13 s/mm). One of them (LP0—Table 1) was used for optimising the parameters of the analytical method and the other two (LP and SP) were used as test samples. No resin for particle binding was added. The panels obtained were adequately stable by the self-bonding mechanisms of bark (Gupta et al. 2011). The panels were acclimatised at 20 °C and 65% RH until
mass constancy was reached. The spruce panel had a density of 583 kg/m³ and the larch panel 602 kg/m³ afterwards.

The production of adhesive-free laboratory panels was chosen in order to study the effects of material compaction and pressing temperature on VOC species by eliminating adhesive interactions. In case of industrial application, it is assumed that adhesive addition would alter the emission characteristics due to:

- Introduction of VOC components belonging to the adhesive or originating from adhesive degradation.
- Different (lower) press factor when using binding resin and hence different heat impact on the panel.
- Introduction of diffusion barriers (adhesive layer) and altered sorption behaviour of the material.

Loose particles and panels were sealed in airproof plastic bags and transported to the laboratory for further analysis. Circular samples with a diameter of approximately 148 mm were cut out of the panels and the edges were sealed with an aluminium foil. The weight of the bark panel discs varied between 215 and 225 g (Table 2). Seven samples with different treatment were considered for the analysis (Table 1).

### 2.2 Experimental setup and procedure

VOC emissions were analysed using the experimental setup of Fig. 1. A small aluminium cylindrical vessel with a FLEC-cell used as a lid and a total volume of 1.68 l was used as a VOC test chamber. This apparatus will be denoted “FLEC-chamber” in the current paper and is not to be confused with a FLEC-cell, which is used in a different manner (DIN EN ISO 16000-10 2006). All samples were stored until sample taking in a testing chamber with a volume of 1 m³ at 23 °C and 50% RH and an air change rate of 1 h⁻¹. The sample’s VOC emissions were determined in the small chamber (1.68 l volume) on day 3 and 14 after the start of the acclimatisation.

The area-specific flow rate in the FLEC-chamber was set at 1.0 m³/(m² h), the unsealed area at the bottom of the samples was not considered in the calculation. For the analysis of loose particles, 220 g of bark were used for VOC analysis. A higher vessel under the FLEC-cell was used for larch bark, due to its lower bulk density and higher volume. The denotation of the test samples and the exact testing parameters in the small chamber are summarised in Tables 1 and 2.

### 2.3 VOC chemical analysis

A defined air volume of 2 l out of the chamber with a sampling rate of 100 ml/min was drawn through sorption tubes with a sample taking pump (FLEC Air Pump 1001). The VOC were adsorbed on a porous polymeric resin based on 2,6-diphenylenoxide (Tenax TA). This material is well suited for the adsorption of compounds with a GC-retention index of 600–2600, such as VOC with a boiling point of 50–250 °C and SVOC with a boiling point of 250–390 °C (World Health Organization 1987). The loaded Tenax TA-test tubes were analysed with thermal desorption and a linked gas chromatograph with mass spectroscopic detection (Agilent 6890N/5973i equipped with a Zebron ZB-5MSi 60 m x 0.25 m, 0.25 µm column). An example of two GC/MS-chromatograms is shown in Fig. 2.
The following substances, representing more than 85% of the cumulated peak area of the GC/MS-chromatograms, were evaluated: acetic acid, pentanal, pentanol, toluene, hexanal, furfural, heptanal, alpha-pinene, benzaldehyde, hexanoic acid, beta-pinene, 3-carene, p-cymol, limonene, 2-octenal, nonanal, longifolene and cadinene. Moreover,
the sum parameters TVOC, sum of terpenes, aldehydes, and acids were determined by summing up the concentrations of the relevant substances.

All evaluated volatile substances in this experiment, apart from cadinene, were quantified by using substance-specific calibration functions and by additional correction of the GC/MS signals (peak areas) through usage of the internal standard cyclodecane (ISTD in Fig. 2). Cadinene concentrations are given as toluene equivalents.

