

Filtration performance down to nano-particles

*Original*

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# FILTECH 2009

## CONFERENCE PROCEEDINGS

### VOLUME II

#### CONTENT VOLUME I

Scientific Committee	I-2
Session Survey	I-3
Conference Programme	I-4
Session Chairmen	I-17
Survey Lectures	I-19
Papers L-Sessions	I-95
Keyword List (Page Indicator)	I-587

#### CONTENT VOLUME II

Scientific Committee	II-2
Session Survey	II-3
Conference Programme	II-4
Session Chairmen	II-17
Papers G-Sessions	II-19
Papers M-Sessions	II-477
Keyword List (Page Indicator)	II-773

#### Conference Dates:

October 13 – 15, 2009

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Tuesday – October 13, 2009

Opening Ceremony		09:45-10:30	
Plenary Lecture		10:30-11:30	
	<b>Introduction to the characterisation of products prior to formulating a solid-liquid separation problem</b> , Dr. Christophe Peuchot, IFTS - Institute for Filtration and Separation, France		I-19
S1	Survey Lecture	13:15-14:30	
	<b>Advances in Pore Structure Evaluation by Porometry</b> , Dr. Krishna Gupta, Porous Materials, Inc - USA		I-21
L1	Sedimentation Analysis in the Gravity Field	13:15-14:30	
	<b>Evaluation of consolidation-sedimentation properties in batch gravity sedimentation of concentrated suspension</b> , N. Katagiri*, T. Hashimoto, E. Iritani, Nagoya University, Japan		I-95
	<b>Modeling the settling velocity of flocs using fractal geometry</b> , A. Vahedi*, B. Gorczyca, University of Manitoba, Canada		I-103
	<b>Laboratory scale evaluation of inclined settling</b> , T. Sobisch*, D. Lerche, LUM GmbH, Germany		I-112
M1	Waste Water Treatment	13:15-14:30	
	<b>Improved treatment of secondary effluent with ultrafiltration</b> , T. Peters*, Dr.-Ing. Peters consulting for membrane technology and environmental engineering, Germany		II-477
	<b>Processing and characterization of ceramic membranes for efficient removal of lignin from bleaching effluents</b> , M. Ebrahimi*, S. Kerker, A. Wienold, University of Applied Sciences Giessen-Friedberg; H. Neul, A. Ante, Bamag GmbH; M. Hilpert, Sappi Fine Paper Europe; P. Mund, Atech Innovations GmbH, Germany; P. Czermak, Kansas State University, USA		II-485
	<b>Electro-ultrafiltration of liposomal dispersions for the removal of trace micropollutants</b> , H. Saveyn*, M. Hakimhashemi, B. De Bock, P. Saveyn, P. Van der Meeren, Ghent University, Belgium		II-491
G1	Air Filter I	13:15-14:30	
	<b>The effect of pleat count and air velocity on the initial pressure drop and fractional efficiency of HEPA filters</b> , I. S. Al-Attar*, E. S. Tarleton, Loughborough University; R. J. Wakeman*, Consultant Chemical Engineer, UK; A. Husain, Kuwait Institute for Scientific Research KISR, Kuwait		II-19
	<b>Interaction of fluid with porous structure in filtration processes: Modelling and simulation of pleats deflection</b> , H. Andrä, O. Iliev, M. Kabel*, Z. Lakdawala, K. Steiner, Fraunhofer Institute for Industrial Mathematics ITWM, Germany; V. Starikovicius, Vilnius Gediminas Technical University, Lithuania		II-27
	<b>Importance of mechanical filtration in HVAC bags and panel air filter applications</b> , A. Boni*, Hollingsworth & Vose Europe, Germany; J. Manns, D. Healey, S. Cox, Hollingsworth & Vose, USA		II-32
S2	Survey Lecture	15:00-16:15	
	<b>Membrane Pore Characterization Techniques - Status Quo and Future Development</b> , Prof. Kuo-Lun Tung, Chung Yuan Christian University, Taiwan		II-37

<b>L2</b>	<b>Sedimentation Analysis in the Centrifugal Field</b>	<b>15:00 -16:15</b>
	<b>Sedimentation and consolidation behaviour of flocculated suspensions characterized by different methods measuring transmission</b> , T. Sobisch*, A. Zierau, D. Lerche, LUM GmbH; A. Bjeoumikov, IFG GmbH; M. Holke, IAP e.V., Germany	
	<b>The use of analytical centrifugation for the assessment of particulate matter compressibility</b> , P. Van der Meeren*, D. Curvers, H. Saveyn, Ghent University, Belgium; P. J. Scales, University of Melbourne, Australia	
	<b>Characterization of sedimentation and consolidation behaviour of kaolin suspensions in presence of dispersant</b> , C. Le Coeur*, O. Larue, E. Vorobiev, University of Compiègne, France; T. Detloff, T. Sobisch, A. Zierau, D. Lerche, LUM GmbH, Germany	
<b>M2</b>	<b>Produced Water Treatment</b>	<b>15:00 -16:15</b>
	<b>Application of inorganic membrane technology in the efficient treatment of oilfield produced water</b> , M. Ebrahimi*, D. Willershausen, L. Engel, University of Applied Sciences Giessen-Friedberg; P. Mund, P. Bolduan, Atech Innovations GmbH, Germany; P. Czermak, Kansas State University, USA	
	<b>Treatment of hypersaline oilfield produced water in a membrane sequencing batch reactor</b> , A. Fakhru'l-Razi*, A. R. Pendashteh, D. R. A. Biak, C. A. Luqman, Z. A. Zurina, University Putra Malaysia, Malaysia; S. S. Madaeni, Razi University, Iran; W. M. Zahid, King Saud University, Saudi Arabia	
	<b>Peroxidase-peroxide system catalyzed the removal of phenol and total hardness from produced water</b> , K. F. Mossallam*, F. M. Sultanova, N. A. Salimova, Azerbaijan State Oil Academy, Azerbaijan	
<b>G2</b>	<b>Air Filter II</b>	<b>15:00 -16:15</b>
	<b>Experimental investigation on the particle distribution and rearrangement in filter media</b> , T. Häusle*, A. Hammen, H. Sauter, Mahle Filtersysteme GmbH, Germany	
	<b>Fungal colonization of fibrous air filter media: Influence on filter's permeability and fungal particules release</b> , J. C. Bonnevie-Perrier, L. Le-Coq*, Y. Andrés, Ecole des Mines de Nantes, France	
	<b>The Eurovent certification program of air filters</b> , I. Bodenstaff*, AAF International, Netherlands	
<b>S3</b>	<b>Survey Lecture</b>	<b>16:45 -18:00</b>
	<b>Towards predicting filtration and separation - Progress and challenges</b> , I-48 Dr. Andreas Wiegmann, Fraunhofer Institute for Industrial Mathematics ITWM - Germany	
<b>L3</b>	<b>Sedimentation and Flotation for Sorting</b>	<b>16:45 -18:00</b>
	<b>Use of colloidal gas aphrons for separation of water based printing inks and impurities from paper stock suspensions</b> , D. Voß*, S. Schabel, University of Darmstadt, Germany	
	<b>Separation of fibre fines and inorganic fines in recovered paper suspensions</b> , I-145 G. Hirsch; S. Schabel, D. Voß*, University of Darmstadt; M. Feist; H. Nirschl, Karlsruhe University, Germany	
	<b>Tracer studies of the flow structure in a DAF pilot plant</b> , L. Jönsson*, I-153 University of Lund; M. Lundh, Kretsloppskontoret, Sweden	

**M3 Combined Processes 16:45 -18:00**

**Regeneration of stainless steel pickling solutions by a multi-stage process consisting of retardation, electrodialysis and membrane electrolysis,** H. J. Rapp\*, Osmo Membrane Systems; F. Rögener, M. Sartor, T. Reichardt, BFI, Germany II-521

**Total regeneration of mixed pickling acid from stainless steel production - Combination of nanofiltration and thermal processes,** F. Rögener\*, T. Reichardt, BFI; J. Schmidt, F. Knaup, Steuler Anlagenbau, Germany II-529

**Industry state of the art in membrane filtration of fruit juice and wine for product clarification,** E. Zimmer\*, D. Jermann, Bucher Processstech AG, Switzerland II-534

**G3 Surface Filtration 16:45 -18:00**

**Dust emission characteristics of pulse jet bag filters,** H.-S. Park\*, K. S. Lim, KIER - Korea Institute of Energy Research, Korea II-55

**Testing and analysis on performance of PSA filter media used for bag filter,** Z. Liang\*, H. Shen, Donghua University, P.R. China II-61

**Removal of fine particulate matter from exhaust gases by metallic micro-sieves,** E. Stahl\*, J. Robert, G. Deerberg, Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT, Germany II-67

