

Zhou Mingxing,
Meng Zhuo*,
Sun Yize,
Ye Zhibiao

Application of Power Ultrasound to Chemical Dissolution for Quantitative Analysis of Cotton and Polyester Blended Fabrics

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Donghua University,
College of Mechanical Engineering,
Shanghai, China
*E-mail: mz@dhu.edu.cn

Abstract

In this paper, the feasibility of applying ultrasonic technology to quantitative analysis of cotton and polyester blended fabrics by the chemical dissolution method is explored. Four varieties of blends with different cotton contents were employed and dissolved in sulfuric acid solution with ultrasound assistance. Although three different frequencies: 20, 28 and 40 kHz were adopted, the experimental results indicate that there is no difference between them. For some fabrics that cannot be untwisted into yarns, ultrasonic technology has good experimental results while only needing a fabric form prepared from a small, long strip. By comparison with the traditional mechanical oscillation method stated in ISO 1833-11:2006, the more meaningful role of ultrasonic lies in the fact that the experimental time and usage of solvent can be reduced by half, which is in agreement with resource savings and environmental protection. Broadly speaking, the study demonstrated that ultrasound assisted chemical dissolution for the quantitative analysis of cotton and polyester blended fabric is feasible.

Key words: power ultrasound, chemical dissolution, quantitative analysis, cotton, polyester, blended fabrics.

Introduction

The fibre content of blended fabric has an important influence on fabric performance, style and production price, which also is a vital identification for the textile trade. Physical separation, chemical dissolution and the microscopic method have been the most commonly used quantitative analysis methods in the past decade. Especially chemical dissolution, which has been used for a few decades, is accurate and robust in textile quantitative analysis [1]. After dissolving one component in an appropriate chemical reagent, the content was calculated by precise weighing. One by one, all the fibre contents were obtained with different chemical solvents. However, a large amount of laboratory technicians were needed, and they had to come in direct contact with the chemical reagent, which would cause health issues [2]. Because of a lack of optimal control, the excessive use of auxiliary chemicals has created environmental issues [3]. What is more, the applying of mechanical agitation in the process of chemical dissolving was inconvenient for the mechanization and automation of textile quantitative analysis. As there was an urgency to improve testing efficiency, a more rapid and efficient method was required.

The power ultrasound technique, with the advantages of penetrating the isolation layer on the fibre and promoting sol-

vent diffusion, has long been studied as an alternative to the conventional method during some textile process such as dyeing [4], bleaching [5] and washing [6]. Its remarkable benefits come from heating and cavitations. The formation and rapid collapse of micro-bubbles formed by ultrasonic waves are generally considered responsible for most of ultrasound's physical and chemical effects. Dyeing with intermittent ultrasound was confirmed to be more effective than making full use of ultrasound energy continuously [7]. Compared with the conventional process, raw wool washing with ultrasound can lower the process temperature and provide the same results [8]. In addition, scouring wool in small batches using ultrasonic techniques reduced water and detergent consumption and shortened the time of scouring without damage and entanglements [9]. There were also researches that indicated that the powerful agitation of the liquid border layer caused by cavitation could increase the reaction rate of enzymatic desizing [10, 11]. From the investigations above, it can be seen that ultrasound has been employed to fabric wet processing with good effects. However, few applications of ultrasound for quantitative analysis of blended fabrics have been found.

In this paper, quantitative analysis of blended fabrics by the chemical dissolution method assisted by ultrasound is carried out, and by comparison with the tra-

ditional mechanical oscillation method, the advantages of the ultrasonic technique are verified. Considering the universality, blended fabrics composed of cotton and polyester were used as the experimental material, where cotton was selected as the component to be dissolved. As in the process of the traditional chemical dissolution method, according to ISO 1833-11:2006, blended fabrics were dried and weighed in the beginning. After dissolution, the fibres removed from the initial specimen were determined by the loss of weight measured by a precision electronic balance. The advantages of applying ultrasound in chemical dissolution for quantitative analysis of blended fabrics are embodied by the prospect of lowering the dissolving time, reaction temperature, and amount of solvent usage etc.

Materials and methods

Materials

Four types of cotton and polyester blended fabrics with different cotton contents were employed in this study. The cotton content of each fabric, measured by the traditional method, is considered as the standard value. These four blended fabrics shown in **Table 1** are commercial samples and are provided by the Shanghai Textile Research Institute.