3 Results and discussion

The results from the specific concentration measurements are presented in Table 3. Instead of comparing single substance concentrations, the emphasis was placed on differences in the concentration of substance groups in this report. The absolute values may deviate from measurements taken according to the standardised methods (like EN 16516 2018 or ISO 16000-9 2006), because the chosen testing conditions differ from those defined in the standards (test duration is 14 days instead of 28 days, chamber volume is 1.68 l m³ instead of minimum 20 l). Longer testing time results in decreasing VOC concentrations, although this varies with substances (Makowski and Ohlmeyer 2006). For each factor level, only one sample was tested due to cost reasons, no inferential statistical methods were applied. The results should be interpreted as qualitative trends rather than focusing on the exact absolute values.

3.1 TVOC: total amount of volatile organic compounds

In this study, the amount of TVOC is the sum of all substances analysed. The bark species considered are quite different in this respect (Fig. 3). Spruce bark shows substantially higher TVOC emissions than the larch bark for all samples. This fact is particularly pronounced with loose bark. Compared with loose bark, the production of panels greatly reduces the emission of TVOC (Fig. 4). The highest TVOC values were measured with the oven-dried spruce bark.

Table 3 Concentration of substances in the testing chamber

| Substance or substance group | Subst. class | Concentration (µg/m³) | LP0 | LP | SP | LO | LA | SO | SA | EU-LCI |
|-----------------------------|-------------|-----------------------|-----|----|----|----|----|----|----|--------|
|                             |             | d3 d14 d14 | d3 d14 | d3 d14 | d3 d14 | d3 d14 | d3 d14 | d3 d14 | Value |
| Acetic acid                 | Acid        | 102 52 73 94 106 78 58 46 194 148 125 224 174 1200 |
| Pentanal                    | Aldehyde    | 3 20 17 22 9 40 8 <1 3 71 29 1 4 800 |
| Pentanol                    | Alcohol     | 42 18 20 32 21 46 18 34 17 89 39 34 19 730 |
| Tolual                      | Aromate     | 5 3 2 8 <1 9 <1 2 1 41 2 3 <1 – |
| Hexanal                     | Aldehyde    | 36 139 108 196 88 274 48 21 42 587 272 44 76 900 |
| Furfural                    | Aldehyde    | 35 46 60 32 22 7 7 2 1 5 6 3 2 10 |
| Heptanal                    | Aldehyde    | 1 3 2 5 3 7 2 1 1 15 9 3 3 900 |
| alpha-Pinene                | Terpene     | 201 23 8 116 39 97 25 114 45 968 263 707 243 2500 |
| Benzaldehyde                | Aldehyde    | <1 <1 <1 <1 <1 <1 <1 <1 <1 <1 <1 <1 <1 <1 – |
| Hexanoic acid               | Acid        | 49 20 20 25 26 54 30 34 18 159 110 38 20 – |
| beta-Pinene                 | Terpene     | 105 26 8 95 44 40 10 18 8 303 132 215 115 1400 |
| 3-Carene                    | Terpene     | 71 24 20 24 19 43 26 43 24 34 17 34 58 1500 |
| p-Cymol                     | Aromate     | 28 9 3 12 2 6 2 7 3 7 4 6 2 – |
| Limonene                    | Terpene     | 61 19 5 35 8 15 5 21 7 32 18 36 18 5000 |
| 2-Octenal                   | Aldehyde    | <1 16 4 10 5 17 3 <1 <1 <1 <1 <1 <1 <1 7 |
| Nonanal                     | Aldehyde    | <1 3 3 11 6 16 6 <1 <1 2 51 26 8 7 – |
| Longifolene                 | Terpene     | 6 4 4 40 23 4 <1 9 3 47 23 54 20 – |
| Cadinene                   | Terpene     | 2 <1 2 2 41 29 3 1 22 6 77 49 125 74 – |
| TVOC                        |             | 748 425 358 797 448 754 250 790 374 2670 1145 1536 836 |
| Terpenes                    |             | 446 97 46 350 163 201 66 227 93 1460 502 1170 529 |
| Aldehydes                   |             | 76 226 193 276 132 360 75 25 48 765 362 60 91 |
| Acids                       |             | 151 72 93 119 131 132 88 495 211 307 235 262 194 |

*d3, d14, d28 day 3, 14 and 28 after climatisation start, EU-LCI European Commission, Joint Research Centre, Institute for Health and Consumer Products (2013)*
Focusing on larch bark, the differences in TVOC concentrations (respectively emissions) between the three sample types are less pronounced than those of the spruce bark samples. The TVOC concentrations of the two loose bulk samples in this study are comparable and in the range of the spruce bark panel emissions. The larch bark panel shows the lowest day 3-TVOC concentrations and their rate remains comparatively unchanged until the fourteenth day. At this time, the amount of TVOC emissions of the loose larch bark and the spruce bark panel is similar.