**Wednesday – October 14, 2009**

**L4 Sedimentation in Centrifuges/Hydrocyclones 08:30 -09:45**

**Flow patterns and sediment build-up in tubular bowl centrifuges,** L. E. Spelter\*, H. Nirschl, Karlsruhe University, Germany I-161

**CFD simulation of flow and sedimentation in centrifugal field,** X. Romání Fernández\*, H. Nirschl, Karlsruhe University, Germany I-169

**Separation efficiency determining parameters in high gradient magnetic centrifugation,** K. Wagner\*, M. Stolarski, C. Eichholz, H. Nirschl, University Karlsruhe, Germany I-177

**L5 Poster Session I 08:30 -09:45**

**· Cake Filtration ·**

**Green liquor sludge separation, a comparison between gasifier and recovery boiler produced liquors,** T. Mattsson\*, T. Richards, Chalmers University of Technology, Sweden I-185

**Pilot scale research on oily sludge compression treatment,** X. Hu\*, S. Chengzhi, Y. Shufan, D. Jun, C. Chaozhong, Northeastern University; L. Chonghua, et al., PetroChina Liaohe Petrochemical Company, P.R. China I-193

**· Separation Enhancement by Physical and Chemical Slurry Treatment · Fundamentals of stability of sulfur in iron chelate,** K. Forsat\*, K. Mohammadbeigy, Research Institute of Petroleum Industry (RIPI), Iran I-201

**Dairy effluent treatment plant with UASB reactor,** E. Henríquez Díaz, O. Pérez Báez, A. Naranjo Ojeda, d. I. C. Ling Ling\*, University of Las Palmas de Gran Canaria, Spain I-208

**The comparability and optimization of different process of sludge dewatering, I-213**  
Y. B. Li\*, J. Jin, Liaoning Provincial Environmental Protection Bureau, P.R. China

**M4 Poster Session I**

**08:30 -09:45**

**Effect of polymer swelling on the nanofiltration performance of poly(vinyl alcohol), II-542**  
O. Farid, J. P. Robinson\*, University of Nottingham, UK

**$\beta$ -Cyclodextrin-Modified polysulfone membranes for the removal of endocrine disrupting chemicals (EDCs), II-550**  
S. Choi\*, S.-Y. Kwak, Seoul National University - Korea

**Preparation and characterization of aluminum oxide cermet microfiltration membrane using atmospheric plasma spraying, II-552**  
C.-C. Hsiung\*, T.-C. Ling, K.-S. Chang, K.-L. Tung, T.-T. Wu, Y.-L. Li, C.-H. Kang, W.-Y. Chen, D. Nanda, Chung Yuan University, Taiwan

**Preparation and characterization of novel hydrophilic low pressure nanofiltration membranes for water softening, II-560**  
M. Jahanshahi, A. Rahimpour\*, N. Mortazavian, Babol University of Technology, Iran

**Nanoporous polyethersulfone membranes prepared with synthesized poly(sulfoxide-amide) as additive in the casting solution for milk filtration, II-567**  
A. Rahimpour\*, Babol University of Technology; S. S. Madaeni, Razi University; A. Shockravi, S. Gorbani, Teacher Training University, Iran

**Supported lipid membrane systems for commercial aquaporin water filtration applications, II-575**  
J. S. Groth, M. Perry, T. Vissing, Aquaporin A/S; J. S. Hansen, J. Vogel, S. Ibragimova, C. H. Nielsen, O. Geschke, J. Ern us, Technical University of Denmark; C. R. Hansen, Copenhagen University, Denmark

**Drying of transformer oil with different filter techniques, II-579**  
C. Glasner\*, J. Robert, G. Deerberg, Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT, Germany

**Immobilization of fungal laccase on membrane and its use for decolorization of dye, II-587**  
N. Katagiri\*, Y. Ogi, E. Iritani, Nagoya University, Japan

**Membrane bioreactor with submerged ceramic flat membranes for the production of organic acids, II-594**  
T. Hahn\*, Z. Kovacs, I. Hannemann, K. Grau, University of Applied Sciences Giessen-Friedberg; H. J. Schmidt, Membrane Engineering GmbH; M. Kraume, Technical University Berlin, Germany; P. Czermak, Kansas State University, USA

**Long term experiences using microfiltration membranes for separation of bacterial biomass in recirculating aquaculture system, II-600**  
A. Gerbeth\*, B. Gemende, N. Pausch, M. Schwind, University of Applied Sciences Zwickau; A. von Bresinsky, Fischwirtschaftsbetrieb Andreas von Bresinsky; R.-P. Busse, Busse GmbH, Germany

**Research & development in microfiltration technology (MF) at KISR, II-607**  
A. Alsaffar\*, S. Bou-Hamad, A. Alsairafi, M. Alshimmiri, H. Alnaser, Kuwait Institute for Scientific Research, Kuwait

**G4 Poster Session I**

**08:30 -09:45**

**Simulation of DPF media, soot deposition and pressure drop evolution, II-74**  
K. Schmidt\*, S. Rief, A. Wiegmann, Fraunhofer Institute for Industrial Mathematics ITWM, S. Ripperger, Germany



**Modeling of particle layer detachment under consideration of transient kinetic effects**, Q. Zhang\*, E. Schmidt, University of Wuppertal, Germany II-81

**Theoretical considerations on optimization of fibrous filters structures for removal of fractal-like nanoaggregates**, A. Podgórski\*, M. Goszczynska, Warsaw University of Technology, Poland II-89

**Inertial deposition of aerosol particles in fibrous filters at low and intermediate Reynolds numbers**, V. A. Kirsch\*, Frumkin Institute of Physical Chemistry and Electrochemistry; D. A. Pripachkin, A. K. Budyka, Karpov Institute of Physical Chemistry, Russia II-92

**Theoretical study of the efficiency of nano-sized aerosol particles in a single fiber**, J. M. Silva, F. O. Arouca\*, J. A. S. Gonçalves, J. R. Coury, Federal University of São Carlos, Brazil II-99

**Experimental investigations of electrostatic precipitators with high flow velocities**, M. Kaul\*, E. Schmidt, University of Wuppertal, Germany II-106

**Investigation of regeneration mode in a compact granular bed filter for high temperature filtration**, K. Pathmanathan\*, J. E. Hustad, O. K. Sønju, NTNU Norwegian University of Science and Technology, Norway II-110

**Particle and H<sub>2</sub>S removal of ceramic filter system**, K. S. Lim\*, H.-S. Park, S. J. Park, KIER Korea Institute of Energy Research, Korea II-118

**Recovery of VOCs using small scale prototype unit based on electrically conducting carbon monolithic adsorbents**, P. Sklenickova, S. Tension, MAST Carbon International Ltd.; A. Wheatly, P. Row, Wellman Defense Ltd., UK II-123

**Venturi scrubber venturi efficiency for collection of particulate pollutants emitted by the burning vegetal biomass fuel**, M. A. Martins Costa, F. de Almeida Filho, S. Pupo de Moraes, B. de Araújo Lima, B. Santos Ferreira, D. Aparecido Silva Lopes, Paulista State University; M. Lopes Aguiar, N. A. Gómez-Puentes\*, Federal University of São Carlos, Brazil II-128

**Trajectory of the liquid jet into the throat of a pease-anthony venturi scrubber**, N. A. Gómez-Puentes\*, V. G. Guerra, J. R. Coury, J. A. S. Gonçalves, Federal University of São Carlos, Brazil II-136

**L6 Filter Media Characterization 11:00 -12:15**

**Cartridge bubble point tester**, A. Jena, K. Gupta\*, Porous Materials Inc., USA I-218

**A new method of measuring pore size distributions using multi-modal particle size standards**, G. R. Rideal\*, J. Storey, Whitehouse Scientific, UK; B. Schied, BS-Partikel, Germany I-226

**Microstructure simulation of virtual woven filter media**, E. Glatt\*, S. Rief, A. Wiegmann, Fraunhofer Institute for Industrial Mathematics ITWM, Germany I-231

**L7 Cake Filtration Analysis I 11:00 -12:15**

**Determination of pressure dependence of permeability characteristics from single constant pressure filtration test**, E. Iritani\*, N. Katagiri, Nagoya University, Japan I-239

**Characterization of packed beds obtained by filtration of colloidal suspensions**, M. Hieke\*, H. Anlauf, H. Nirschl, Karlsruhe University, Germany I-247

**Continuous pressure or discontinuous press filtration to separate slurries of very small particles – A theoretical comparison**, H. Anlauf\*, M. Hieke, Karlsruhe University, Germany I-252

**M5 Deposition Control 11:00-12:15**

**Improved deposition control for membrane bioreactors with immersed flat sheet membrane modules**, H. Prieske, L. Böhm\*, A. Drews, M. Kraume, Technical University Berlin, Germany **II-615**

