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Original scientific paper

RHEOMETRIC AND DYNAMIC MECHANICAL ANALYSIS OF COMPLEX NATURAL RUBBER BASED COMPOSITES

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This work describes the preparation and characterization of complex natural rubber (NR) based composites containing hybrid nano- and conventional fillers intended for base seismic application. Thorough rheometric and dynamic mechanical analyses in strain sweep mode at 2 Hz and 10 Hz (two frequencies laying in the range 0 – 15 Hz in which most of the earthquakes have the dominant frequencies) were performed on complex natural rubber (NR) based composites containing hybrid nanofiller (carbon nanotubes, expanded montmorillonite) and different amounts of conventional fillers like carbon black (CB) and silica. The rheometric studies showed that the influence of the combination of the different fillers on curing parameters is quite complex, but mainly the introduction of the fillers reduces the scorch and optimum cure time of the compounds. The dynamic mechanical analysis showed a pronounced non-linear strain dependence of the storage modulus and a remarkable increase of the loss factor tan δ for all composites, especially for those containing high CB content, compared to the NR-gum. To describe this strain-dependency of the storage modulus the cluster-cluster aggregation (CCA) model was used. The values of the fitting parameters $\Delta E'_0$, γ_c , and E'_{∞} calculated by this model show that they are affected by the type of the fillers present in the NR matrix and also by the applied frequency.

Keywords: natural rubber; hybrid composites; rheometric study; filler networking; Payne effect

INTRODUCTION

Natural rubber (NR) is used extensively in many different industrial and structural applications due to its outstanding mechanical properties: high resilience, high tensile and tear properties and good crack growth resistance. The mechanical properties of rubber as a soft material mostly depend on reinforcement by fillers which are an inevitable ingredient in rubber compounds. Colloidal fillers, like carbon black (CB) or silica, play an important role in this improvement of the mechanical properties of high performance rubber materials. However, due to their high structure and tendency to agglomerate, strong shear fields or filler modification are needed to ensure fine dispersion. Furthermore high quantities are needed to provide a sufficiently high level of reinforcement but on detriment of elasticity [1].

Besides these conventional fillers which are used to modify rubber properties for more than a century, the application of a new class of fillers – nanofillers, has attracted a huge scientific interest in this last decade. The implementation of novel nanofiller systems like layered double hydroxides [2], organomodified nanoclays [1, 3–10], halloysites and carbon nanotubes (CNT) [11–16] in elastomeric matrices is a subject of an intense research. The most investigated, thanks to their unique properties and demonstrated abilities to enhance the mechanical properties of elastomers, are layered silicates [1, 3–10] and carbon nanotubes (CNT) [11–16]. Montmorillonite (Mt) is commonly used owing to its large availability, low cost, high surface area and high cation exchange capacity. CNT on their behalf, are of particular interest due to their exceptional combinations of physical properties such as high aspect ratio, large flexibility, low density and superior electrical and thermal properties [17].

But not all desired properties of the filled rubber could be achieved by addition of low quantities of nano-particles. For instance, the energy dissipating capacity, a property that is significant in the design of structures and mechanical devices connected to problems involving mechanical resonance and fatigue, shaft whirl, heating and cyclic stress, is mainly governed by the filler-filler interactions and demands higher quantities of filler present in the rubber matrix.

Therefore, recently, an increasing interest is becoming evident for hybrid systems based on both nanofillers and conventional fillers [18–21]. These works show significant enhancement of material dynamic-mechanical properties thanks to the use of the hybrid filler system. It was reported by Galimberti et al. [18, 21] that the initial modulus values obtained with the hybrid CB-organo-montmorillonite (OMt) and CB-nanographite filler systems are much higher than those calculated through the simple addition of the two initial moduli of the composites containing only conventional fillers or only nanofillers.

In this study we performed thorough rheometric and dynamic mechanical analyses on complex NR based composites containing both nanoand conventional fillers. As a hybrid nano-filler a combination of 2 phr of multi-wall carbon nanotubes (MWCNT) and 16 phr of expanded organomontmorillonite (EOMt) was used. The optimization of this ratio was done in our previous study [22]. The overall content of the conventional fillers in all composites was kept constant at 40 phr in which the content of the individual fillers CB and silica were varied form 0 phr to 40 phr. Our interest was to investigate how the different combinations of fillers influence the processability and crosslinking of the rubber compounds. The main goal was to explore the rubber-filler and filler-filler interactions in order to understand the mechanism of energy dissipation in these complex NR-based systems. Wang [23] suggested that breakdown and reformation of the filler network, accompanied by the release of the trapped rubber from the filler network, causes an energy dissipation process during dynamic deformation. This mechanism suggests that the Payne effect or strain dependence of dynamic modulus of rubber can serve as a measure of filler networking which originates from filler-filler interaction as well as polymer-filler interaction [23].

