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Research paper

Use of roughness maps in visualisation of surfaces

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Abstract

In this study we will present a new method to describe surface roughness. This method builds a roughness map of the studied area. The roughness map can give information of localised roughness. The test surfaces used in the evaluation of the method were tablets, which were made of lactose monohydrate, theophylline anhydrate, sodium chloride and potassium chloride. The roughness determinations were made by a laser profilometer. The new matrix method gives detailed roughness maps, which are able to show local variations in surface roughness values and provide an illustrative picture of the heterogeneity of surface roughness of various materials.

Keywords: Laser profilometer; Scanning electron microscopy (SEM); Tablet; Surface; Roughness; Roughness map

1. Introduction

Surface roughness is a critical parameter in various fields of material sciences. In painting, for example, the surface should contain some degree of roughness in order to provide enough adhesion between the paint and the subject surface. Also different kinds of papers have very different degrees of surface roughness, since there are different types of paper qualities e.g. for laser printing, ink-jet printing and newspaper. With pharmaceutics, surface roughness influences particle flow properties, wettability, surface friability, and the quality of final tablet coating. In the granulation and coating processes the surface roughness presumably has a remarkable effect on the adhesion of the polymer solution on the surface.

One well-established method for surface roughness measurement is non-contact laser profilometry or optical profilometry which has previously been used in pharmacy [1–5]. Laser profilometry has also been used in many different fields of material research like paper coating, dental materials and surgical prosthesis [6–8]. Laser profilometry is accurate, quantitative, flexible method and it can be used to study areas with diameters up to several centimetres. Problems with the laser profilometer can arise with materials, which have poor reflection properties.

Various roughness calculation methods have previously been used in the literature, but these methods often give only one value for the roughness of the surface, and they fail to visualize or give regional information of the roughness on the surface [1,2]. Fractal dimension has been also used to characterise surface roughness in different size scales [9–12]. On the other hand, it has been shown that the measured roughness is dependent on the size of the measurement area and the resolution [13].

The purpose of this study was to describe a new method, which can give information of localised roughness across a wider area and build up easily readable roughness maps of the surfaces. The test surfaces used in the evaluation of the new 'matrix method' were tablets made of lactose monohydrate, theophylline anhydrate, sodium chloride and potassium chloride.

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2. Materials and methods

2.1. Materials

The materials in this study were M325 lactose monohydrate (DMV International, Netherlands) and Ph. Eur. grade of theophylline anhydrate (BASF, Ludvigshafen, Germany); for reference purposes, materials used were analytical-grade of sodium chloride (NaCl) (Riedel-de Haën, Germany) and potassium chloride (KCl) (Riedel-de Haën, Germany). The particle size of the powders was determined with a Leica MZ-6 optical microscope (Leica DMLB, Leica Mikroskopie and Systeme GmbH, Germany) which was equipped with Leica QWin image-analysis software (Leica QWin V2.6, Leica DMLB, Leica Mikroskopie and Systeme GmbH, Germany). At least 800 particles were used in each particle size determination by measuring the horizontal and the vertical dimensions of which the averages were calculated.

2.2. Tablet compression

Tablets were compressed with a 13 mm evacuable IR tablet die (Specac Ltd, UK) and a hydraulic press (Pye Unicam, UK). The compression forces were 30 and 80 kN and the compression time was 2 min. Corresponding compression pressures were 225 and 600 MPa. The tablet die was evacuated by a vacuum pump during the compression in order to get strong and non-breakable tablets. Tablet weight varied from 300 to 450 mg. Prepared tablets were attached to metal sample plates with double-sided tape in order to assist in the handling and identification of the correct tablet surface.

2.3. Surface characterisation

2.3.1. Scanning electron microscopy

An SEM (Zeiss DSM 962, Germany) was used in order to obtain a large and accurate view of the tablet surfaces. The SEM pictures were also used as a reference for the laser profilometer measurements. The magnifications used were 200 lactose monohydrate and 500 for theophylline anhydrate, respectively.

2.3.2. Laser profilometry

Tablet surface roughness was measured with a laser profilometer (UBM Microfocus Measurement System, UBM Messtechnik GmbH, Ettlingen, Germany), which was also used to get 3D images from tablet surfaces. Tablet surfaces were studied using an image size of 2×2 mm and the resolution of 1000 points/mm. The roughness values R_a and R_{rms} were determined from one 2×2 mm area of each sample. After data collection the image data was levelled to remove slope caused by tilting of the tablet surface and tilting caused by the sample plate and double-sided tape using a data-analysis programme (Ubsoft version 2.8 DOS, UBM Messtechnik GMbH, Ettlingen, Germany). Laser profilometer images, showing actual measurement height data (2000×2000 data points per image), were further analysed by Mathematica 4.0 programme (Wolfram Research, USA).

2.3.3. Roughness parameters and roughness maps

Two basic roughness parameters R_a and R_{rms} were used in this study.

The average roughness (R_a) parameter was calculated from the height data according to Eq. (1)

$$R_a = \sum_{n=1}^{N} \frac{|Z_n - \bar{Z}|}{N}, \quad (1)$$

where Z_n was the individual height value of one measurement point and $\bar{Z}$ the mean value of all the height data points. N was the number of measurement points. The root mean square roughness (R_{rms}) was calculated from the standard deviation of the height data (Eq. (2)).

$$R_{rms} = \frac{\sqrt{\sum_{n=1}^{N}(Z_n - Z^2)}}{N-1} \quad (2)$$

The roughness parameters R_a and R_{rms} were calculated in two different ways: first by the standard method using the analysis program submitted with the equipment (Ubsoft version 2.8 DOS, UBM Messtechnik GMbH, Ettlingen, Germany) and then by our new matrix method, which gives

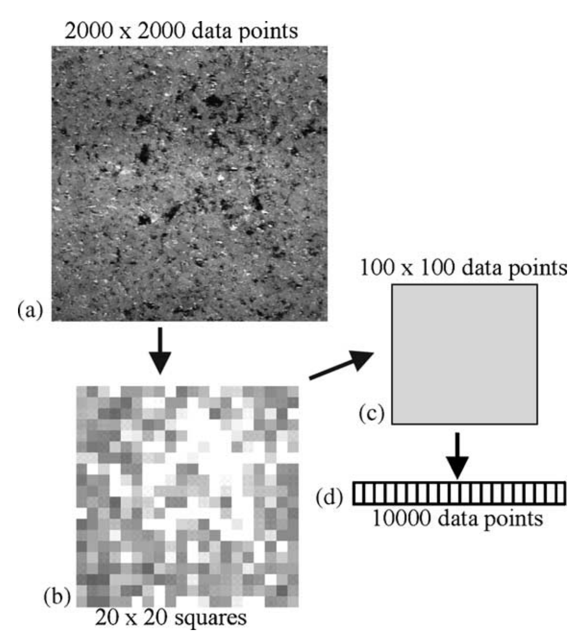

Fig. 1. Scheme of the matrix calculation method. (a) The original laser profilometer image (2×2 mm) showing the actual height differences, (b) the roughness map (2×2 mm) calculated from the same image, (c) one of the 400 squares (100×100 μm) and (d) the 10,000 measurement points in the square were organised into a row of numbers from which the roughness parameters R_a and R_{rms} were calculated.