According to ISO 1833-11:2006, 75% of the mass fraction of the sulfuric acid solution is chosen as the solvent for cot-

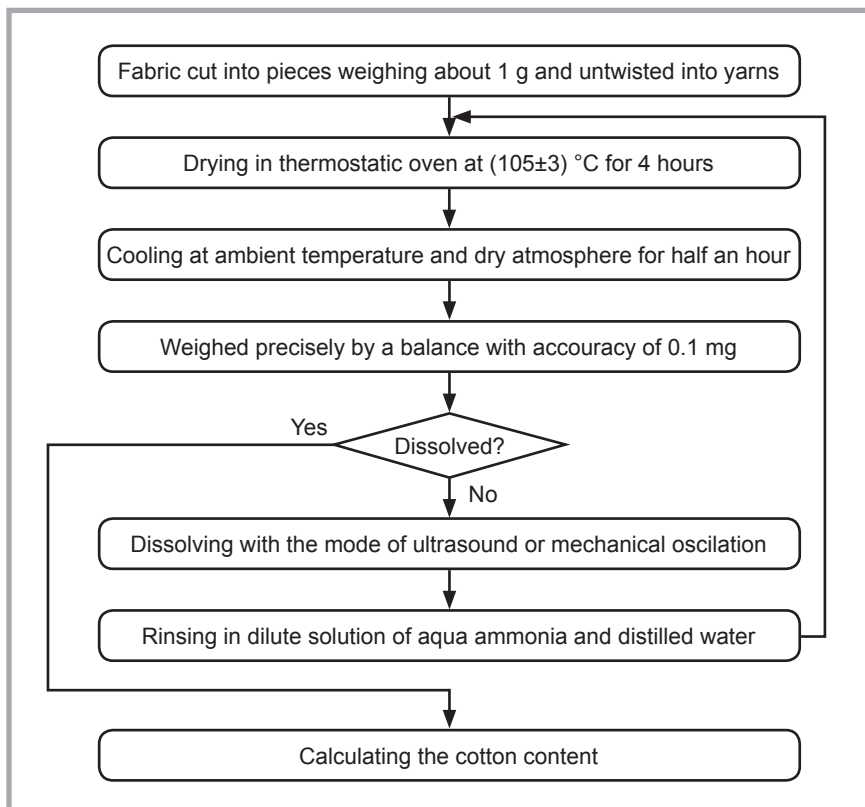


Figure 1. Flow chart of experimental procedure.

ton fibre, while dilute aqua ammonia is applied as neutralisation solution, and distilled water is used for rinsing. All of these solutions are also supplied by the Shanghai Textile Research Institute.

Experimental procedure

BL6-180A (BiLon Ltd., Shanghai, China) ultrasonic cleaners with a frequency of 20, 28 and 40 kHz, respectively, are used as a source of mechanical action for auxiliary dissolving. The ultrasonic cleaners with a thermo-controlled bath of capacity 6l can reach a temperature up to 80 °C.

Blended fibres are submerged in a glass beaker containing an exact amount of solvent, where the dissolving process is carried out. The glass beaker is immersed in a water bath of the ultrasonic cleaner at a certain temperature. After dissolution, the remaining fibres are rinsed with neutralisation solution and distilled water, then vacuum filtered to a sand core crucible. Next the sand core crucible with insoluble fibres is dried in a thermostat-

ic drying oven (HOC-9030A, Hengqian Technology Ltd, Shanghai) for more than 4 hours (no more than 16 hours), maintaining a temperature of (105±3) °C. After drying, it is cooled in a drying vessel at ambient temperature for half an hour, and finally weighed by a BSA124S-CW Sartorius, with an accuracy of 0.1 mg.

For clarity, the whole process of experimentation is shown in *Figure 1*, from which it can be seen that the difference between the ultrasonic method and traditional mechanical oscillation method is only in the dissolving procedure.

Experimental results evaluation

The content of the dissolved component is evaluated by the weight loss after dissolving, which is expressed by the following *Equation (1)*:

$$WL = \frac{W_b - W_a}{W_b} \times 100\% \quad (1)$$

Where, WL represents the content of dissolved fibre, W_b and W_a are the weights of the blended fabric before and after dissolution, respectively.

Table 1. Blended fabric used in the experiment.