**Effects of water quality and antiscalants on silica scaling of reverse osmosis membranes**, W. Hater, C. zum Kolk, P. Izquierdo, BKG Water Solutions; G. Braun\*, T. Götz, C. Esendiller, Cologne University of Applied Sciences, Germany **II-623**

**Particle deposition in rotating filter disks**, Y. Taamneh\*, Tafila Technical University, Jordan; L. Steinke, S. Ripperger, University of Kaiserslautern, Germany **II-640**

**G5 Industrial Gas Cleaning 11:00-12:15**

**Enhanced energy efficiency solutions for industrial baghouse filters**, G.-M. Klein\*, T. Schrooten, T. Neuhaus, R. Esser, F. Ott, T. Daniel, Intensiv-Filter GmbH & Co. KG, Germany **II-144**

**Conical lamina filter elements for higher filtration and energy efficiency and micro-fiber membrane filter media**, K. Schumann\*, Schumann Kompaktfilter, Germany **II-152**

**Improved performance of bag filters through fabric surface modification**, A. K. Choudhary\*, A. Mukhopadhyay, National Institute of Technology, India **II-157**

**S4 Survey Lecture 13:15-14:30**

**Development history and system integration aspects of diesel particle filters in commercial vehicles**, Dr. Achim Dittler, Daimler AG, Germany **I-64**

**L8 Cake Filtration Analysis II 13:15-14:30**

**Constant pressure filtration of fibre/particle mixtures**, K. Chellappah\*, E. S. Tarleton, R. J. Wakeman, Loughborough University, UK **I-260**

**Multi-staged creep effect in consolidation of tofu and okara as soft colloids**, E. Iritani\*, T. Sato, N. Katagiri, Nagoya University, Japan **I-268**

**Filtration-consolidation analysis of solid/liquid expression from biological tissue**, N. Grimi\*, E. Vorobiev, Technical University of Compiègne, France; N. Lebovka, National Academy of Sciences of Ukraine, Ukraine; J. Vaxelaire, University of Pau and Pays de l'Adour, France **I-276**

**M6 Membrane Fouling 13:15-14:30**

**Use of surface interaction free energy in the prediction of organic fouling of reverse osmosis (RO) membrane**, R. Bai\*, J. Miao, C. Liu, P. Tay, National University of Singapore, Singapore **II-638**

**Fouling transition in high molecular weight flexible polymer cross-flow ultrafiltration**, L. Béguin\*, IFTS Institute of Filtration and Techniques of Separation; H. Duval, M. Rakib, Ecole Centrale Paris, France **II-646**

**Effect of air-sparging on the performance of cross-flow microfiltration of yeast suspension**, K.-J. Hwang\*, C.-E. Hsu, P.-Y. Si, Tamkang University, Taiwan **II-654**

**G6 Filter Test Systems I 13:15-14:30**

**Comparison of differently generated soots used for filter testing**, S. Haep\*, H. Fissan, H. Kaminski, C. Asbach, B. Stahlmecke, H. Finger, Institute of Energy and Environmental Technology (IUTA), Germany **II-165**

**Filtration of nanoparticles: presentation of FANA test bench**, N. Michielsen\*, T. Lelandais, C. Brochot, S. Bondiguel, IRSN Institute for Radiological Protection and Nuclear Safety, France **II-172**

**Portable filtertester for nanometer and micrometer sized particles - the new all in one solution, lightweight no consumeables, no emissions,** F. Schneider, R. Hagler, M. Pesch, Grimm Aerosoltechnik GmbH, Germany **II-178**

**L17 Depth Filtration Processes 15:00 -16:15**

**The difficulty with filtering gel particles when producing man-made fibers and optical films,** S. Strasser\*, K. Brandt, Lenzing Technik GmbH, Austria **I-479**

**Development and characteristics of a new ion exchange filter cartridge made of phosphorylated hemp fibre yarn,** B. Gemende\*, N. Pausch, H. Mueller, A. Gerbeth, University of Applied Sciences Zwickau; M. Leiker, Produktions- und Umweltservice GmbH; J. Hofmann, U. Freier, K. König, Universität Leipzig; M. Feustel, A. Richter, Textilforschungsinstitut Thüringen-Vogtland e.V., Greiz, Germany **I-486**

**Media for water separation from biodiesel-ultra low sulfur diesel blends - comparison with super absorbant monitor media,** C. M. Stanfel\*, F. Diani Pangestu, Ahlstrom Filtration, LLC, USA **I-494**

**L9 Cake Filtration Processes I 15:00 -16:15**

**Experimental study on the influence of process variables on the performance of a horizontal belt filter,** M. Huhtanen\*, A. Häkkinen, J. Kallas, Lappeenranta University of Technology; B. Ekberg, Larox Corporation, Finland **I-284**

**Design of a new high performance drum filter for the chemical industry,** T. Langeloh, Bokela GmbH, Germany **I-292**

**Plate and frame pressure filter optimisation using plant load cell data: Advantages, challenges and outcomes,** R. G. de Kretser\*, H. Saha, C. Biscombe, P. J. Scales, University of Melbourne, Australia **I-300**

**M7 Modelling and Simulation 15:00 -16:15**

**Modeling of enzymatic synthesis of fructooligosaccharides in continous membrane reactors,** Z. Kovacs\*, L. Engel, K. Grau, T. Hahn, M. Ebrahimi, University of Applied Sciences Giessen-Friedberg, Germany; P. Czermak, Kansas State University, USA **II-669**

**Modelling the separation of protein solutions by means of cross flow filtration,** T. Grein\*, S. Ripperger, University of Kaiserslautern; A. Piry, W. Kühnl, U. Kulozik, Munich University, Germany **II-678**

**3D reconstruction of ultrafiltration cakes from binarised images,** F. Courteille, F. Bourgeois\*, M. Clifton, M. Meireles, Laboratoire de Génie Chimique UMR 5503, France **II-686**

**G7 Filter Test Systems II 15:00 -16:15**

**Filtration performance down to nano-particles,** P. Tronville\*, Politecnico di Torino, Italy; R. Vijayakumar, AERFIL LLC, USA **II-183**

**Essential improvements for a reliable fractional efficiency testing of air filters,** M. Schmidt\*, L. Mölter, Palas® GmbH, Germany **II-191**

**Investigation of the filtration behaviour of an artificial filtration test rig in comparison to an industrial filter unit – Differences and possibilities of scale up,** G. Gasparin\*, Evonik Fibres GmbH, Austria **II-199**

**L10 Depth Filtration Analysis I 16:45 -18:00**

**Advanced fibrous media simulations based on 3D structural data of real filter media,** M. J. Lehmann\*, S. Hiel, E. Nißler, P. Trautmann, MANN+HUMMEL GmbH, Germany **I-308**

**Analysis of the behaviour of an automotive fuel filter using a Brinkman-Darcy approximation and a probability density function for the two-phase flow**, L. Valiño, R. Mustata, J. Hierro, Laboratorio de Investigación en Tecnologías de la Combustión, J. L. Hernández\*, C. Blasco, Robert Bosch España Gasoline Systems S.A., Spain I-316

**On coupled particle level and filter element level simulation for filtration processes**, Z. Lakdawala\*, O. Iliev, S. Rief, A. Wiegmann, Fraunhofer Institute for Industrial Mathematics ITWM, Germany I-324

**L11 Cake Filtration Processes II** 16:45 - 18:00

**Ultra-fine coal dewatering with hyperbaric disc filters**, G. Krammer\*, J. Kappel, R. Raberger, Andritz AG, Austria I-329

**Saving of wash liquid at filtration**, R. Bott\*, T. Langeloh, Bokela GmbH, Germany I-337

**Secondary-Dewatering of solid-liquid separation in sodium bi-carbonate separation applications**, D.-E. Keller\*, KMPT AG, Germany I-345

**M8 Special Membranes** 16:45 - 18:00

**Catalyst crosslinked membranes for use in solvent resistant nanofiltration**, K. T. Cliff\*, S. Tarleton, Loughborough University, UK II-678

**Optimization of the channel form geometry of porous ReSiC ceramic membrane modules**, S. Alexopoulos\*, G. Breitbach, B. Hoffschmidt, University of Applied Sciences Aachen, Germany II-686

**Textiles for the filtration of activated sludge in membrane bioreactors (MBRs)**, L. Böhm\*, V. Iversen, S. Hermann, A. Drews, J. Münz, M. Kraume, TU Berlin, Germany; E. Fatarella, Next Technology Tecnotessile, Società Nazionale di Ricerca Tecnologica r.l., Italy; B. Lesjean, Berlin Centre of Competence for Water, Germany II-694

**G8 Hot Gas Cleaning** 16:45 - 18:00

**Use of CFD-software with simulation of particle formation and precipitation in high temperature processes**, T. van der Zwaag\*, C. Asbach, S. Haep, Institute of Energy and Environmental Technology IUTA; E. Kruis, University of Duisburg-Essen; K. Reuter-Hack, Karlsruhe University, Germany II-205