It seems that loose bark particles emission is stronger than that of panels because of relieved VOC diffusion within the bulk and because of higher surface area. This is in accordance with Liu et al. (2015), who reported a strong influence of material structure on VOC diffusivity. As a result, the emission’s potential is reduced faster and the decrease in TVOC concentrations over time is stronger for loose particles compared to panels.

Focusing on spruce bark, oven-dried bark had the highest day 3-TVOC concentration with 74% higher than that of air-dried bark. The panel showed 70% lower emissions than oven-dried bark. On day 14, this ratio was eased. This “bake-out”-effect reported by Jiang et al. (2017) only occurred at the high temperatures in the hot press.

A comparison of the TVOC concentrations of the two panels showed that on day 3 the spruce bark panel has an 88% higher TVOC value than the larch panel (Fig. 5). On day 14, the difference is only 25%. Decreasing TVOC concentrations in the range of 450–800 µg/m³ on day 3 and 400–500 µg/m³ on day 14 should be unproblematic according to the EU LCI values (European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013). Moreover, because the emissions decrease over time, it appears obvious that by extrapolating the results to a standardised test duration (28 days), the TVOC concentrations should be even lower. However, the statements above have to be seen as estimations due to testing methods and TVOC definitions deviating from the EU LCI concept (European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013).

### 3.2 Terpenes

The terpene emissions make up 23–76% of TVOC emissions on day 3 and 13–66% on day 14. This is in accordance with the terpene ratio in coniferous wood shown in other investigations (Roffael 2006). The relation between different samples is similar to the one of the TVOC emissions. A difference is that the terpene emissions of spruce bark panels are clearly higher than those of all larch bark samples (Fig. 6). As with TVOC, the terpene emissions of adhesive-free panels are lower than those of loose particles, which can be referred to the smaller surface area, restricted diffusion paths (Liu et al. 2015) and bake-out. The measured decreasing terpene concentrations seem to be unproblematic from the perspective of the European product emissions evaluation (European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013) (Table 3),
Although direct comparability is not given in this study. This statement is supported by the observation that terpene emissions decrease over time and by the presumption that on day 28 the measured concentrations will also decrease.

### 3.3 Aldehydes and furfural

The concentration of aldehydes shows a different picture compared to those of TVOC and terpenes (Fig. 7). Although oven-dried spruce bark also emits higher amounts of aldehydes than the other samples, there is no clear difference between the two bark species. On day three, the aldehyde concentrations of samples LP, LO constitute approximately 50% of the TVOC emissions, which is a very high value compared to wood products (Roffael 2006). The bark panels showed aldehyde concentrations of 207 ± 61 µg/m³ on average during the test. Comparably low aldehyde emissions were observed with the air-dried samples, although they show a slight increase towards day 14. Figure 7 shows that the type of drying has a significant influence on the total aldehyde emissions. Drying the particles with a temperature of 170 °C results in noticeably higher aldehyde emissions than with air-dried samples on day 3. In case of wood, aldehydes are secondary VOCs and originate from the oxidation of unsaturated fatty acids (pentanal, hexanal) or from the split-off of hemicelluloses (furfural) (Roffael et al. 2015). These processes are highly temperature-dependent. Similar processes can be assumed for coniferous bark, which is supported by the present results.

