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Influence of Air Pollution in Cracow on the Interception of the Sessile Oak (*Quercus petraea*)

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Abstract

The aim of this study was to determine the changes in the course of interception and contact angle curves of the sessile oak occurring in the growing season depending on air pollution. Samples were collected from two areas located in the southern Poland characterized by different degrees of pollution. The first research site was established in a tree stand in the Niepołomice Forest District, a control area. The second research position was designated in the urbanized part of Cracow, where air pollution with carbon dioxide, sulphur dioxide, trace metals and polycyclic aromatic hydrocarbons (PAHs) is one of the highest in Europe. It was discovered that from July, when the exposure to pollutants is quite long, the surface of leaves starts to erode. Gradual changes in the structure of leaves take place, which consequently alter the values of interception. It is manifested by the fact that values of interception increase with the growth of wettability of plant surface material. Leaf ageing affects water interception but the speed and extent of the changes induced by ageing process are higher in the polluted environment. It was confirmed that interception growth is accompanied by the decrease of a contact angle. The results obtained proved that air pollution in the city, besides seasonal natural variations, cannot be omitted at constructing interception models.

Keywords: contact angle, polycyclic aromatic hydrocarbons (PAHs), Cracow, urban greening.

Introduction

Interception, i.e. the amount of rainwater retained in the tree crowns, is one of the main components of the water balance of forest and urban ecosystems (Xiao and McPherson 2016). It is treated as a water capacity of the tree crowns and considered a key element in eco-hydrological processes (Crockford and Richardson 2000, Chang 2006, Wang et al. 2012, Zhang et al. 2016). It is measured using different laboratory or field methods (Keim et al. 2006, Wang et al. 2012, Sadeghi et al. 2016). It is one of the least known and parameterized components of hydrological equations (Pypker et al. 2005). Interception is conditioned by wetting, a physical feature of plant material expressed as an angle of inclination between water droplets and the surface of the leaf. This angle may be treated as a bioindicator of environmental pollution (Tranquada and Erb 2014). Aryal and Neuner (2010) created a classification system of wetting degrees based on contact angles. The evaluation of physical features such as contact angles and surface free energy is successfully used for different types of artificial materials (Liber-Kneć and Łagan 2014).

Interception and wetting largely depend on the state of assimilation apparatus surface (Rosado and Holder 2013, Klamerus-Iwan 2014). Research involving coniferous and deciduous trees conducted by Sase et al. (2008) revealed that older leaves are characterised by more uniform wettability as compared with younger leaves. Seasonal and species-specific changeability of leaf wettability was reported by Osuch (1994), Zeng et al. (2000) as well as Klamerus-Iwan and Błońska (2016). The texture of the leaf surface is affected by the changing environment, mainly the level of pollution (Kosiba 2008, Popek et al. 2013). Hydrophobicity and hydrophilicity are largely determined by the amount of waxes on the leaf (Sase et al. 1998) as well as the presence of trichomes (Hamlett et al. 2011, Fernandez et al. 2011). Tree species differ in the amount of pollution accumulated on their surface (Dzierżanowski et al. 2011, Sæbø et al. 2012; Popek et al., 2012).

Air pollution in Cracow and the whole southern Poland is among the highest in Europe. Geographical location, main wind directions, historical and current industrial dust deposition affect high concentration of pollutants (Muskała et al. 2015, Błońska et al. 2016). Pollution in the form of particulate matter (PM10 and PM2.5) and tar substances rich in polycyclic aromatic hydrocarbons and metals which settle on the leaves can cause changes in hydrophilicity of plant material.

The analysis of epicuticular wax and cuticle using SEM technique in the context of hydrophobic properties was performed by Koch and Barthlott (2009) as well as Sgrigna et al. (2015). Contamination with polycyclic aromatic hydrocarbons may change hydrological characteristics of soils (Klamerus-Iwan et al. 2015, Błońska et al. 2016). The study will fill a gap in the knowledge on physical and hydrological features of foliage and the degree of its contamination. As the level of pollution continues to grow (WHO 2006), contribution of anthropogenic factors should be taken into account in the process of constructing hydrological models.