**Evaluation of filtration and recleaning performance of hot gas filter media**, R. Mai\*, H. Leibold, H. Seifert, Forschungszentrum Karlsruhe GmbH, P. Gäng, Fil T Eq GmbH, Germany II-213

**High Temperature Filtration of Pyrolysis Gases from Biogenic Feedstocks**, H. Leibold\*, R. Mai, J. Sitzmann, H. Seifert, Forschungszentrum Karlsruhe GmbH, Germany; A. Hornung, Aston University, UK; Y. Solantausta, VTT, Finland II-219

**Thursday – October 15, 2009**

**L12 Washing of Particles and Cleaning of Media** 08:30 - 09:45

**Flushing – Cleaning of debris and filter cakes from organic solvents**, M. Wilkens\*, U. A. Peuker, Technical University Bergakademie Freiberg, Germany I-350

**The impact of centrifugal force on the quality of cake washing**, F. Ruslim\*, A. Erk, T. Danner, BASF SE, Germany I-357

**Dissolution of magnetite particles in acidic conditions**, R. Salmimies\*, A. Häkkinen, J. Kallas, Lappeenranta University of Technology; B. Ekberg, Larox Corporation, Finland I-362

· **Washing of Particles** ·

**Influence of experimental parameters on local and filtrate properties of kraft pulp displacement washing**, K. Dingwell\*, J. Lindau, M. Sedin, H. Theliander, Chalmers University of Technology, Sweden

· **Backwashing Filtration Processes** ·

**Enhancing classical effluent treatment plant efficiency by introducing BASP rotary wedge wire drum dewatering screens**, B. Patil\*, V. Patil, BASP Industries, India

**Precious metal catalyst recovery in API processing**, L. Vashishta, Diva Envitec Europe Ltd., Great Britain; D. Stöcker, GKN Sinter Metal Filters GmbH, Germany

· **Depth Filtration Processes** ·

**Effects of Biodiesel By-Products on Interfacial Tension and Water Separation Properties of Biodiesel-Ultra Low Sulfur Diesel Blends**, F. D. Pangestu\*, C. M. Stanfel, Ahlstrom Filtration, LLC, USA

· **Chromatography** ·

**Preparative separation and purification of plasmid DNA nano-vectors using anion exchange expanded bed chromatography**, M. Ebrahimipour, M. Jahanshahi\*, Babol University of Technology, Iran

· **Electrocoagulation** ·

**Removal of arsenic from wastewaters by batch airlift electrocoagulation**, H. K. Hansen\*, P. Nuñez, C. Guiterrez, L. M. Ottosen, Technical University Federico Santa Maria, Chile

· **Filter Media** ·

**Choice and optimization of technical woven wire meshes in the solid liquid separation**, M. Knefel, P. Wirtz, GKD - Gebr. Kufferath AG, Germany

**Liquid-Liquid extraction of ammonia using hollow fiber membrane contactors**, M. Ulbricht\*, J. Schneider, M. Stasiak, Membrana GmbH, Germany; J. Munoz, A. Sengupta, B. Kitteringham, Membrana Charlotte, USA

II-709

**Achieving cleaner solutions**, H. Williams\*, Serfilco International, Great Britain; J. H. Berg, Serfilco Ltd, USA

**Development and large scale testing of water reuse process technologies in waste water free houses and companies based on ultrafiltration membranes of Microdyn-Nadir**, A. Huber\*, SCAUT Forschungsgesellschaft mbH; D. Swaboda, GFI GmbH, Germany

**Purification and recycling of water at a food-processing plant based on the example of natural sausage casing production using a physical-chemical-biological system with ultrafiltration membranes from Microdyn-Nadir**, A. Huber\*, SCAUT Forschungsgesellschaft mbH, Germany

**Integrated membrane process for treating desulfurization effluent**, N. Yin\*, F. Liu, Z. Zhong, W. Xing, Nanjing University, P. R. China

**Recycle effect on double-pass concentric circular mass exchanger with an idealized membrane inserted**, C.-D. Ho\*, J.-W. Tu, Y.-C. Chuang, Tamkang University, Taiwan

**Organic solvent nanofiltration in the pharmaceutical industry**, H. Beckers, A. Buekenhoudt, P. Vandezande, R. Vleeschouwers\*, VITO, Belgium

**Iron removal by membrane contactor for assisting ilmenite leaching, II-745**  
E. A. Abdel-Aal, M. H. H. Mahmoud, M. M. S. Sanad, Central Metallurgical R & D Institute, Egypt; A. Criscuoli, A. Figoli, E. Drioli, University of Calabria, Italy

**Filtration of highly concentrated CaCO<sub>3</sub> suspensions using a rotating disk dynamic system, II-756**  
M. Loginov, O. Larue, L. H. Ding, E. Vorobiev\*, University of Compiègne, France; N. Lebovka, National Academy of Sciences of Ukraine, Ukraine

**Process intensification by using dynamic Krauss-Maffei Cross Flow Filtration (DCF) without recirculation of retentate, II-763**  
G. Grim\*, KMPT AG, Germany

**Bench-scale unit for characterisation of particle adhesion on ceramic membrane surfaces, II-766**  
T. Quadt\*, E. Schmidt, University of Wuppertal, Germany

**G9 Poster Session II 08:30 -09:45**

**Isobaric pressurised air filter testing under overpressure up to 10 bar in accordance with ISO 12500, II-223**  
S. Schütz\*, L. Mölter, M. Schmidt, Palas® GmbH, Germany

**Method of achieving more accuracy in testing air cleaners, II-232**  
A. G. Denysenko\*, Kharkiv Petro Vasylenko National Technical University of Agriculture, Ukraine

**Errors on measurements of size distributions of nano-sized aerosol particles using the SMPS spectrometer, II-237**  
F. O. Arouca\*, N. R. Feitosa, J. R. Coury, Federal University of São Carlos, Brazil

**Study of the behaviour of filter media used in high pressure filtration systems, II-244**  
E. H. Tanabe\*, A. B. N. Brito, E. J. Ricco, J. R. Coury, M. L. Aguiar, University of São Carlos, Brazil

**Determination of adhesion force of organic and inorganic particle in synthetic fiber fabric filters, II-252**  
M. M. Campos, E. H. Tanabe\*, M. L. Aguiar, University of São Carlos, Brazil

**Statistical study on the operational variables influence in the dust cake structure, II-260**  
S. M. S. Rocha, J. J. R. Damasceno\*, C. R. Duarte, Federal University of Uberlândia; M. L. Aguiar, Federal University of São Carlos, Brazil

**Numerical investigations of a content on thallium in the filter ash, II-267**  
Chizhko\*, Moscow Environmental Center, Russia

**Study of the influence of the thickness of the dust cake in drainage of the fluid, II-273**  
S. M. S. Rocha, J. J. R. Damasceno, L. G. M. Vieira, Federal University of Uberlândia; M. L. Aguiar\*, Federal University of São Carlos, Brazil

**Experimental investigations into the effects of post-coating on surface filtration, II-281**  
Q. Zhang\*, E. Schmidt, University of Wuppertal, Germany

**Virtual cyclone as a pre-dust-collector of bag filters, II-287**  
H.-S. Park\*, K. S. Lim, KIER Korea Institute of Energy Research, Korea

**Collection efficiency of fiber filters on the filtration of nano-sized particles from aerosols, II-292**  
N. R. Feitosa, F. O. Arouca\*, J. R. Coury, Federal University of São Carlos, Brazil

**Investigations into the collection of fine dust by plants, II-299**  
H. Mölleken, E. Schmidt, University of Wuppertal, Germany

**L14 Separation Enhancement by Magnetic Forces 11:00 -12:15**

**Selective magnetic separation – A revolution in solid-liquid separation?** I-411  
H. Nirschl\*, C. Wagner, C. Eichholz, University of Karlsruhe, Germany

**DEM-Simulation of magnetic field effects in solid-liquid-separation,** I-419  
C. Eichholz\*, H. Nirschl, University of Karlsruhe, Germany

**Removal of pesticides and benzene from water by using nanomagnetic filtration,** I-427  
S. Alfadul\*, A. Alabdulaa'aly, M. Kahn, KACST King Abdulaziz City for Science and Technology; M. Abdalla, Saud University, Saudi Arabia

**L15 Depth Filtration Analysis II 11:00 -12:15**

**Comparing fibre composition of nonwovens: Filtration performance and sustainability potential,** I-433  
H. H. Kleizen\*, Parker Filtration BV, Delft University of Technology, The Netherlands

**Advanced CFD simulation of filtration processes,** I-440  
M. Dederig, IBS Filtran; O. Iliev\*, Z. Lakdawala, Fraunhofer Institute for Industrial Mathematics ITWM, Germany; R. Ciegis, V. Starikovicus, Vilnius Gediminas Technical University, Lithuania; P. Popov, Texas A&M University, USA