DMA strain sweep measurements, carried out by applying cyclic deformations in the tensile mode at two different frequencies, 2 Hz and 10 Hz, respectively, were used to determine the loss factor, $\tan \delta$ and *G'* (storage) modulus. This study is part of a wider project which purpose is obtaining complex rubber compound suitable for base seismic isolation and these frequencies, seemingly quite close to each other, lay in the range in which most of the earthquakes are dominant and that is 0–15 Hz [24]. Finally, the CCA-model was implemented to understand more deeply the Payne effect, the strain dependency of the storage modulus, which is considered to be one of the mechanisms of energy dissipation.

EXPERIMENTAL

Materials

The rubber compounds were based on natural rubber (NR) using a Standard Malaysian Rubber grade (SMR 10). The Organo-Montmorillonite (OMt) used was Nanofil 15 supplied from Süd-Chemie AG Moosburg, Germany. Distearyl dimethylammonium chloride (QUAT) was used as an organic modifier. The specific gravity of this OMt was 1.8 g/cm⁻³ with an average particle size of 25 um. Carbon nanotubes, NC7000, a multiwall carbon nanotubes (MWCNT) produced by catalytic carbon vapor deposition (CCVD) process, were supplied by NANOCYL S. A. (Belgium). They were 90% pure containing 10% metal oxides and had an average diameter of 9.5 nm and an average length of 1.5 µm. The stearic acid was purchased from ACROS Organics, Geel, Belgium with 97% purity. The vulcanizing accelerators N-tert-Butyl-2benzothiazolesulphenamide (TBBS) and N-cyclohexyl-2-benzothiazolesulphenamide (CBS) were kindly provided from Rhein Chemie Rheinau GmbH, Mannheim, Germany. The carbon black was Corax N 330 (ORION Engineered Carbons GmbH, Köln, Germany), silica filler used was Ultrasil VN3 (Evonik Industries AG, Germany). Sulfur (S), N-(1, 3-Dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) and zinc oxide (ZnO) used in this study were of industrial grade.

Preparation of the hybrid rubber composites

The procedure of preparation of the expanded organo-montmorillonite (EOMt) is described in detail in ref. [22]. For the compounds containing nanofillers, the MWCNT were firstly dispersed in ethanol in a ratio 1:20 by weight. This pre-dispersion is required regarding the safe manipulation of the MWCNT, but it also enhances their dispersion in the rubber [16]. Then the batch composed of 100 phr NR and 10 phr MWCNT was mixed in an internal mixer (Haake Rheomix) at a fixed rotor speed of 60 rpm, at 90 °C for 20 min. Afterwards, this masterbatch was diluted with NR to a target of 2 phr MWCNT and was mixed together with 16 phr of EOMt, N330 and Ultrasil VN3 (for both fillers content was varied from 0 to 40 phr), ZnO, 6PPD and stearic acid in the internal mixer at 90 °C with 60 rpm rotor speed for 10 min. For the compound containing only conventional filler the NR was mixed together with 58 phr N330, the ZnO, 6PPD and stearic acid in the internal mixer at 90 °C with

60 rpm rotor speed for 10 min. In the last step of the preparation the vulcanization accelerators TBBS and CBS together with the sulfur were added in an open two roll mill (Polymix 110 L, size: 203 mm × 102 mm Servitec GmbH, Wustermark, Germany) at 90 °C. The milling was performed at 0.5 mm and later at 0.3 mm nip gap for 5 min. The formulations of NR compounds expressed as parts per hundred of rubber (phr) are shown in Table 1. The test samples were molded and cured to 2 mm tick plates by compression molding (150 kN) on an electrically heated hydraulic press at 150 °C. The rubber samples were vulcanized up to their respective optimum cure time (t_{90}) , previously determined with a vulcameter (Scarabaeus, Langgöns, Germany), and then stored for 24 h before the tests were performed.