Fabric specimen	FS1	FS2	FS3	FS4
Cotton content, %	71.7	63.7	76.4	77.5

According to the commercial test standard, two data obtained by the same condition are acceptable when the difference is within +/-1%. The results obtained by the two methods are compared, from which the effects of ultrasound are reflected.

Results and discussion

Comparison of ultrasound frequencies

In textile wet processing, ultrasound frequencies commonly used are within the range of 20 kHz~50 kHz [12, 13]. Three different frequencies: 20, 28 and 40 kHz are employed in this research. For evaluation of the difference between the three frequencies applied to the chemical dissolution of blended fabrics, other experimental conditions such as the dissolving time, experimental temperature and liquor ratio are fixed. Fabric specimen FS1 was selected as a test sample, which was cut into 30 pieces, with each weighing about 1 g, and untwisted into yarns. The 30 specimens were divided into three groups equally corresponding to the three ultrasound frequencies and dissolved at 50 °C for 30 minutes at a liquor ratio of 100 (ml/g).

From *Figure 2*, it can be seen that data obtained by the three ultrasound frequencies are acceptable as the biggest difference is no more than 1%. With the sign test, the three data sets are analysed two by two at a 5% significance level. Results showed that there is no difference between the three ultrasound frequencies when applied to quantitative analysis by chemical dissolution.

Effect of dissolving time

30 minutes was applied at the frequency given above, which is much less than the standard value of 60 minutes according to ISO 1833-11:2006. For further research of the influence of time, blended fabric FS2 was dissolved at 50 °C at a liquor ratio of 100 (ml/g) and ultrasound of 20 kHz. The blended fabric was cut into 20 pieces, each weighing about 1 g, and then untwisted into yarns. The 20 fabric pieces were divided into four groups equally for dissolving for 20, 25, 30 and 35 minutes, respectively.

From *Figure 3*, it can be seen that cotton is dissolved almost completely even after 20 minutes. However, the error bar shows that data fluctuation is a little wider than for the other three dissolving times, which means that dissolving for 20 minutes is unstable. For 25 minutes

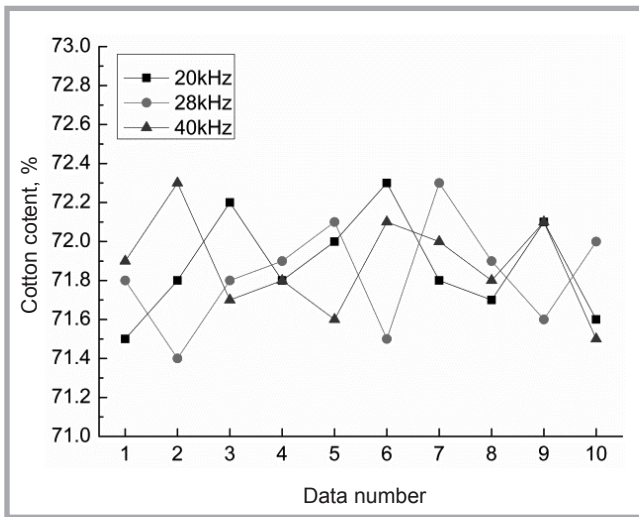


Figure 2. Results of blended fabric dissolved under three ultrasound frequencies.

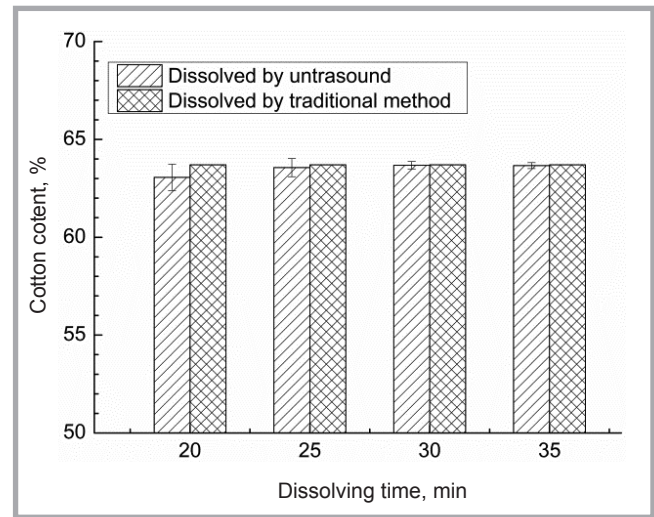


Figure 3. Results of blended fabric FS2 dissolved with different experimental times.