The issue of an increasing amount of pollution in the cities has been raised many times (Matysek et al. 2012) and it is a subject of the works conducted within the framework of the European programmes (COST Action FP1204). However, the effect of pollution on water interception and its relationship to hydrological properties has not been investigated sufficiently. The purpose of this study was to determine variations in the interception and contact angle curves of the sessile oak occurring in the growing season depending on air pollution. The series of laboratory experiments were designed to test hypothesis that long-term exposure of leaves to substances polluting city air is capable of changing leaf texture and its hydrophobicity.

Materials and Methods

Research areas and sampling

Plant material was obtained from two areas characterised by diverse level of pollution. The first research site was located in the Niepołomice Forest District (DB, Z1) and the material collected there was used as control samples. Urbanised part of Cracow – which represents one of the most heavily polluted areas in Europe (EEA 2007) struggling with air pollution with carbon dioxide, sulphur dioxide, trace metals and polycyclic aromatic hydrocarbons (PAHs) – was selected as the second research area (DB, Z2).

Five trees with finely developed crowns were designated in each research area. Ten leafy twigs with the length of approximately 1.2 m were obtained from each tree. The twigs were obtained from different parts of the tree using pruning shears. Then the twigs were secured with moistened cellulose wadding and kept in tightly sealed conditions to avoid leaf turgor loss. Analysis of the interception and contact angles was performed immediately after delivering plant material to the laboratory. All experiments were repeated in the middle of each month from April to November 2016. Additionally, the content of polycyclic aromatic hydrocarbons was determined for leaves collected in Cracow in April and September.

Measurement of the interception and contact angles

Leafy twigs delivered to the laboratory were divided into 25 smaller pieces having a length of approximately 30-35 cm. Each twig was weighted and subsequently sprinkled with fixed amount of water using special dispenser. Quantity of water applied to spray each twig amounted to 100 g. Plant material weighting was repeated after sprinkling. The mass before and after the experiment was used to calculate the amount of retained water. The value obtained was expressed as the percentage of water retained on the twig. The experiment required keeping steady temperature (20-21 ºC) and humidity (40-45%) inside laboratory. Another key factor regarded the temperature of distilled water used for sprinkling which reached the value of 21 ºC (±1 ºC). Above-mentioned parameters were determined based on research carried out by Klamerus-Iwan (2013, 2016). Twenty-five measurements were taken for each twig collected from sessile oaks growing within the Niepołomice Forest District and within Cracow urban area. After 8-month experiment the set of data included 400 interception values determined for 2 research areas.

The experiment was designed to demonstrate seasonal variability of leaf water drop adhesion depending on the level of pollution. In order to achieve this research objective water was dripped on the leaves of individual twigs and then photographed using a camera with suitable parameters. Photos were taken in a light box to capture the shape of the water droplet and a contact angle between the water droplet and the surface of the leaf. Contact angles were calculated using SigmaScan 5.0 Pro software package (SPSS 1999). Measurements taken included an interior angle between the water droplet and the surface of the leaf. The results obtained were compared with wettability categories suggested by Aryal and Neuner (2010). Methodology and terminology used to describe parameters of water droplets and contact with the surface are compatible with those commonly used in hydrology (Nanco et al. 2013, Rosado and Holder 2013).

PAHs content was determined for leaves collected in the centre of Cracow in April and September. Every leaf was flushed twice with 50 ml hexane, then hexane was filtered through laboratory paper filter and evaporated to dryness. Mass of dry residue and dry powder trapped by the filter was measured. Dry samples were dissolved in acetonitrile (1 ml each), sonicated for 30 min, filtered and analyzed using HPLC. The Agilent Technologies 1260 system was equipped with a fluorescence detector and a 4.6×150 mm, 5 µm Eclipse XDB-C18 HPLC column (Agilent Technologies). Mobile phases consisted of water (A) and acetonitrile (B) at a flow rate of 1 mL/min. The volume of the sample injection amounted to 5 µL. Compounds were eluted using the following gradient: 0–5 min 20:80 A:B→13:87 A:B, 5–7.5 min held at 13:87 A:B, 7.5–15 min 13:87 A:B→0:100 A:B. Ten-point calibration curves were prepared for the following compounds: phenanthrene (Phe), naphthalene (Na), pyrene (Pyr), chrysene (Chr), benzo[a]pyrene (BaP), acenaphthylene (Acy), anthracene (Ant) and fluoranthene (Flt). These compounds were selected on the basis of their high concentration in industrial pollutants.