Table 1. Formulation of the NR compounds

Sample code	NR^*	MWCNT	EOMt	N330	Ultrasil VN3
NR-gum	100	0	0	0	0
NR-h-CB40-S0	100	2	16	40	0
NR-h-CB20-S20	100	2	16	20	20
NR-h-CB0-S40	100	2	16	0	40
NR-CB58	100	0	0	58	0

*Mass of the ingredients was taken in parts per hundred of rubber (phr). The ingredients which amounts were kept constant in all compounds were ZnO 5 phr, stearic acid 2 phr, 6PPD 1 phr, CBS 1.5 phr, TBBS 0.2 phr and sulfur 1.5 phr.

Characterization

Curing studies were performed using Scarabaeus SIS-V50, a rubber processing analyzer (Scarabaeus, Langgöns, Germany) in the isothermal time sweep mode at 150°C for 60 min.

Amplitude sweep measurements of the dynamic mechanical analysis were performed on rectangular samples with dimensions $10 \text{ mm} \times 35 \text{ mm}$ using an Eplexor 2000 N (NETZSCH GABO Instruments GmbH, Ahlden, Germany) in the tension mode at room temperature, at a frequency of 2 Hz, a static tensile load of 60% pre-strain was applied and then the samples were oscillated to a dynamic load from 0% to 40% strain, and at a frequency of 10 Hz, a static tensile load of 60% pre-strain was applied and then the samples were oscillated to a dynamic load from 0% to 50% strain.

RESULTS AND DISCUSSION

The rheometric curves and rheometric characteristics of the gum compound and the compounds with hybrid nano- and conventional fillers are shown in Figure 1 and Table 2, respectively. It is evident that the presence of fillers (both hybrid and conventional) results in an increase of the maximum rheometric torque as compared to the gum.

The compound containing 40 phr of reinforcing CB and the hybrid nanofiller (2 phr MWCNT and 16 phr EOMt) showed the highest maximum torque value and then it decreases with the decrease of the CB and increase of silica content. The lowering of the torque in the filled rubber compounds is a common fact when non-reinforcing clay, calcium carbonate and silica without silane coupling agent, such as in this case, are used as fillers [25]. The influence of the different fillers on the curing parameters is quite complex. The scorch time reduces significantly when 40 phr CB and the hybrid nanofiller are added to the gum compound, from 6.54 minutes to 1.74 minutes. For both nanofillers such an effect is already reported [1, 26, 27]. At present there are no clear explanations how MWCNT interfere with the cure chemistry, but it is plausible to think that the catalyst employed for the synthesis of MWCNT which remained trapped inside the carbon material may affect the vulcanization [27]. On the other hand, the accelerating effect of the organoclay is essentially attributed to the presence of an amine

modifier inserted into the clay galleries, since it is well known that amine groups activate the functional groups of the accelerants, giving rise to a synergic effect that leads to a faster and more effective vulcanization reaction [1, 27].



Figure 1. Rheometric curves if the gum compound and the compounds with hybrid nano- and conventional fillers

Compound	Minimum rheometric torque (R_{min})	Maximum rheometric torque (R_{∞})	Scorch time t_2	Cure rate index (CRI) $100/(t_{90}-t_2)$	Curing time (<i>t</i> ₉₀)
	dNm	dNm	min	min ⁻¹	min
NR-gum	1.04	9.24	6.54	14.88	13.26
NR-h-CB40-S0	1.16	18.22	1.74	15.50	8.19
NR-h-CB30-S10	1.15	17.66	1.66	21.32	6.35
NR-h-CB20-S20	1.23	16.49	1.82	23.36	6.10
NR-h-CB10-S30	1.66	15.82	2.18	22.17	6.69
NR-h-CB0-S40	1.81	14.56	2.70	25.19	6.67

Table 2. Curing characteristics of the NR based compounds

The same trend of changes can be observed in the optimum cure time (given by t_{90}). What is interesting is its significant reduction when silica filler is introduced into the compound. The introduction of silica also markedly influences the curing kinetics of the compounds, which can be interpreted in terms of the cure rate index (CRI). CRI is a parameter proportional to the average slope of the cure curve $(100/t_{90}-t_2)$ in the curing step region. The higher the value of CRI, the faster is the curing process. It is apparent that the curing rate is significantly increased when silica is added to the compound compared to the gum and the compound containing the hybrid nanofillers and only CB. This influence of silica on the curing parameters is in agreement with other studies which reported similar phenomena [28, 29]. This higher viscosity could be also observed as higher values of minimum torque determined from rheometer cure curves (Figure 1). In order to explore the rubber-filler and filler-filler interactions in these complex NR-based composites DMA strain sweep measurements were performed at two different frequencies 2 Hz and 10 Hz.