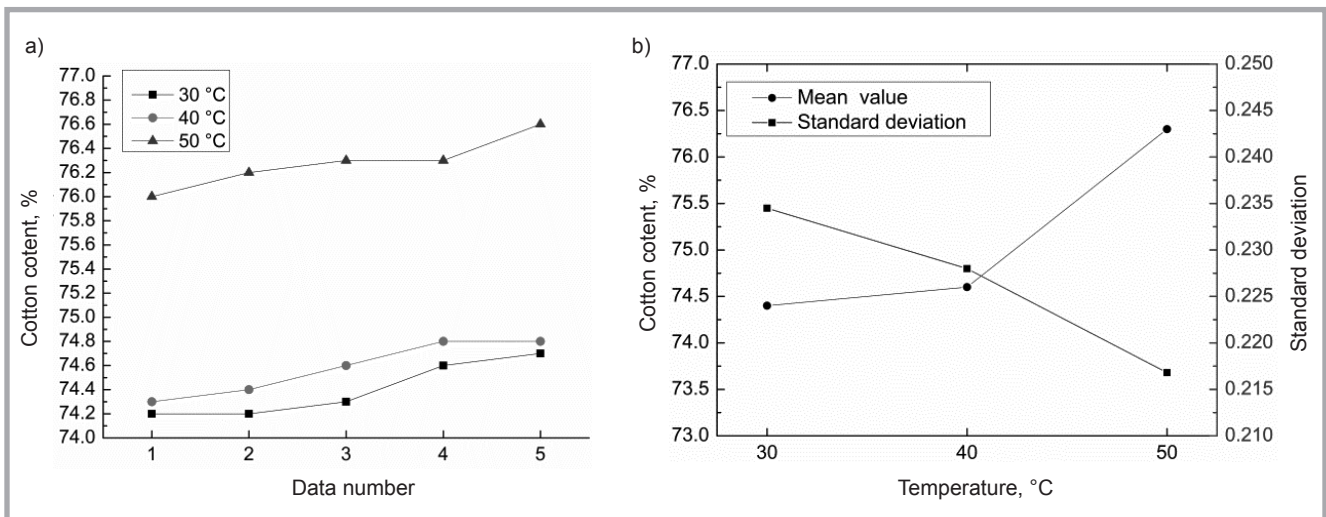


Figure 4. Results of blended fabric FS3 dissolved at three different temperatures: a) cotton contents obtained at each temperature, b) mean value and standard deviation at each temperature.

dissolving, the mean value is closer to the standard value and the error bar is smaller. When dissolving for 30 minutes, both the mean value and error bar suggest that cotton is dissolved completely and steadily, which can be verified by the results for 35 minutes dissolving. Hence cotton dissolving for 30 minutes is considered optimum for quantitative analysis of blended fabrics by ultrasound.

Influence of experimental temperature

According to ISO 1833-11:2006, the standard temperature was 50 °C for cotton dissolution. On the one hand, higher chemical reaction temperature goes against improvement of ultrasonic efficiency. On the other hand, energy conservation needs a lower dissolution temperature. Hence blended fabric FS3 is used for investigating the influence of

temperature on quantitative analysis with an ultrasound of 20 kHz. The blended fabric is cut into 15 pieces, each weighing about 1 g, and then untwisted into yarns. The 15 fabric pieces are divided into three groups equally for dissolving at 30, 40 and 50 °C, respectively. Of course, the dissolving time is 30 minutes and the liquor ration is 100 (ml/g) in the experiment.

From the left graph in **Figure 4**, it is observed that the cotton contents obtained at 30 °C and 40 °C are closer and much less than the results dissolved at 50 °C. However, when dissolved at 50 °C, the results are almost around the standard value of 76.4% and the biggest difference is no more than 1%. From the mean value and standard deviation on the right graph, it is also suggested that cotton is dissolved sufficiently and is stable at

50 °C. Hence for cotton dissolved by sulfuric acid solution, the experimental temperature cannot be lower when assisted by ultrasound.