**Filtering of clay colloids in MX-80 d etritis material,** I-445  
T. Richards\*, Chalmers University of Technology; I. Neretnieks, Royal Institute of Technology, Sweden

**G10 Monitoring and Control 11:00 -12:15**

**Monitoring and control of emission of particulate materials in industrial production of alcohol,** II-306  
M. A. Martins Costa, F. de Almeida Filho, M. L. Aguiar, F. Hiromitus, São Paulo State University; E. H. Tanabe\*, Federal University of São Carlos, Brazil

II-313

**Online quality controll in for road tunnel air filters - the new dimension in performance and air quality,** F. Schneider\*, Grimm Aerosoltechnik; E. Deux, FILTRONtec GmbH, Germany

II-319

**Fast online efficiency testing and emission measurement of cleanable filter elements,** G. Lindenthal, Ingenieurbüro für Partikeltechnologie und Umweltmesstechnik; M. Weiß\*, M. Schmidt, Palas® GmbH, Germany

**G11 Filter Media Clogging 11:00 -12:15**

**Clogging of industrial high efficiency particulate air filter in case of fire,** I-325  
F.X. Ouf\*, V.M. Mocho, Institute for Radiological Protection and Nuclear Safety, France

**Effect of air humidity on the clogging of mini-pleated and plane HEPA filters by hygroscopic and non-hygroscopic and particles,** II-333  
A. Joubert\*, J. C. Laborde, L. Bouilloux, IRSN; D. Thomas, S. Callé-Chazelet, Nancy University, France

**Clogging mechanisms involved in the aging of cleanable filter media,** II-341  
J. Schubert\*, G. Mauschitz, W. Höflinger, Vienna University of Technology, Austria

**L16 Separation Enhancement by Physical and Chemical Slurry Treatment 13:15-14:30**

**Removal of colloidal particles from colloidal waste by use of particle immobilization in gel,** I-453  
M. Iwata\*, Suzuka National College of Technology, Japan; M. S. Jami, International Islamic University Malaysia, Malaysia

**Peroxidase catalyzed the removal of phenol from synthetic waste water,** I-461  
K. F. Mossallam\*, F. M. Sultanova, N. A. Salimova, State Oil Academy, Azerbaijan

**Eshidiya industrial wastewater treatment,** I-471  
S. Emeish\*, Al-Balqa' Applied University, Jordan

<b>S5</b>	<b>Survey Lecture</b>	<b>13:15-14:30</b>
<b>Membrane bioreactors in waste water treatment - Status and trends, I-80</b> Prof. Matthias Kraume, Berlin Technical University, Germany; Dr. Anja Drews Oxford University, Great Britain		
<b>G12</b>	<b>Ab- and Adsorption</b>	<b>13:15-14:30</b>
<b>Fugitive dust suppression using water spraying systems with low water consumption in enclosed systems, II-349</b> J. Faschingleitner*, G. Mauschitz, W. Höflinger, Vienna University of Technology, Austria		
<b>Odour reduction by means of textiles – innovative coatings, II-357</b> H. Finger*, F. Schmidt, St. Haep, D. Bathen, Institut für Energie- und Umwelttechnik e. V. (IUTA), Germany		
<b>The truly custom-made adsorbent system, II-364</b> S. Fichtner*, S. Kaemper, J.-M. Giebelhausen, B. Boehringer, A. Arnold, M. Mueller, Blücher GmbH, Adsor-Tech GmbH, Germany		
<b>G13</b>	<b>Nanofibre Filter Media</b>	<b>13:15-14:30</b>
<b>Fabrication and performance evaluation of nano-fibrous filters for filtration of sub-micron aerosols, II-370</b> W. W.-F. Leung*, C.-H. Hung, P.-T. Yuen, The Hong Kong Polytechnic University, P. R. China		
<b>Improved filterefficiency through integrated nanofibers, II-377</b> W. Rupertseder*, T. Ertl, A. Seeberger, A. Jung, IREMA-Filter GmbH, Germany		
<b>Development of nonwoven composites air filters based on micro and nano-fibers, II-383</b> J. Payen*, P. Vroman, M. Lewandowski, A. Perwuelz, Ecole Nationale Supérieure des Arts et Industries Textiles (ENSAIT), France		
<b>L18</b>	<b>Backwashing Filtration Processes</b>	<b>15:00-16:15</b>
<b>RFF – Backwash fibre filter innovation in depth filtration, I-502</b> J. Baumgartinger*, Lenzing Technik GmbH, Austria		
<b>Internal filter for fischer-tropsch wax/catalyst separation, I-506</b> M. A. Khodagholi*, A. A. Rohani, M. R. Hemmati Mahmoudi, Research Institute of Petroleum Industry, Iran		
<b>Filtration and particle analysis for heavily contaminated engine lube oil, I-514</b> F. Gruschwitz*, M. Förster, N. König, MAN Diesel SE; H. Nirschl, H. Anlauf, Karlsruhe University, Germany		
<b>L19</b>	<b>Precoat Filtration</b>	<b>15:00-16:15</b>
<b>Filtration of high density microbial fermentation biomass using BASP biotech filter, I-522</b> V. Patil*, B. Patil, BASP Industries, India; H. Katinger, University of Natural Resources and Applied Life Sciences, Austria		
<b>Filtration system for isolation of decoquinat bio molecules, I-530</b> B. Patil*, V. Patil, BASP Industries, India		
<b>Organic precoat filter aids - Update on current statur and future developments, I-539</b> E. Gerdes*, J. Rettenmaier & Söhne, Germany		
<b>G14</b>	<b>Modelling and Simulation I</b>	<b>15:00-16:15</b>
<b>Simulation of particle separation at woven wire filters, II-391</b> H. Rieger*, H. Sauter, Mahle Filtersysteme GmbH, Germany		
<b>Simulation of fluid flow and particle deposition in three dimensional nonwoven structures, II-399</b> T. Warth*, M. Piesche, University of Stuttgart, Germany		



**Penetration of aerosol particles through polydisperse fibrous filters – II-406**  
**Model and experiment**, A. Podgórski\*, A. Jackiewicz, Warsaw University of Technology, Poland

**G15 Special Filter Media 15:00-16:15**

**Nano filtration media - Challenges of modelling and computer simulation**, II-413  
L. Cheng\*, S. Rief, Fraunhofer Institute for Industrial Mathematics ITWM, Germany

**Investigation of filter service life increased by collating high efficiency media**, II-420  
P. P. Tsai\*, The University of Tennessee, USA

**High-performance spunlace filter media for process air and liquid filtration**, II-429  
V. Lorentz\*, Norafin GmbH, Switzerland

**L20 Filter Media Development and Application 16:45-18:00**

**Influence of filter cloth behaviour on the layout of cake forming filters**, I-542  
E. Ehrfeld, Bokela GmbH, Germany

**Comparison of pore size distribution of non-woven fibrous filter evaluated by computer simulation with the distributions measured by DFM and microscopic observation**, I-550  
K. Matsumoto\*, K. Nakamura, Yokohama National University; T. Yunoki, Tritec Corporation, Japan

**Continuous welding and simultaneous edge sealing of pleated filter media**, I-557  
A. Korz\*, K. Herzer, Textile Fusion Technologies GmbH, T. Westermann, Pfaff Industriesysteme und Maschinen AG, Germany

**L21 Selective Separation and Classification 16:45-18:00**

**"Bubble bobble" in the lauter tun – A new way for mash separation in the brewhouse**, I-561  
J. Tippmann\*, H. Scheuren, J. Voigt, K. Sommer, Munich University, Germany

**Performance of dynamic filtration in particle classification**, I-569  
L. Steinke\*, Y. Taamneh, S. Ripperger, University of Kaiserslautern, Germany

**Modeling sieving filtration using simple network models**, I-577  
U. Beuscher\*, W. L. Gore & Associates Inc., USA

**G16 Modelling and Simulation II 16:45-18:00**

**Influence of an oscillating flow on single fibre efficiency**, II-435  
P. Kopf\*, M. Piesche, University of Stuttgart, Germany

**Deposition of nanoparticles in the composites of nano- and micro-sized fibers. The electrostatic effects**, II-443  
L. Gradon\*, Warsaw University of Technology, Poland

**Effect of slip flow on the pressure drop in fibrous filters**, II-449  
Z. Bin, Tongji University, P.R. China; V. Bertola, The University of Edinburgh, UK; E. Cafaro, P. Tronville\*, Politecnico di Torino, Italy

**G17 Mist and Droplet Separation 16:45-18:00**

**Collection of oil droplets from pyrolysis gases by an electrostatic precipitator**, II-457  
A. Bologna\*, H.-R. Paur, H. Seifert, K. Woletz, Forschungszentrum Karlsruhe, Germany

**Measurements of metal working fluid mist emissions at high concentrations**, II-465  
T. Laminger\*, W. Höflinger, Vienna University of Technology, Austria

**A model for steady-state oil transport and saturation in a mist filter**, II-473  
D. Kampa\*, J. Meyer, B. Mullins, G. Kasper, Karlsruhe University, Germany

The Programme lists countries and regions and is subject to amendments. Errors and omissions expected.