Figure 2. Strain dependence of E' (a) and tan δ (b) for the NR-gum and NR composites at 2 Hz



Figure 3. Strain dependence of E' (a) and tan δ (b) for the NR-gum and NR composites at 10 Hz

It is well known that the storage modulus for the filled rubbers depends on deformation amplitude, besides on frequency and temperature. This nonlinear behavior is the so called Payne effect [30, 31] and is explained with the existence of a fillerfiller network in the polymer matrix, above the filler percolation threshold. The strain dependence of the storage modulus (E') and the loss factor $(\tan \delta)$ at 2 Hz and 10 Hz are reported in Figure 2a and b and in Figure 3a and b, respectively, for the NRgum compound and the NR composites containing conventional and hybrid nanofillers. There is a pronounced non-linear strain dependence of the storage modulus (Figures 2a and 3a) for all composites, especially for those containing high CB content (NR-h-CB40-S0, NR-h-CB30-S10). It is interesting to note that there is a more pronounced difference between the storage modulus of the compounds containing a high amount of CB at higher frequency of 10 Hz, while for the compounds with a high silica content this difference is more pronounced at lower frequency of 2 Hz. This is connected to the structure of the filler networks and the way they respond to a cyclic dynamic strain. Tan δ (a measure of the dissipation energy) follows this trend, namely, a remarkable increase for all composites, especially for those containing high amounts of CB, compared to the NR-gum.

To understand deeply the Payne effect and to describe this strain-dependency of the storage modulus, we will use the cluster-cluster aggregation (CCA) model. The storage modulus, well above the percolation threshold, can be approximated by a function of the Havriliak-Negami type [32]:

$$\Delta G'(\gamma_0) \cong \Delta G'_0 \left[1 + \left(\frac{\gamma_0}{\gamma_c}\right)^{2m} \right]^{-\tau}$$
(1)

Here, γ_c is the strain amplitude, where half of the clusters are broken, *m* being an empirical exponent and $\tau \approx 3.6$ is the elasticity exponent of percolation [33].

The theoretical models for filled rubber are developed for shear modes but we attempted to use this model to predict the dynamic behavior in tension mode.

Figure 4 and Figure 5 show the experimental results of the strain-dependency of the modulus E' for the different NR based composites containing different combinations of fillers and as seen already with this approximation a fairly good description of the Payne effect is possible. The solid lines correspond to fitted curves according to Eq. (1).

The fitting parameters $\Delta E'_0$, γ_c , E'_{∞} , and mare summarized in Table 3. It becomes obvious that their values are affected by the type of the fillers present in the NR matrix but also by the frequency, except for the empirical Kraus-parameter m. The composites containing more CB exhibit a more pronounced Payne effect, which is reflected in the highest value of $\Delta E'_0$ for the NR-h-CB40-S0 composite. Addition of silica reduces the $\Delta E'_0$ value which is lowest for NR-h-CB0-S40 composite. The same trend is observed for both frequencies with the difference that for the higher frequency of 10 Hz the Payne effect is more pronounced than for the frequency of 2 Hz.



Figure 4. Plot of storage modulus vs. strain amplitude for the different NR based composites at 2 Hz



Figure 5. Plot of storage modulus vs. strain amplitude for the different NR based composites at 10 Hz

Composite	$\Delta E'_0$ (MPa)	γ_c (%)	т	<i>E</i> ' _∞ (MPa)
NR-h-CB40-S0	18.9	9.9	0.44	7.2
NR-h-CB30-S10	18.7	9.3	0.49	6.9
NR-h-CB20-S20	15.3	9.5	0.51	6.7
NR-h-CB10-S30	15.0	12.12	0.46	6.2
NR-h-CB0-S40	14.12	12.91	0.47	5.8

Table 3. Material parameters from least square fits of Eq. (1) to the experimental data
shown in Figure 4 (τ =3.6)

Table 4. Material parameters from least square fits of Eq. (1) to the experimental data
shown in Figure 5 (τ =3.6)

Composite	$\Delta E'_0$ (MPa)	$\gamma_c(\%)$	т	<i>E</i> ' _∞ (MPa)
NR-h-CB40-S0	26.8	11.9	0.46	7.8
NR-h-CB30-S10	21.6	11.1	0.46	7.2
NR-h-CB20-S20	16.6	12.2	0.46	6.5
NR-h-CB10-S30	16.6	12.2	0.46	6.7
NR-h-CB0-S40	17.3	13.6	0.45	6.4