Moreover, data on the quality of the air collected by the *Voivodeship* Inspector for Environmental Protection within air quality monitoring system was used to present mean monthly values for selected pollutants detected in the centre of Cracow. Emissions data were obtained from the samples collected from the automatic measuring unit located at Krasińskiego Avenue about 2 m above the surface of the ground.

Statistical analysis

Statistical analyses were performed using Statistica 10 software package (StatSoft 2010). Arithmetic mean and standard deviation were used as a measure of dispersion of the set of values. *PCA* (Principal Component Analysis) procedure was applied to reduce the number of variables and to establish correlation between them. Detailed analysis of principal components is a useful tool applied to establish which variable has the most significant influence on a particular principal component. *PCA* was used to determine the effect of polycyclic aromatic hydrocarbons on the interception and a contact angle. Moreover, data were analysed using the Pearson's r correlation test to determine dependence between mean values of interception and the levels of air pollutants. The differences between the mean values were evaluated with the nonparametric Mann-Whitney U test.

Results

Assessment of the interception and leaf hydrophobicity

Interception of sessile oak twigs collected in the Niepołomicka Forest differs from the interception obtained for twigs collected from the centre of Cracow. At the beginning of the growing season twigs from the forest area show higher values of the interception as compared to the twigs from polluted area. Opposite trend was observed in July. Higher interception values were reported for twigs collected in the urban area.

The experiment revealed significant differences of the interception loss between two sampling sites. The highest average interception for the whole growing season was obtained for twigs collected in the city centre (18.44%). The average interception for the whole growing season for twigs collected in woodland amounted to 17.76%. The most considerable variability of interception was reported in October. What is more, interception calculated for twigs collected from the city centre demonstrated higher degree of variability.

Water droplet contact angles, providing information on the degree of wettability, determined for two locations declined during the growing season. Decreasing contact angles indicate that adhesiveness of the droplet to the plant material is increasing. The course of the contact angle curves is inversely proportional to the interception curves. Contact angles for the twigs collected in the urban area at the beginning of the growing season are higher, what means that forces of adhesion between water and the leaf surface as well as an overall contact area were lower. After July values of contact angles obtained for the urban area decreased, which is an indicator of improved adherence to the leaf surface (Table 1).

Decreasing contact angle between a water droplet and the leaf surface is a sign of the increase of leaf hydrophilicity. Based on the results obtained, we revealed

Table 1. Variations of the interception and contact angles with standard deviation

	Interception		Angle	
	Z1	Z2	Z1	Z2
IV	4.64±1.23	3.06±0.84	114.48±4.77	117.88±6.88
V	6.90±1.16	6.39±1.06	112.68±4.98	120.96±4.33
VI	10.45±1.99	9.26±1.54	109.60±4.80	114.24±2.11
VII	14.29±1.11	15.64±1.62	92.80±4.76	90.04±3.31
VIII	19.01±1.19	19.89±2.08	74.20±3.71	72.40±2.33
IX	24.43±1.98	26.27±2.63	58.60±4.87	54.68±2.38
X	27.68±2.44	31.70±3.65	41.20±4.14	33.2±2.94
XI	34.62±1.74	35.23±1.71	19.04±5.00	13.76±2.47
H	192.99	191.88	188.81	190.77
p-value	<0.05	<0.05	<0.05	<0.05

that leaf wettability increases with the advancement of ageing process and as a result of exposure to air pollutants. (Table 2). In July, non-wettable leaf surface changes into wettable. Twigs collected in the urban area develop extreme-hydrophilic properties earlier as compared to the control group.

Table 2. Comparison of contact angles and wettability for two research sites (wettability categories according to Aryal and Neuner 2010)

Month	Z1	Wettability category	Z2	Wettability category
IV	114	Non-wettable	118	Non-wettable
V	113	Non-wettable	121	Non-wettable
VI	110	Non-wettable	115	Non-wettable
VII	93	Wettable	90	Wettable
VIII	74	Highly wettable	73	Highly wettable
IX	59	Highly wettable	55	Highly wettable
X	41	Highly wettable	34	Extreme-hydrophilic
XI	20	Extreme-hydrophilic	14	Extreme-hydrophilic

Z1 – a contact angle for twigs collected in Niepołomice; Z2 – a contact angle for twigs collected in the centre of Cracow.

