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Application News

Fourier Transform Infrared Spectroscopy

Quality control of dental material for the production of Night Guards using FTIR Analysis

User Benefits

- Fast screening of samples
- Analysis space minimal
- Quick handling using IRSpirit

■ Introduction

Quality control is not only a matter of daily consumer products. All things in touch with the human body especially in contact with body internal demands like pharmaceuticals, or food, etc are depending on an intensive quality control. The producers must follow a lot of requested tests before launching a pharmaceutical or clinical product. One of the interesting areas of the human body is the mouth and its interieur. It is the bit or denture. Without the bit the human can only eat the food in pasty, liquid or porridge like form.

Figure 1: View to a ready prepared night guard (cruncher rail or chin), colourless and transparent

Known is the grinding of the teeth when the load of the biting pressure becomes high. Especially to be heard at night. In this case it was of interest which materials were used for the preparation of a night guard and if they will be under control of European Pharmacopoeia.

The production of a night guard is done in three steps. It needs at first a negative form of the teeth. This is done with a flexible mass made from dry alginate powder. The powder will be mixed with water and a pasty very fluidic mass is prepared. This paste is used for the impression spoon which is pressed to the lower tooth jaw (in this case). After a short forming under little pressure (biting), the now rubber like mass (see fig. table1, 1st step) will be ready. In 2nd step will be prepared a positive form using plaster for filling the negative. And final the plaster cast (table1, 2nd step) as positive will be the base of the negative form for the night guard (table 1 3rd step). The forming from step 2 to 3 can be diverse- melting of polymer over the plaster or a 3D Scan from the plaster, and final printed with the 3D printer, etc.

The European Pharmacopoeia contains a Monograph about medicine which is used for the out and inside of the mouth [1], contains a regulation for Plaster in medical usage [2] and about sodium alginate. Even the polymer has its control in the Pharmacopoeia [3].
Independent from the demands of the pharmacopoeia the FTIR can be used for a quick screening for all the materials used for the crunching rail production.
A quick analysis with the Shimadzu FTIR in combination with ATR can be done with all parts directly, destroying free in manner of no chemical treatment is needed.

■ Methods

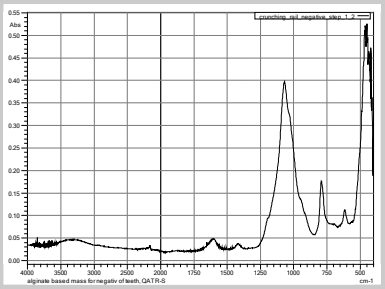
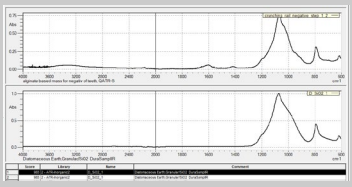
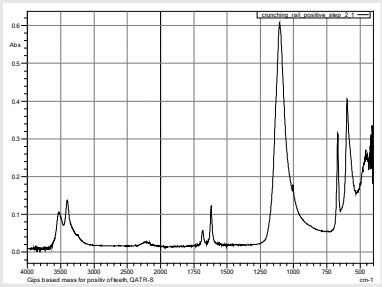
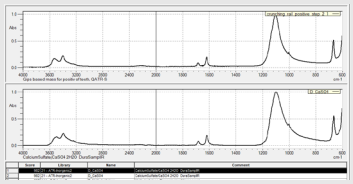
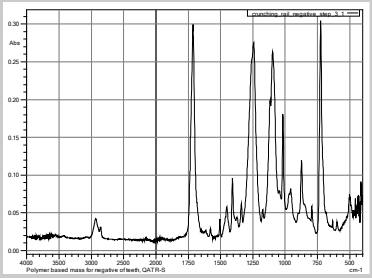
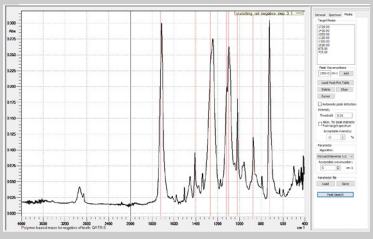
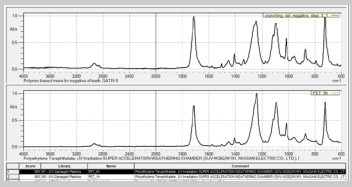
FTIR-ATR

To get a quick overview about materials the FTIR method is the right choice. Within seconds you can identify the material. As it is the talk about solids which will be tested without any chemical treatment. Just a piece of material generated as slice or particles., so as it is. The accessory for the analysis of the solid is the single reflection ATR technique with diamond window. For a good ATR measurement, the substance or polymer must be soft so that it can be pressed homogenously over the complete measurement area against the diamond window. The measurement window has a size of approx. 2 mm.