Apart from furfural, the observed aldehyde concentrations (pentanal, hexanal, heptanal, benzaldehyde, nonanal) are not problematic from the perspective of the European product emissions evaluation (European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013) (Table 3). 2-Octenal-concentrations from loose spruce bark after 14 days accounted for 20 µg/m³ suggesting that its emission should be critically evaluated in a prolonged 28 days test (since the EU-LCI value after 28 days is 7 µg/m³). The furfural emissions from the panels LP and SP were much higher than the emissions from bark particles: LO, LA and SO, SA. This is in good agreement with the finding by Candelier et al. (2011) for solid wood, who reported increasing furfural emissions with temperatures above 200 °C. The AgBB- and the harmonised EU-LCI-value for furfural is 10 µg/m³ on day 28 (UBA Germany 2018, European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013). The furfural concentration in this study was 22 µg/m³ on day 14 for panel SP (with decreasing trend) and 60 µg/m³ for panel LP (with increasing trend). Consequently, it would be necessary to take further long-time measurements (28 days or longer) of furfural emissions in order to evaluate whether furfural is a problematic substance for indoor use of bark panels. Stratev et al. (2016) and Weigl et al. (2014, 2016) showed that under model room and real environment conditions VOC emissions from wood-based building products (like OSB or CLT) steadily decrease even after day 28. Finally, the press program (especially press temperature and press factor) seems to have a huge influence on furfural emissions, which might be an interesting aspect for optimisation. Moreover, the influence of different bark species on the furfural constitution should be addressed in further research.

### 3.4 Acids

On average, organic acids make up 23% (SD = 19%) of TVOC emissions on day 3. On day 14, they represent on average 32% (SD = 13%) of TVOC emissions. Air-dried larch bark showed by far the highest acid ratio of TVOC emissions of 63% on day 3 and 56% on day 14 (Fig. 8). When focusing on absolute numbers, it also had the highest acid emissions of all samples on day 3 resulting in test chamber acid concentrations of nearly 500 µg/m³ and was upon the highest emissions on day 14. Literature research
has shown that measurements of acetic acid could be significantly lower using TENAX instead of silica gel for sampling (Schieweck et al. 2018). Thus, the obtained values have to be considered carefully. It seems that the acid emissions of larch bark are reduced by technical drying and hot pressing. Spruce bark showed slightly higher acid emissions after technical drying than after air-drying and the lowest level after hot pressing.

The total acid concentration of both panels (LP and SP) was around 100 µg/m³ and, with consideration of the concentrations over time trend, should be uncritical according to European classification schemes regarding indoor air quality (European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013) defines a boundary value for acetic acid of 1200 µg/m³). Nevertheless, the used measuring system deviates from that of the standard and the measurements are not directly comparable.

4 Conclusion

The following conclusions can be drawn from the results of this investigation.

- Larch bark has considerably lower TVOC- and terpene emissions than spruce bark, independent of processing. The sum of VOC emissions of the investigated barks can be reduced by high-temperature treatment. All investigated samples showed decreasing TVOC- and terpene emissions over time.
- The drying process has a relevant influence on the aldehyde and acid emission of bark and consequently, influences the TVOC emissions as well. Technical drying at a temperature of 170 °C results in significantly higher aldehyde emissions out of both spruce and larch bark on day 3 and out of spruce on day 14. The high temperatures in the hot press also result in high aldehyde emissions. This is mainly due to furfural constitution. Air-dried bark has relatively high acid emissions, which are reduced by approximately 50% when the material is hot-pressed.
- Apart from furfural, octenal and possibly acetic acid, the measured sum of VOC, terpene and aldehyde (emitted from the adhesive-free panels) are estimated to meet the requirements of the European product emissions evaluation (European Commission, Joint Research Centre, Institute for Health and Consumer Products 2013). However, the experimental results of this paper are much more focused on the emission patterns and should only give a rough estimation whether guidelines are met or not.
- Both spruce and larch bark panels proved to have high furfural emissions, which were most probably formed because of heat-induced degradation of hemicelluloses initiated in the hot press. The drying process does not seem to have a significant influence in this respect. Prior to starting a potential industrial production, bark panels of these types should undergo further emission evaluation, using 28 days chamber tests as well as longer termed tests (several months) in order to estimate the potential furfural emissions reduction through storage.

In conclusion, there are indications that furfural and, to a lower extent, octenal emissions from spruce and larch bark panels could be hindrance for the indoor usage of engineered wood products based on these raw materials. It can be though suggested these emissions can be controlled (reduced) by altering the pressing and storage parameters. Acetic acid emissions should be evaluated using sampling on silica gel, because it is known that this leads to significantly higher values (Schieweck et al. 2018) and the validity of the present study could be improved in this respect.

These hypotheses as well as the role of adhesives for the internal VOC diffusion and VOC emissions should be discussed in further investigations.

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Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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