Correlation between PAHs, interception and contact angles

Analysis of air pollution revealed substantial differences regarding occurrence of chemical substances within the area of Cracow recorded in the growing season, which is from April to November (Table 3). Total concentration of air pollutants selected for the experiment reached the highest values in autumn (October, November) and the lowest in July. The most significant variances were reported for nitric oxide (NO_2), the least significant for carbon monoxide (CO). Similarly, to other pollutants, the highest concentrations of PM10 and PM2.5 were documented in autumn (Table 3).

Table 3. Levels of selected air pollutants (μg/m^3) according to the measurement taken by the Voivodeship Inspector for Environmental Protection

Month	NO2	NOx	NO	CO	C6H6	PM10	PM2.5
IV	63	209	95	755	1.7	58	39
V	64	189	82	657	1.1	47	30
VI	66	184	77	631	0.9	34	22
VII	59	145	56	557	1	30	21
VIII	61	166	69	616	1.2	36	21
IX	66	214	96	838	1.9	52	33
X	50	202	99	804	2.3	56	33
XI	58	260	132	1245	3	69	45

All monitored PAHs reached the highest levels of concentration in September. According to the measurements taken, the air was the most heavily polluted with naphthalene and acenaphthylene. Concentrations recorded in September amounted to 4.656.86 ng/g for acenaphthylene and 1.606.55 ng/g for naphthalene and in April 4.656.86 ng/g and 1.353.53 ng/g, respectively. The lowest concentrations were recorded for anthracene and chrysene (Table 4). Analysis using nonparametric Mann-Whitney *U* test revealed statistically significant variances in the concentration of all monitored PAHs in September and April (Table 4).

Table 4. Levels of selected aromatic hydrocarbons (ng/g) accumulated on the surface of the leaves collected in Cracow in May and September; results of Mann-Whitney *U* test

Month	*naphtalene*	*acenaphthylene*	*phenanthrene*	*anthracene*	*fluoranthene*	*pyrene*	*chryzene*	*benzo[a] pyrene*
IV	1342.33	5288.12	557.00	13.51	155.10	87.80	29.16	31.58
IV	1337.32	4259.27	687.71	14.32	79.27	85.03	27.58	34.96
IV	1362.80	4229.63	572.52	13.90	165.73	82.36	28.94	34.33
IV	1315.62	4256.14	572.35	14.04	81.20	89.35	29.93	48.99
IV	1409.58	5251.18	569.27	13.70	126.29	79.10	28.78	39.93
IX	1607.60	6400.68	1143.85	16.47	218.82	123.73	33.35	57.53
IX	1585.85	6501.29	1139.94	15.28	241.84	129.44	33.56	54.32
IX	1582.88	6484.00	1130.07	16.23	187.13	120.90	32.99	47.98
IX	1574.56	6443.58	1135.33	16.48	222.68	122.02	33.50	68.87
IX	1681.91	6466.70	1114.90	15.60	189.48	121.24	31.67	55.14
mean IV	**1353.53^b**	**4656.87^b**	**591.77^b**	**13.89^b**	**121.52^b**	**84.73^b**	**28.88^b**	**37.96^b**
mean IX	**1606.56^a**	**6459.25^a**	**1132.82^a**	**16.01^a**	**211.99^a**	**123.47^a**	**33.01^a**	**56.77**

As a result of conducting statistical analysis, variables 1 and 2 were selected. The sum of the variables accounted for 93.42% of the variance. Variable 1 accounted for 88.91% and variable 2 for 4.51% of the variance. Variable 1 represents concentration of polycyclic aromatic hydrocarbons in leaves collected in April and September and the interception. Variable 2 represents inclination angles between water droplets and the surface of the leaf. Analysis revealed negative correlation between inclination angles and the value of interception (Figure 1). The decrease of droplet inclination angles