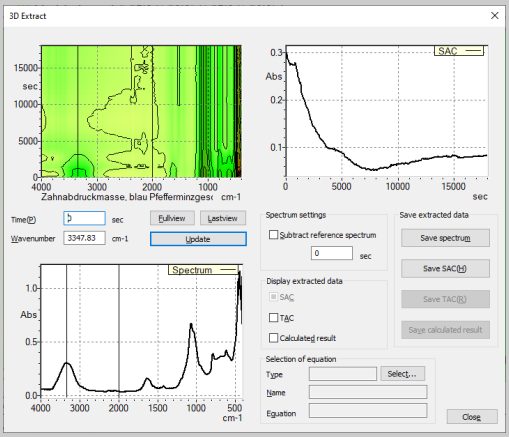
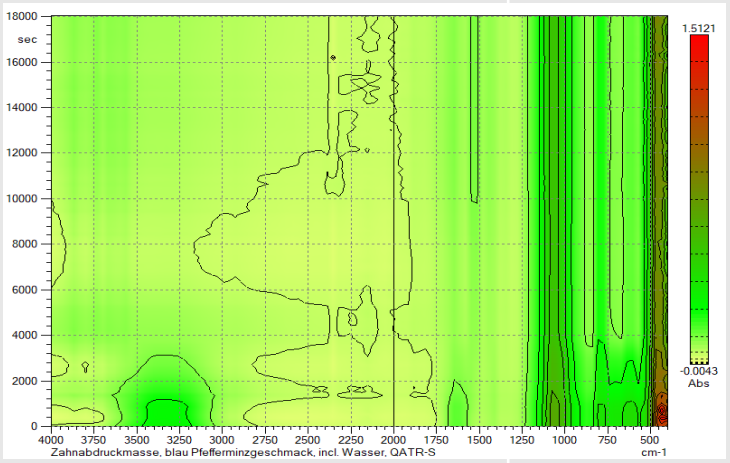
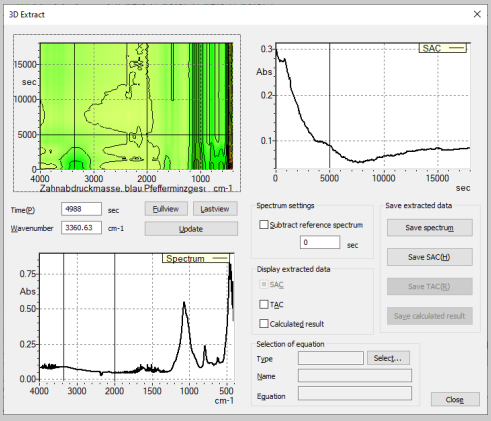
Analysis with FTIR

All three parts showed good infrared spectra. The contact of the material to the measurement crystal was good because all materials were soft enough to cover the window. Even the small polymer piece of 2 to 3mm in size. At all the library search helped identifying the chemicals, see table 1. The polymer was also analysed with peak search and pharmacopoeia limits for the identification of the signals. Additional it was of interest to observe how long the drying of the alginate needs to be still good enough as impression. See table 2 which contains the analysis result.

■ Table 1: Analysis table about FTIR results for dental issues

Item	Photo	IR-spectrum	Remark
1st step Negative form after bite into im-pression spoon	 The material is soft, flexible but stable and a piece of the smoothy material was analyzed.		 Search result: Diatomic Earth Granulate/SiO_2 plus, the sample spectrum showed add on at hydroxy group position (source H_2O) material.
2nd step Positiv form made with plaster	 With knife a small amount of particles from the form were scratched.		 Search result: $CaSO_4$ $2H_2O$
3rd step Negative prepared from polymer -the night guard	2 to 3 mm slice of the polymer from the final product, the crunching rail 	 	Search result is PET. Identification of PET via Pharmacopoeia is: 1725, 1410, 1265, 1120, 1100, 1020, 875, 725 cm^{-1} for a transmission film, the search with signal position tolerance of +-2 cm^{-1} resulted in a clear match (score 1000 from 1000) for the peak search. 