# SESSION CHAIRMEN

## TUESDAY, OCTOBER 13, 2009

<b>S1 - Survey Lecture: Advances in Pore Structure Evaluation by Porometry</b>	13:15-14:30 h
Chairman: Harald Anlauf	
<b>L1 - Sedimentation Analysis in the Gravity Field</b>	13:15-14:30 h
Chairman: Dietmar Lerche	
<b>M1 - Waste Water Treatment</b>	13:15-14:30 h
Chairman: Siegfried Ripperger	
<b>G1 - Air Filter I</b>	13:15-14:30 h
Chairman: Thomas Caesar	
<b>S2 - Survey Lecture: Membrane Pore Characterization Techniques – Status Quo and Future Development</b>	15:00-16:15 h
Chairman: Richard Wakeman	
<b>L2 - Sedimentation Analysis in the Centrifugal Field</b>	15:00-16:15 h
Chairman: Dietmar Lerche	
<b>M2 - Produced Water Treatment</b>	15:00-16:15 h
Chairman: Steve Tarleton	
<b>G2 - Air Filter II</b>	15:00-16:15 h
Chairman: Thomas Caesar	
<b>S3 - Towards predicting filtration and separation - Progress and challenges</b>	16:45-18:00 h
Chairman: Siegfried Ripperger	
<b>L3 - Sedimentation and Flotation for Sorting</b>	16:45-18:00 h
Chairman: Harald Anlauf	
<b>M3 - Combined Processes</b>	16:45-18:00 h
Chairman: Jaroslav Pridal	
<b>G3 - Surface Filtration</b>	16:45-18:00 h
Chairman: Wilhelm Höflinger	

## WEDNESDAY, OCTOBER 14, 2009

<b>L4 - Sedimentation in Centrifuges/Hydrocyclones</b>	8:30-9:45 h
Chairman: Michael Kopf	
<b>L5 - Poster Session I</b>	8:30-9:45 h
Chairman: Graham Rideal	
<b>M4 - Poster Session I</b>	8:30-9:45 h
Chairman: Kuo-Lun Tung	
<b>G4 - Poster Session I</b>	8:30-9:45 h
Chairman: Gerd Mauschitz	
<b>L6 - Filter Media Characterization</b>	11:00-12:15 h
Chairman: Christophe Peuchot	
<b>L7 - Cake Filtration Analysis I</b>	11:00-12:15 h
Chairman: Urs Peuker	
<b>M5 - Deposition Control</b>	11:00-12:15 h
Chairman: Kuo-Lun Tung	
<b>G5 - Industrial Gas Cleaning</b>	11:00-12:15 h
Chairman: Takeshi Yoneda	
<b>S4 - Development history and system integration aspects of diesel particle filters in commercial vehicles</b>	13:15-14:30 h
Chairman: Eberhard Schmidt	
<b>L8 - Cake Filtration Analysis II</b>	13:15-14:30 h
Chairman: Peter Scales	
<b>M6 - Membrane Fouling</b>	13:15-14:30 h
Chairman: Thomas Peters	
<b>G6 - Filter Test Systems I</b>	13:15-14:30 h
Chairman: Gerhard Kasper	
<b>L17 - Depth Filtration Processes</b>	15:00-16:15 h
Chairman: Hermanes Kleizen	

<b>L9 - Cake Filtration Processes I</b> Chairman: Gernot Krammer	15:00-16:15 h
<b>M7 - Modelling and Simulation</b> Chairman: Thomas Peters	15:00-16:15 h
<b>G7 - Filter Test Systems II</b> Chairman: Gerhard Kasper	15:00-16:15 h
<b>L10 - Depth Filtration Analysis I</b> Chairman: Hermann Nirschl	16:45-18:00 h
<b>L11 - Cake Filtration Processes II</b> Chairman: Marja Oja	16:45-18:00 h
<b>M8 - Special Membranes</b> Chairman: Thomas Peters	16:45-18:00 h
<b>G8 - Hot Gas Cleaning</b> Chairman: Achim Dittler	16:45-18:00 h

## THURSDAY, OCTOBER 15, 2009

<b>L12 - Washing of Particles and Cleaning of Media</b> Chairman: Urs Peuker	8:30-9:45 h
<b>L13 - Poster Session II</b> Chairman: Hans Theliander	8:30-9:45 h
<b>M9 - Poster Session II</b> Chairman: Kuo-Jen Hwang	8:30-9:45 h
<b>G9 - Poster Session II</b> Chairman: Hans-Joachim Schmid	8:30-9:45 h
<b>L14 - Separation Enhancement by Magnetic Forces</b> Chairman: Karsten Keller	11:00-12:15 h
<b>L15 - Depth Filtration Analysis II</b> Chairman: Eugène Vorobiev	11:00-12:15 h
<b>G10 - Monitoring and Control</b> Chairman: Hans-Joachim Schmid	11:00-12:15 h
<b>G11 - Filter Media Clogging</b> Chairman: Markus Lehner	11:00-12:15 h
<b>L16 - Separation Enhancement by Physical and Chemical Slurry Treatment</b> Chairman: Eiji Iritani	13:15-14:30 h
<b>S5 - Membrane bioreactors in waste water treatment - Status and trends</b> Chairman: Eberhard Schmidt	13:15-14:30 h
<b>G12 - Ab- and Adsorption</b> Chairman: Markus Lehner	13:15-14:30 h
<b>G13 - Nanofibre Filter Media</b> Chairman: Gernot Krammer	13:15-14:30 h
<b>L18 - Backwashing Filtration Processes</b> Chairman: Martin Lehmann	15:00-16:15 h
<b>L19 - Precoat Filtration</b> Chairman: Christophe Peuchot	15:00-16:15 h
<b>G14 - Modelling and Simulation I</b> Chairman: Gernot Krammer	15:00-16:15 h
<b>G15 - Special Filter Media</b> Chairman: Wallace Leung	15:00-16:15 h
<b>L20 - Filter Media Development and Application</b> Chairman: Reinhard Bott	16:45-18:00 h
<b>L21 - Selective Separation and Classification</b> Chairman: Harald Anlauf	16:45-18:00 h
<b>G16 - Modelling and Simulation II</b> Chairman: Martin Lehmann	16:45-18:00 h
<b>G17 - Mist and Droplet Separation</b> Chairman: Gerd Mauschwitz	16:45-18:00 h

# FILTRATION PERFORMANCE DOWN TO NANOPARTICLES

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## ABSTRACT

Man-made nanoparticles escaping into the environment and potentially causing adverse health effects are a serious concern for all interested parties. However, safety issues are currently not fully understood and assessed.

We analyze the nanoparticle removal performance of three different wet-laid fibrous filter media used in both general ventilation and contamination control applications. Media characteristics were evaluated experimentally and their performance measured on a small scale test rig in the 0.1-3.0  $\mu\text{m}$  size range. The data were extrapolated down to 10 nm according to the most recent expressions available for Brownian diffusion. The calculations show that the efficiency at 10 nm is clearly higher than the one at 1000 nm and far higher than at MPPS for the three different media considered here.

In the second part of the paper we analyze the peculiarities of fractional efficiency measurements down to particles with size of few nanometers. Special attention is devoted to the phenomena influencing the test rig design and the measurement procedure. We discuss the capabilities and the cost of the instrumentation currently available on the market for measuring the data needed and for widening the particle size range of the most common standardized current test methods.

## KEYWORDS

Brownian Motion, Fibrous Filter, Filter Test, Filtration Performance, Fractional Efficiency, Nanofiltration, Nanoparticles, SMPS

## 1. Introduction

Nanoaerosols are made of engineered nanomaterials, nanoparticles and nanostructures having one or more dimensions of the order of 100 nm or less. There is much interest in nanosized materials because, at the nano-scale, their physical properties are quite different from the properties of the bulk material from which they are made. However, it has been established for many years that exposure to particles, including nanoparticles, can cause illness in individuals or exposed populations.

The potential risks to health from inhalation of nanoparticles are due to several factors.

- Nanoparticles can reach parts of biological systems which are not normally accessible by larger particles, e.g. the possibility of passing directly from the lungs into the blood stream and to all of the organs, or even through deposition in the nose, directly to the brain (translocation).
- Nanoparticles have, for particle collections with equal masses, much higher surface area than larger particles. If surface area is linked to toxicity this implies potentially higher toxic effects.