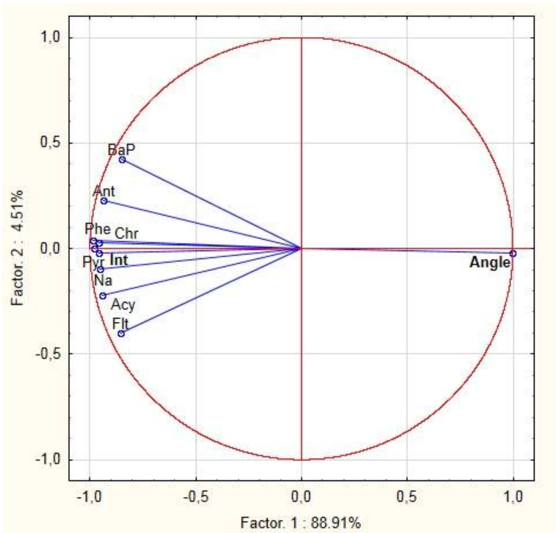

Figure 1. Projection of the variables on the factor-plane for different polycyclic aromatic hydrocarbons (Na stands for naphthalene, Acy stands for acenaphthylene, Phe stands for phenanthrene, Ant stands for anthracene, Flt stands for fluoranthene, Pyr stands for pyrene, Chr stands for chrysene, and BaP stands for benzo[a]pyrene)

correlates with the increase of the amount of water retained on the surface of the leaf.

No direct correlation was found between mean values of air pollution and monthly means calculated for the interception. It may be explained by the fact that pollutants accumulate on the surface of leaves of deciduous species each month and the effects of pollution are observed in autumn.

Discussion

The study revealed seasonal variability in the amount of water retained on the leaves irrespective of the sampling site. Similar observations were made by other researchers (Klamerus-Iwan and Błońska 2017). Authors concluded that wettability and the level of interception increases in line with the age of the leaf. Present research demonstrated that interception of the sessile oak leaves increases as a result of ageing process in both analysed research areas. However, the speed and extent of the changes observed depends on the site where the plant material was collected. Analysis of wettability patterns indicated that leaves change surface properties from non-wettable to extreme-hydrophilic (Aryal and Neuner 2010). Decreasing contact angles provide information about the increase of leaf wettability occurring in subsequent months of the growing season. Annual interception and wettability variances may be associated with changes affecting leaf morphology and the structure of the assimilative apparatus (Martin and Willert 2000, Popek et al. 2013). What is more, pollutants deposited on the surface of the leaves also influence examined parameters. From July, when exposure to pollutants is long enough, the surface of the leaves undergoes erosion and the structure of the leaf changes affecting leaf interception. It is manifested by the increase of interception and plant material wettability. Starting from July, sessile oaks growing in the urban area retain more water than trees growing in the forest. It may result from deposing hydrophobic aromatic hydrocarbons on the surface of the leaves. In that period leaf hydrophobicity increases. Contact angles show the opposite trend. Repeating interception and wettability measurements every month in urban and forest habitats provides insight into the mechanisms underlying changes of leaf hydrophilicity occurring during the growing season.

Pollution can significantly influence the level of interception. This effect is particularly marked in autumn, when the emissions of dangerous pollutants increase. Trees can reduce the amount of PM2.5 by intercepting and retaining these particles on their leaves and bark surfaces (Jouraeva et al. 2002). Smith (1981) suggested that the quantity of accumulated pollutants depends on the size of the leaf. Moreover, the number of PAHs accumulated on the surface of the assimilative apparatus depends on physical properties of particular polycyclic aromatic hydrocarbons and environmental conditions (Franzaring 1997, Bohme et al. 1999). Abiotic stress that trees experience in urbanised areas (Bytnerowicz et al. 2007, Matysek et al. 2012) is a major cause of visible changes affecting the assimilative apparatus. Changes in the morphology of the assimilative apparatus are responsible for alterations in the course of interception curves and leaf hydrophobicity. Precise measurement of total amount of accumulated pollutants requires taking into account other factors such as amount and frequency of rainfall which are responsible for pollutant wash-off from the surface of leaves (Nowak et al. 2008).

The increase in the values of interception may also be related to the quantity of wax which shows variation within the growing season and depends on the tree species (Neinhuis and Barthlott 1998).

The results obtained in our study illustrate that interception models, besides natural seasonal fluctuations, should take into account air pollution in the city.

Conclusion

In our study, seasonal changes in the contact angle and water capacity of sessile oak have been confirmed. The increase of interception is negatively correlated with a contact angle and is an indicator of greater leaf hydrofobicity. Changes in the leaves surface energy lead to changes in the contact angles between the leaves and water drops. A relationship between the contact angle and the degree of wettability has been found. Annual interception and wettability variances may be associated with changes affecting leaf morphology and the structure of the *assimilative apparatus.*

The extent of changes to leaf texture and hydrofobicity depends on time of exposure to pollutants. Leaves growing in urban environment change wettability pattern into extreme-hydrophilic more rapidly.

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