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Influences of the experimental setup configuration on mass transfer measurements in absorption systems

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ABSTRACT

The standardisation of mass transfer measurements for absorption systems is a key factor for the deduction of accurate mass transfer models for random and structured packing. Several papers (Hoffmann et al. (2007). Trans IChemE, A, 85 (A1), 40; Kunze et al. (2012). Chem. Ingen. Techn., 84, 1931; Rejl et al. (2009). Chem. Eng. Res. Des., 87, 695) already deal with this problem and recommendations are given for appropriate test systems, the execution of the experiments and particularly also for the experimental setup to be used. However, systematic investigations of the influences of the experimental setup configuration on the results of mass transfer measurements in absorption systems are published rarely.

Mass transfer measurements with the system ammonia–air/water have been performed at a pilot plant consisting of a DN 600 saturation column and a DN 450 measuring column, both made from polypropylene, and equipped with different random and structured packing. Although the used experimental setup of the pilot plant follows strictly the recommendations published in Hoffmann et al., experimental results may differ significantly depending on, for example, the locations of the gas and liquid sampling in the column, the raw gas concentrations of ammonia or the pre-treatment of packing.

The paper presents the results of the test series considering different effects on the derived mass transfer performance of the packing and addresses also problems as well as solutions concerning the sampling in a two-phase regime. The aim is to provide a valuable contribution to the efforts for the standardisation of mass transfer measurements in absorption systems.

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1. Introduction

Mass transfer measurements at pilot plants are the basis for the dimensioning of industrial columns as well as for the mass transfer models developed during the last years. For this reason the quality of mass transfer measurements is very important which have to be conducted as conscientiously as

possible. A lot of influences on mass transfer measurements exist which need to be considered. Many of them are already mentioned in Hoffman et al. (2007), Rejl et al. (2009) as well as in Kunze et al. (2012). Nevertheless, there are further important influences to be reflected and in this paper the effects of the chosen raw gas concentration, sampling position, and the pre-treatment of plastic packing are considered.

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Symbols

A	cross sectional area of the column [m ²]
c	concentration [mol/m ³]
DN	diameter nominal
F	F-factor [Pa ^{0.5}]
FTIR	Fourier transformed infrared
H	height of packed bed [m]
H ₂ O	water
He(T _G)	Henry constant dependent on gas temperature [bar]
HTU _{OV}	height of a transfer unit (overall) [m]
k _{NH₃} (T _G)	Henry constant dependent on gas temperature [mol/(l ³ atm)]
MM	molar mass [kg/mol]
NH ₃	ammonia
NTU	number of transfer units [–]
p	pressure [Pa]
T	temperature [°C], [K]
u _L	liquid load [m ³ /(m ² h)]
$\dot{V}$	volume flow [m ³ /h]
x	liquid mole fraction [mol/mol _{ges}]
X	liquid mole load [mol/mol _{inert}]
y	gas mole fraction [mol/mol _{ges}]
Y	gas mole load [mol/mol _{inert}]
ρ	density [kg/m ³]

Indices

G, g	gas phase
i	component i
in	inlet stream
L, l	liquid phase
out	outlet stream
OV	overall
S	saturation

2. Methods

2.1. Experimental setup

The experimental plant at the Montanuniversitaet Leoben consists of a DN 600 saturation column and a DN 450 measurement column made of polypropylene which can be seen in Figs. 1 and 2.

The air is sucked from the environment and flows through the saturation column where it is saturated with water. The relative humidity of the sucked air and of the air leaving the saturation column are measured. After passing a tube type gas distributor, positioned 1 m below the packing, the gas streams through the measurement column. Afterwards, it enters a radial blower (frequency converted gear, max. 5000 m³/h, max. 5000 Pa pressure increase) and is then finally released to the environment. The gas flow is measured by a flow grid (airflow) downstream of the blower. Furthermore, the temperature of the gas is measured at every in- and outlet of both columns (Endress & Hauser, PT 100 type Omnigrad M TR 13; WIKA, type 55) which have their own liquid circulation. The packing is irrigated by a trough type liquid distributor which is situated 0.15 m above the packing (128 dripping points per m², free cross sectional area of 41%, three different operating ranges are available: 50–30 m³/(m² h), 10–60 m³/(m² h), 30–120 m³/(m² h)). While the saturation column possesses a

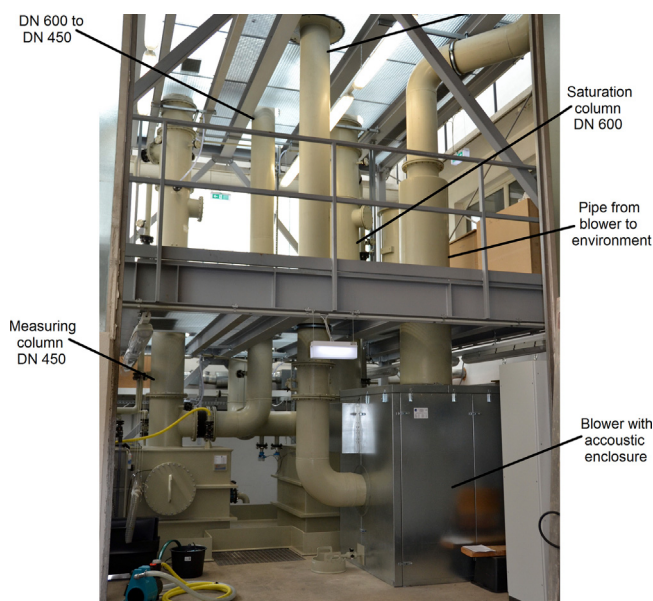


Fig. 1 – Experimental plant “packed column” at Montanuniversitaet Leoben.

closed liquid circulation, the liquid in the measurement column is only used for one single passage. It is collected below the gas entrance of the column in a pan type collector and conducted to a bin outside of the column. The used radial pumps are able to handle a volume flow of up to 50 m³/h. The liquid flow is measured by a magnetic inductive flow meter (Krohne, type optiflux 4000) and the temperature of the inlet stream at the measurement column is gauged by a resistance thermometer (Voltcraft, type K).

The mass transfer characteristic has been determined by using the absorption model system ammonia (NH₃)–air/water. The mass transfer resistance for this system is located in both phases although it is predominant in the gas phase (Hoffmann et al., 2007). This mass transfer system has been chosen because it is established at many experimental plants and therefore measurement data for validation exists.

The gaseous ammonia is injected in the gas duct between the saturation and the measuring column and has to pass a static mixer to ensure an optimal mixing. A constant ammonia dosage is ensured by the use of a pressure regulator, a valve as well as a float-type flow meter.

The concentrations of ammonia in front of and behind the packing are measured for each operating point (constant F-factor and liquid load) by an FTIR spectrometer (Gaset Technologies Oy, DX4000) in real time at a frequency of 1 Hz. Furthermore, the aqueous vapour concentration of the gas stream is also measured every second by the FTIR. This measurement is a key factor in monitoring that no water droplets are sucked in at the gaseous sampling location so that the test results do not get falsified as ammonia is readily soluble in water.

The concentration of ammonia in the liquid phase is analysed by photometric determination after it was preserved by the use of hydrochloric acid as receiver. This method has been selected as it is not sensitive to the CO₂–CaCO₃-equilibrium present in the piped water used due to a lack of huge amounts of deionised water for the necessary measurements (Gucher, 2013).

At the beginning of a test run, the experimental plant is only operated with water and air until steady-state conditions for the chosen operation point are reached. The mass transfer

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