- The reduction in size has been shown to relate to increased solubility for some nanomaterials. This effect might lead to increased bioavailability of materials which are considered to be insoluble at larger particle sizes.
- Since nanomaterials and nanoparticles have new and different properties from larger particles of the same material, altered chemical and/or physical properties might be expected to be accompanied by altered biological properties, some of which could imply increased toxicity.
- Some high aspect ratio nanoparticles can be inhaled and enter the alveolar region of the lung and are not easily removed. Their physical dimensions inhibit their removal by lung clearance mechanisms and they do not dissolve in the lung fluids. Hence they remain in the lung for a long period of time, causing inflammation and ultimately disease.

The above issues indicate that more needs to be done to assess the potential risks associated with nanomaterials. In the meantime a cautious approach should be taken in their handling and disposal. The risk depends on the dose of the particles in the organ where disease can occur, and the toxicity of nanoparticles. Dose is hard to assess directly, but can be obtained from the exposure to nanoparticles, i.e. the combination of particle concentration in the air which a person breathes in and the duration of the exposure.

If there are no nanoparticles in the air, no dose will accumulate and, despite the potential toxicity of the particles, there will be no risk to health. Therefore in many working atmospheres the preferred method to control risks is a strong effort to mitigate, manage or reduce exposure. Fibrous air filters can be very effective in removing nanoparticles from air streams and may play an essential role in this strategy. However, no standardized test method for measuring the efficiency of filters in removing particles below the 100 nm size is currently available. Test methods for HEPA and ULPA filters measure the removal efficiency corresponding to the most penetrating particle size (MPPS) or close to it, i.e. usually between 100 nm and 200 nm. The most widely used test methods for general ventilation filters supply no data in the defined nanoparticle range, since they test only down to 200 nm (EN779:2002) and to 300 nm (ANSI/ASHRAE 52.2-2007). The absence of references supplying the measured performance of air cleaning devices in removing nanoparticles makes it difficult to draft any regulation for handling nanoparticles safely.

Even if current research shows that the theory of particle removal by fibrous air filters is valid down to 3 nm, it is reasonable to expect that some factual evidence will be requested by regulatory authorities to trust air filters as a valid mean to minimize exposure risks.

The present paper aims at answering the basic question: should the scope of standardized test methods be widened to include the nanometer size range, or is the information obtained with already available test methods enough for nanoparticle exposure assessment?

We divide this problem in two parts. The first one describes the experimental characterization and performance of three wet-laid fiber glass media and the calculated extrapolation of their efficiencies down to 10 nm according to the most recent theory. The second part describes the peculiarities of efficiency measurements down to nanoparticle size, the phenomena influencing test rig design and measurement procedures, and the costs and capabilities of the instrumentation currently available on the market for measuring the data needed.

## 2. Measurement of filter media properties

Three types (F6, F8 and H13) of media samples were cut from rolls supplied by the manufacturer, using randomized locations down the length and width of the roll. Variances of measured parameters were found to be small, but random sample selection avoids any systematic parameter biases.

More than 20 scanning-electron microscope (SEM) images were taken of each of the three media types. Approximately 1000x magnification allowed the least diameters to be seen and measured, while preserving enough area of the filter media to allow representative sampling. Again, a randomization technique was used to eliminate bias and simultaneously weight the diameters by the length of each diameter interval present. Parallel lines were scratched across the SEM photographs at randomly-located positions. Wherever these lines intersected a fiber, the width of the fiber was measured in the direction normal to the fiber axis. A special scale was prepared to allow rapid sorting of the fiber diameters at each line/fiber intersection. Several hundred intersections (hence fiber diameters) were included from each media type.

Table 1 - Basic parameters of media tested and values obtained from calculations

Sample	Units	Sample Data		
		F6	F8	H13
Rated velocity ( $v_r$ )	m/s	0.0617	0.0617	0.0231
Geometric mean diam. ( $D_g$ )	$\mu\text{m}$	4.110	1.561	0.775
Geometric std. dev. ( $\sigma_g$ )	-	1.921	2.141	2.198
Fractional solids ( $\alpha$ )	-	0.076	0.081	0.092
Pressure drop ( $\Delta p$ )	Pa	10	42	121
Effective fiber diameter ( $d_f$ )	$\mu\text{m}$	8.179	3.618	1.512
Measured efficiency at 100 nm	%	10.8	44.9	99.3
Measured efficiency at 1000 nm	%	32.0	93.3	99.5
Calculated efficiency at 10 nm	%	81.8	99.2	100
Calculated efficiency at 100 nm	%	23.9	55.4	99.6
Calculated efficiency at 1000 nm	%	20.2	86.9	99.99986

The fiber-size data were fitted to the log-normal distribution to obtain the geometric mean diameter and standard deviation for the fibers in each media.

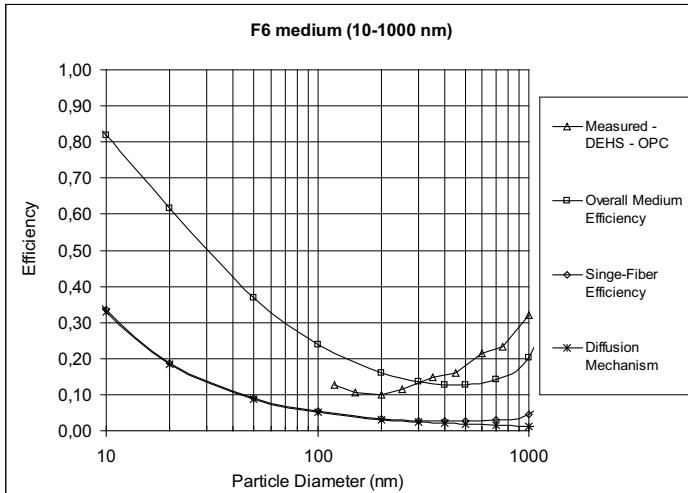
The (volume) fractional solids in fibrous media is:

$$\alpha = \frac{M_m \left[ \frac{\eta}{\rho_{fiber}} + \frac{1-\eta}{\rho_{binder}} \right]}{L} \quad (1)$$

Fibers of glassfiber filter media have a melting point above the ignition temperature of organic binders. Baking at 500 °C burns the binder out of the media. Weighing media samples before and after baking thus allows the calculation of fiber mass fraction ( $\eta$ ). Values of 2450 kg/m<sup>3</sup> for fiber density and 1000 kg/m<sup>3</sup> for binder density were thought reasonable. The thickness of the filter medium was measured, along with the compression function (the relation between the thickness of the medium and the pressure drop across it) using the technique described in Rivers (2000).

The resistance-vs.-velocity characteristics of flat sheets of media were measured in a test duct which exposed an area of media 300 mm by 300 mm. Complete filter cells from these grades of filter media contain enough media area to reduce the average media velocity below 0.1 m/s and hence the expected resistance of a single sheet of medium to very low, difficult-to-measure levels. Accuracy of resistance measurement

was improved by measuring the resistance of a 10-sheet stack. In the range of interest, resistance is essentially proportional to velocity. Fractional efficiency curves for the three types of media were measured in the same test rig using DEHS synthetic aerosol and an optical particle spectrometer able to provide data in the 100-7500 nm size range.



**Figure 1 – Fractional efficiency of F6 medium**

The single fiber efficiencies due to the deposition mechanisms by which an aerosol particle can be deposited onto a fiber in a filter were computed using the expressions supplied by Hinds (1999). We considered mechanical collection mechanisms only. The common expression for the diffusion mechanism:

$$E_D = 2 \cdot \frac{1}{\sqrt[3]{Pe^2}} = 2 \cdot \left( \frac{d_f \cdot U_0}{k \cdot T \cdot C_c} \right)^{-\frac{2}{3}} \quad (2)$$

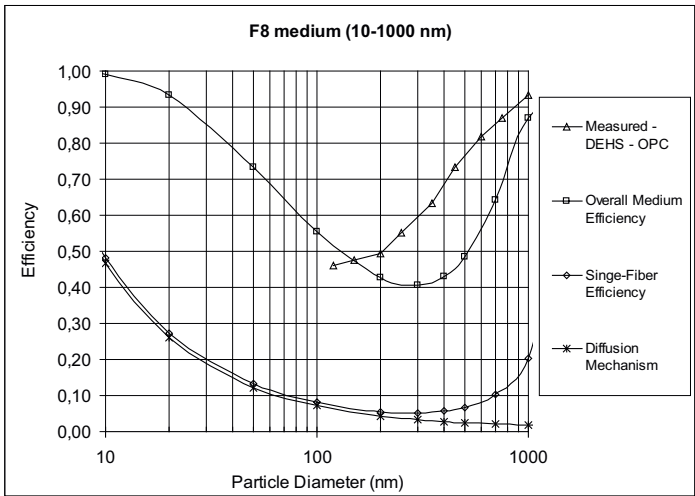
was replaced by the more recent expression proposed by Wang (2007):

$$E_D = 0.84 \cdot Pe^{-0.43} \quad (3)$$

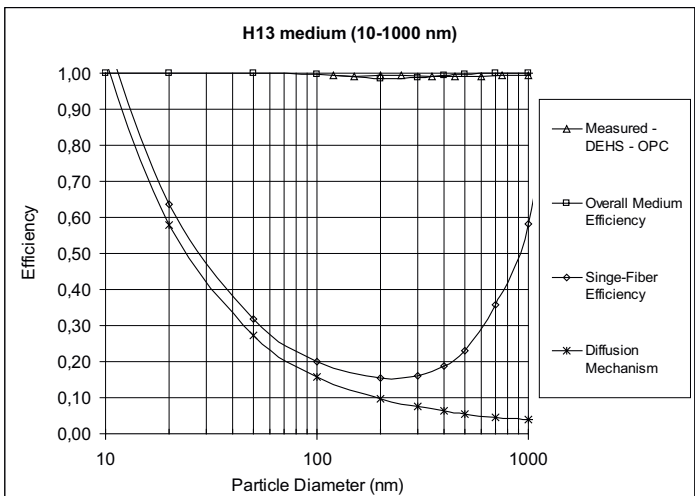
Expression (3) yields a lower efficiency due to the diffusion mechanism than expression (2).