■ Table 2: Analysis table about FTIR alginate material and drying over time

Item	Photo	IR-spectra, Time-Curve at fixed analytical wavenumber, Topview	Remark
At start of analysis the alginate im-pression	 The fresh prepared material is soft, blue coloured, flexible but not fluid, rubber like. And after certain time it is drying to hardness.	 The SAC (selected absorption curve) at 3350 cm $^{-1}$ shows the drying over time.	The alginate spectrum is dominated by the water profile at 3350, 1630 and is as well the offset in the finger print region from 1500 to 600 cm^{-1} .
Total obser-vation 17864 sec (~5 h)	The water bands at 3350 and 1630 cm^{-1} are decreasing, in the middle of the graph is the region of diamond (accessory window) and environ-mental air (water vapour and CO_2 changes), the effect occurs because from the hardening of the material the measurement crystal will be not proper occupied by alginate solid anymore.		
At 5000 sec (~83 min), the minimum in timecurve	 The same piece but dried. With less water the colour changed from blue to crème - white.		After the drying is remaining the diatomic-earth IR-spectrum. The water signal disappeared at 5000 sec. Up to the end (5 h) the influence of the environ-mental water vapour (compared to the background measurement).

■ Result

All results are summed in the tables 1 and 2. As expected was in all three cases found the material which was declared on the raw material package.

The plaster was found as $CaSO_4$. The polymer rail was prepared from PET which could be identified with the support of library search and compared to the listing of the EP 10.6 quality control of PET for the identification.

Initiation of the analysis was the declaration of the alginate powder described as "dust-free alginate impression material". Expecting alginate-powder it was astonishing that it was found the diatomic-earth infrared spectrum. Which is correct. The diatomaceous earth is nothing different than the very old alginate sediments. Alginate is mostly prepared from the brown algae as residue product from iodine production. The advantage of the alginate is that it is a food (E400 and other) which forms a gelatine/jelly when mixed with water. The sodium alginate is used as an impression-making material in the dentistry.

The polymers used for the crunching rail can be PMMA, polycarbonate or PET for example. PET was found which can be qualified with European Pharmacopoeia packaging rules [3].

With the time course function, which allows observation of infrared spectrum over the time, it was possible to find the time in which the alginate impression could be flexible enough and on the other hand stable enough until it was used for the preparation of the positive impression with plaster. Drying of the material is an issue because the alginate impression form will shrink and is not more fitting after a while to the owner of the tooth impression. So, the time frame from preparation of jelly – impression of teeth – preparing of the plaster impression is of interest [4].

Figure 2: Time line of the production from a crunching chin, most important is the speed between alginate powder and its 1st contact with water.

At all it was possible to show possibilities of quality control for the materials in use of dental affairs.

■ Conclusion

The FTIR-spectroscopy is very helpful technique for the speedy identification of raw materials. With the support of the Library Search also untrained persons can have easily results. With the application time-course a physical characteristic of a material change can be studied. Two styles of information can be the result.

1. Infrared spectra from start and end of the reaction/kinetic study
2. The kinetic graph of the physical property. In this case the evaporation of water, separation of main material and little changes in the water bonding over the time.

■ Literature

[1] „Oromucosal Preparations", EP10.5, 01/2021:1807

[2] "Plasters, Medicated", EP10.5, 07/2021:3032

[3] "Polyethylene Terephthalate for containers for preparations not for parental use", EP10.6, 01/2008_30115, corrected 7.3

[4] "Alginate Impressions: A practical perspective", V V. Nadini, et all, J Conserv Dent. 2008 Jan-Mar, 11(1): 37-41

■ The Package IRSpirit

- ❑ *Main Unit*
  IRSpirit-T: Fourier Transform Infrared Spectrophotometer compact, speedy and stable, with TGS Detector and KBr-window
- ❑ *Accessory*
  QATR-S with diamond window and fixed anvil pressure
- ❑ *Software and Libraries*
  LabSolutionsIR and integrated libraries

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