Influencing of fabric forms

In the previous discussion, all the blended fabrics are untwisted into yarns for experiment, and good effects are obtained. However, there are blended fabrics that cannot be untwisted into yarns, and finding an alternative fabric form is necessary. Blended fabric FS1 is used and divided into two parts: Part one is cut into pieces weighing about 1 g and shredded into strips of about 0.1 cm × 0.5cm, meanwhile part two is cut into pieces weighing about 1 g and untwisted into yarns of about 1.5 cm long. From each part ten specimens are prepared, which are dissolved by ultrasound of 20 kHz

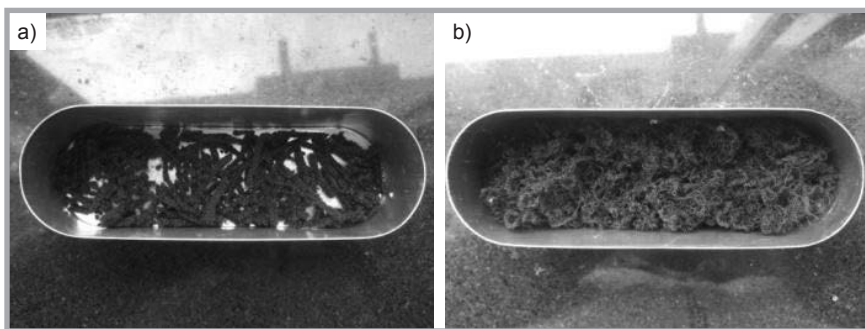


Figure 5. Different fabric forms for dissolution by ultrasound: a) cut pieces of about 0.1 cm × 0.5 cm, (b) pieces untwisted into yarns about 1.5 cm long.

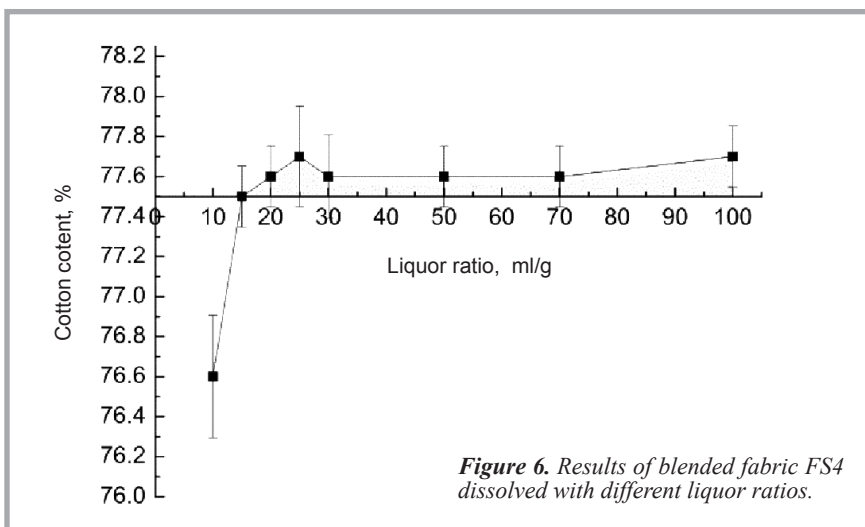


Figure 6. Results of blended fabric FS4 dissolved with different liquor ratios.

at 50 °C for 30 minutes. The two fabric forms are shown in **Figure 5**.

Dissolution results with the two fabric forms are shown in **Table 2**, from which it can be seen that all the data are valid when considering 1% tolerance. It can be reasonably stated that if the cotton is dissolved sufficiently with the two fabric forms, the two data sets will follow the same Gaussian distribution. At a 5% significance level, the two groups are analysed. Results indicate that there is no difference between the two data sets, which means the fabric form shredded into strips is an effective alternative for blended fabric that cannot be untwisted into yarns.

Influence of liquor ratio

The liquor ratio, defined as the ratio of dye solution and fabric in the textile dyeing industry, is defined as the ratio of sol-

vent (ml) and fabric (g) in this study. For quantitative analysis of blended fabrics by the method of chemical dissolution, the standard liquor ratio was 100, which deserved further research for the purpose of resource savings and environmental protection. Blended fabric FS4 is employed for discussing the influence of liquor ratio on quantitative analysis with ultrasound. The blended fabric is untwisted into 24 yarn pieces, each weighing about 1 g, and the 24 fibre pieces are divided into 8 groups equally corresponding to a liquor ratio of 10, 15, 20, 25, 30, 50, 70, and 100. The dissolving time is 30 minutes and the experimental temperature 50 °C.