Diffusion is the only important mechanism for particles below 0.2  $\mu\text{m}$ , but is of decreasing importance for particles above that size. It is well known that the competing deposition mechanisms are most effective in different size ranges. Hence all filters have a particle size that gives minimum efficiency, usually in the range from 50 to 500 nm. The single fiber approach takes the distribution of the fiber sizes into account indirectly. Since the flow field and collection efficiency associated with each fiber size are influenced by the presence of fibers of other sizes, as a practical means, the effective fiber diameter  $d_f$ , based on pressure drop measurements, is used as an approximation for these calculations. Moreover the fibers may be clumped together and the medium may not be uniform. The use of the effective fiber diameter avoids this problem but it turns out to be very different from the geometric

mean diameter of the log-normal distribution describing the actual physical appearance of the medium itself. The results of the measurements and of the calculations are shown in Figures 1, 2 and 3. Media F6 and F8 are meant for use in general-ventilation applications, while H13 is intended for use in contamination control applications.



**Figure 2 - Fractional efficiency of F8 medium**



**Figure 3 - Fractional efficiency of H13 medium**

### 3. Testing filters at nanometer particle diameters

Current filter testing and standardization practices rely on the higher filter efficiencies predicted at nano-sizes to make claims that filters perform better than specified filter class at nanoparticle sizes. The work reported in this paper has confirmed the predicted higher efficiency at nano-sizes for several filter media. However, building



commercial testing systems for filter performance measurements on nano-size particles poses new challenges. Building commercial test systems involves scaling up the aerosol generating and measurement systems discussed above to handle air flow rates about  $1 \text{ m}^3/\text{s}$ . The general requirements are discussed below.

Particle measurements: Any filter efficiency test system must have the ability to measure concentrations of particles within specific particle size ranges over the entire size range of interest. High quality data requires that the measurement devices have nearly the same response over the range of interest. Even for current measurement systems, measurements over 2 orders of magnitude in particle size and concentration can become a challenge for commercially available measurement devices. When one considers measurements of particles from 5 nm to 10,000 nm, the challenge may become nearly impossible. The use of more than one measurement device may be required to provide data over the entire size range.

Particle size specific concentrations can be measured either by using particle spectrometers or by size classifiers and particle detectors. Current filter test systems mostly use optical particle spectrometers (OPC). These devices provide particle counts in several particle size intervals. Several commercial devices allow users to select the size intervals. Alternately, particles can be classified according to their size and the classified particles counted by detectors. Electrostatic classification of particles and counting by condensation particle counters (CPC) or electrometers are the most common combinations used. In this case, particles are classified according to their electric mobility, which is directly related to particle size. Classification by diffusion is also a technique for particle size specific measurements, although not commonly used. Where one requires measurement for sizes for which calibrated PSL particles are available, it is also common to use these particles without classification. The advantages of the OPCs are their relatively low cost and their ease of use. Further, most standards relevant to particle contamination and filtration are based on OPC measurements, making data from these devices readily comparable. Their main disadvantage is their detection limitations at smaller sizes. Although the size range of many commercial counters extend down to 100 nm, the response of optical scattering devices drops off significantly for particles under 150 nm. Thus it will be impractical to use these devices for determining filter performance for particles in the sub 100 nm sizes.

Electrostatic classification is the most common method used for particle research and calibration, especially for sub-micrometer particles. Since the size classification can be derived for a given geometry of the instrument, it is also referred to as a reference method. Since the classification is according to the electric mobility of a particle, larger particles carrying multiple units of charge will have double the mobility and, hence, could be classified with singly charged particles with half their size. However, nearly all sub micron particles are singly charged and will be classified according to their size. Commercial electrostatic classifiers account for the effect of multiple charge based on the charge distribution on the aerosol classified. The most common detector for the classified particles is the CPC. Since it can detect nearly all the particles as small as a few nanometers, it is also often called a reference counter. This combination of electrostatic classification and CPC is currently commercially available and is used extensively in nanoparticle research. An alternate means of detecting classified particles is to use an electrometer to collect the particles and measuring the current. Modern electrometers are often more compact and robust than CPCs.

In diffusion classification, penetration of particles through a series of diffusion tubes or screens is measured using a detector, such as the CPC, and the size specific

concentration determined from the data. Although diffusion classification does not depend on the charge on the particle, since particles of all sizes will penetrate to different extent through the diffusion element, the classified aerosol is somewhat poly-disperse. Hence these measurements require extensive data reduction to generate particle size specific concentration. Perhaps for this reason they are not in common use outside of particle research.

Further, as discussed earlier, the efficiency of even lower grades of media can be quite high for nano-size particles. Hence, the downstream counts for these particle sizes will be quite low for many filters, requiring long sample times to obtain statistically valid counts with any of the devices discussed above. Since time is invaluable in product manufacturing, instruments with large sample flows may be needed for nanoparticle measurements.

Particle Losses: Loss of particles during sampling and transport is always a concern since such losses can introduce unknown errors in the data. In current filter testing practice, the focus has been primarily on large particles, typically greater than 2000 nm. Since large particles are lost in sharp bends in tubing, valves, and constrictions, current testing system designs minimize these elements in sampling lines. Where practical, sample lines upstream and downstream of filters are made equal in length in an attempt to equalize the losses, if any, and minimize errors in the computation of filter efficiency. Smaller particles are considered to behave like gases and their losses are generally ignored. However, the losses due to Brownian Diffusion increase significantly for nano-sizes. Much like the higher capture efficiency for nanoparticles in filters, nanoparticles are also more readily "captured" in the sample lines. Hence nanoparticle sampling will require more attention to making upstream and downstream sample lines exactly equal. In addition, a two-stage sampling may be needed if the instrument sample flow rate is low, as is common in many CPCs. In this case, a first stage sample is taken at a high sample flow rate, minimizing the residence time and hence the losses in the sample lines. Then the detection instrument with its much smaller flow rate can sample from this larger first stage sample using very short sampling tubes.

Challenge Particles: The common practice of using poly-dispersed aerosols with particle spectrometers or classifiers will be adequate, in principle, for measurements at nano-sizes. However, as noted above, because of the higher filter efficiency for nanoparticles, poly-disperse aerosols with large concentrations in the nano-sizes are preferred. Vapor condensation generators are best suited for this purpose and are commercially available. Alternately, atomizing solutions of the aerosol material and flashing the solvent will yield a large concentration of poly-dispersed nanoparticles of the solute material. This technique is used in the ASHRAE 52.2 standard for generating nano-size KCl aerosol for neutralizing electrostatic charge in filters.

Other factors: In general, all other good testing practices described in many of the national standards are readily applicable for testing filters down to nano-sizes. These include prescription for system validation, dilution of upstream concentrations, and aerosol neutralization as well as general system configuration.

In summary, the established practices prescribed by the prevailing standards offer a good starting point for testing filter efficiency for nanoparticles. Additional precautions in sampling and measurements are required to obtain valid results. The main upgrade for these systems will be the particle measurement devices, and the design of the sample lines to minimize losses. Further, the need for statistically valid counts may require longer and more expensive testing times. Overall, it is our opinion, based on current market for these particle instruments, that filter efficiency measurements for nanoparticles will add over 30% to the cost of the system.

## Conclusions

Our analysis suggests that the additional cost for extending the measuring range down to a few nanometers is not fully justified by the further data obtainable in this way. However, the calculated efficiency depends on many parameters and the accurate characterization of filter media is rather difficult and time consuming. At the same time the technology for evaluating the performance of air filters down to a few nanometers is available on the market and reasonably priced. Interested parties may be willing to pursue efficiency measurements down to a few nanometers, following the nature of air filtration which is mainly experimental.

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