The results of cotton fibre dissolved in blended fabric by ultrasound with different liquor ratios are shown in **Figure 6**, from which it can be observed that cotton is dissolved quickly, even when the

liquor ratio is 10. When up to 15, both the mean value and error bar indicate that cotton is dissolved sufficiently and steadily. This finding suggests that the solvent was excessive for quantitative analysis of blended fabric by the traditional method. However, a small liquor ratio may cause a big random error because it is obvious that fibres are not easily or sufficiently immersed in the solvent. By observing the process of the dissolving experiment, a liquor ratio of 50 is considered optimum for quantitative analysis of blended fabrics by ultrasound.

Conclusions

The traditional method for quantitative analysis of blended fabrics by chemical dissolution was used for manual operation; especially mechanical oscillation and glass rod stirring were adopted in the dissolving process. Owing to the requirement for work efficiency and testing precision, the feasibility of ultrasonic technology applied in quantitative analysis of cotton and polyester blended fabrics by chemical the dissolution method is investigated.

1. According to the analysis of ultrasound frequency, it can be seen that there is no difference between the three frequencies of 20, 28 and 40 kHz.
2. Although the standard dissolving time was 60 minutes according to ISO 1833-11:2006, the experimental time of cotton dissolution can be reduced to 30 minutes when applying ultrasonic technology.
3. However, the experimental temperature has to be consistent with the standard value of 50 °C, which may be determined by the properties of the solvent and dissolved fabric.
4. Test specimens should be prepared in small long strips if the blended fabric cannot be split into yarns, for there is no difference between them during dissolution assisted by ultrasound.
5. A more meaningful role is played by ultrasonic technology applied in chemical dissolution as the solvent can be reduced by half, which is conducive to resource savings and environmental protection.

In general, the study demonstrated that ultrasound assisted dissolution for quantitative analysis of blended fabrics is an efficient method, which is better than the traditional mechanical vibration method in some ways.

Table 2. Dissolution results with different fabric forms.

Fabric form	Cotton contents, %	Mean value	Standard deviation
a	72 71.7 71.7 71.4 71.8 71.6 71.3 71.9 71.0 71.1	71.5	0.347
b	71.7 72 71.8 71.9 71.5 71.7 71.7 71.1 71.4 72.1	71.7	0.296

Acknowledgements

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INSTITUTE OF BIOPOLYMERS AND CHEMICAL FIBRES

LABORATORY OF BIODEGRADATION

The Laboratory of Biodegradation operates within the structure of the Institute of Biopolymers and Chemical Fibres. It is a modern laboratory with a certificate of accreditation according to Standard PN-EN/ISO/IEC-17025:2005 (a quality system) bestowed by the Polish Accreditation Centre (PCA). The laboratory works at a global level and can cooperate with many institutions that produce, process and investigate polymeric materials. Thanks to its modern equipment, the Laboratory of Biodegradation can maintain cooperation with Polish and foreign research centers as well as manufacturers and be helpful in assessing the biodegradability of polymeric materials and textiles.

The Laboratory of Biodegradation assesses the susceptibility of polymeric and textile materials to biological degradation caused by microorganisms occurring in the natural environment (soil, compost and water medium). The testing of biodegradation is carried out in oxygen using innovative methods like respirometric testing with the continuous reading of the CO₂ delivered. The laboratory's modern MICRO-OXYMAX RESPIROMETER is used for carrying out tests in accordance with International Standards.



The methodology of biodegradability testing has been prepared on the basis of the following standards:

- **testing in aqueous medium:** 'Determination of the ultimate aerobic biodegradability of plastic materials and textiles in an aqueous medium. A method of analysing the carbon dioxide evolved' (PN-EN ISO 14 852: 2007, and PN-EN ISO 8192: 2007)
- **testing in compost medium:** 'Determination of the degree of disintegration of plastic materials and textiles under simulated composting conditions in a laboratory-scale test. A method of determining the weight loss' (PN-EN ISO 20 200: 2007, PN-EN ISO 14 045: 2005, and PN-EN ISO 14 806: 2010)
- **testing in soil medium:** 'Determination of the degree of disintegration of plastic materials and textiles under simulated soil conditions in a laboratory-scale test. A method of determining the weight loss' (PN-EN ISO 11 266: 1997, PN-EN ISO 11 721-1: 2002, and PN-EN ISO 11 721-2: 2002).



The following methods are applied in the assessment of biodegradation: gel chromatography (GPC), infrared spectroscopy (IR), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM).

Contact:

INSTITUTE OF BIOPOLYMERS AND CHEMICAL FIBRES
ul. M. Skłodowskiej-Curie 19/27, 90-570 Łódź, Poland
Katarzyna Dziedziczak Ph. D.,
tel. (+48 42) 638 03 31, e-mail: lab@ibwch.lodz.pl

Europe&Emirates Economic Forum

October 24, 2017

Venue: InterContinental Hotel

Moderator: Dariusz Małag – presenter, coordinator and producer of many events including premium class galas, music and sport events.



- 8:30 AM - 9:00 AM Registration of participants
- 9:00 AM - 10:15 AM **Celebratory greeting of the Guests**
Ministry of Development Department of International Cooperation - Krzysztof Dąbrowski
Ambassador of the United Arab Emirates in Poland, Embassy of the United Arab Emirates in Warsaw -J. E. Yousif Eisa Bin Hassan Al Sabri
President of the Emirates & Europe Business Development Cluster
- 10:15 AM - 11:05 AM **Panel: EEBD Cluster – International Economic Cooperation Europe - United Arab Emirates**
Dubai International Financial Centre Authority – Mr. Ali Hassan
Vice President of the Board of Emirates & Europe Business Development Cluster Juan Davi
Country Manager of Emirates Airlines – Maciej Pyrka
Ministry of Development - Department of International Cooperation - Hubert Niewiadomski
The President of Polish Agency of Trade and Investment Tomasz Pisula
Kizad DBD UAE Chief Executive Officer
Klaudia Lach, I Counselor, Ministry of Foreign Affairs, b. Head of Politburo-Economic and Consular ETA Group –
Mr. Akber Mohammed
- 11:05 AM - 11:30 PM **Panel: Development of international innovation**
Moderator: Tomasz Szlązak
Ministry of Economy UAE Director of International Organizations in the Ministry of UAE – Aisha Al Kubaisi
- 11:30 AM - 12:10 PM **Panel: The National Centre for Research and Development**
- 12:10 PM - 12:20 PM **Signing the memorandum between the Investment Service Center and the Agency for Technological Innovation in Georgia**
- 12:20 PM - 12:30 PM Coffee break



- 12:30 PM - 1:20 PM **Panel: The international market as a chance for economic development**
- Ministry of Development - Krzysztof Dąbrowski
 Ministry of Economy of Ukraine
 Kurdistan Government representative - Ziyad Raouf
 Embassy of the United Kingdom
 Ministry of Economy in Georgia
 Embassy of Spain
 Embassy of Estonia
 Mr. George Zviadadze the chairman of LEPL Georgia's Innovation and Technology Agency
 Luxury Connections DMCC in Dubai Mario Anthony - Owner and Managing Director
- 1:20 PM - 1:30 PM **Signing a memorandum between the Investment Services Center and the representatives of the Government of Ukraine**
- 1:30 PM - 1:40 PM **Signing memorandum between the Investment Services Center and Kizad DBD UAE**
- 1:40 PM - 2:00 PM **Panel: Building international relationships through sport**
- Legia Warsaw S.A.
- 2:00 PM - 2:30 PM **Panel: Modern technologies for banking**
- PEKAO S.A. Representatives
- 2:30 PM - 3:00 PM Lunch break
- 3:00 PM - 4:45 PM **Panel: Green Cars Cluster – a Chance for e-mobility development**
- Ministry of Infrastructure and Construction
 Presidents of Polish Cities:
 - Bartosz Bartoszewicz - Vice-President of Gdynia
 - Tadeusz Ferenc - President of Rzeszow
 National Fund for Environmental Protection and Water Management - Krzysztof Masiuk
 National Centre of Research and Development
 President of Green Cars Cluster
- 4:45 PM - 5:30 PM **Panel: Innovation and international technology transfer**
- Moderator: Dr Wojciech Blecharczyk**
 Lodz Industrial Development Agency
 EGTA
 European Center for Laboratory Research CEZAMAT
 President of Medlabs - Różewicki Marian
 Scientific Institutions
 National Chamber of Commerce - Ambassador Jerzy Drożdż
 Georgian Startups
- 8:00 PM **Ceremonial gala and the final of "Leaders of Innovation – Innovation Awards 2017"**