A similar behavior is observed for the large strain E'_{∞} values that are dominated by the pronounced hydrodynamic amplification of structured particles like CB. E'_{∞} is the highest for the NR-h-CB40-S0 composite and then its value decreases with increase of silica content. On the other hand, these structured filler particles like CB, lead to a lower stability of the filler network, as seen by the lower γ_c -values for the composites containing more CB (NR-h-CB40-S0 and NR-h-CB30-S10). This value of γ_c (the strain amplitude, where half of the clusters are broken) increases when silica is more present in the NR matrix. The largest γ_c -values of 12.91% and 13.6% (for 2 Hz and 10 Hz respectively) are found for the NR-h-CB0-S40 composite, suggesting the highest stability, which is due to poor compatibility of silica filler to hydrocarbon rubber, its polar character and the ability to form hydrogen bonds [34]. The empirical Kraus-parameter m, that considers the power law dependency of the number of surviving clusters on the applied strain, shows very small variation with the type of applied fillers and also negligible variations with frequency [32].

CONCLUSION

Based on the above work, devoted to thorough rheometric and dynamical mechanical analyses of complex NR based composites, the following conclusions can be drawn. The rheometric studies showed the highest maximum torque value for the composite containing 40 phr CB and then it decreased with decrease of CB and increase of the silica content. The scorch time and optimum cure time reduced significantly when 40 phr CB and the hybrid nanofiller were added to the gum compound and further decreased with addition of silica, suggesting a better processability of these rubber composites and faster curing process.

The dynamic mechanical analysis showed a pronounced non-linear strain dependence of the storage modulus and a remarkable increase of the loss factor tan δ for all composites, especially for those containing high CB content, compared to the NR-gum. The influence of the frequency on the storage modulus was as follows: a more pronounced difference between the storage modulus of the composites containing high CB content at higher frequency of 10 Hz, and opposite to this, a more pronounced difference for the composites containing high silica content at lower frequency of 2 Hz. This might be connected to the structure of the filler networks and the way they respond to a cyclic dynamic strain.

The CCA-model gave a fairly good description of the Payne effect. From the calculated parameters $\Delta E'_0$, γ_c and E'_{∞} it is obvious that their values are affected by the type of the fillers present in the NR matrix but also by the frequency. The composites containing more CB exhibit more pronounced Payne effect, a trend that is observed for both frequencies with that difference that for the higher frequency of 10 Hz the Payne effect is more pronounced. On the other hand, CB as structured filler particles, lead to a lower stability of the filler network, as seen by the lower γ_c -values for the composites containing more CB (NR-h-CB40-S0 and NR-h-CB30-S10). This value of γ_c increases when silica is more present in the NR matrix, suggesting a higher stability, which is due to poor compatibility of silica filler to hydrocarbon rubber, its polar character and the ability to form hydrogen bonds.

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РЕОМЕТРИСКА И ДИНАМИЧКО-МЕХАНИЧКА АНАЛИЗА НА КОМПЛЕКСНИ КОМПОЗИТИ БАЗИРАНИ НА ПРИРОДНА ГУМА

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Во овој труд се опфатени подготовката и карактеризацијата на комплексни композити на база на природна гума, кои содржат хибридни нано- и конвенционални полнила, а кои се наменети за сеизмичка базна изолација. Извршена е темелна реометриска и динамичко-механичка анализа (режим на смолкнување при фреквенции од 2 Hz и 10 Hz) на овие комплексни композити кои содржат хибридно нанополнило, составено од јаглеродни наноцевчиња и експандиран монтморилонит и различни количини конвенционални полнила, саѓи и силициум диоксид. Реометриската анализа покажа дека влијанието на комбинациите од различни полнила врз параметрите на вмрежување е комплексно, но присуството на полнила главно го

намалува т.н. "скорч" време и времето на оптимално вмрежување. Динамичката механичка анализа покажа изразена нелинеарна зависност на модулите на еластичност од напрегање и значително зголемување на факторот на загуба, *tanó*, за сите композити, особено за оние кои содржат голема количина саѓи. За да се опише зависноста на модулите на еластичност од напрегањето, искористен е моделот на кластер-кластерска агрегација. Се покажа дека вредностите на фитуваните параметри $\Delta E'_0$, γ_c , и E'_{∞} пресметани според овој модел зависат од типот на полнилото присутно во еластомерната матрица, но и од применетата фреквенција.

Клучни зборови: природна гума; реометриска анализа; вмрежување на полнило; ефект на Пајн

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CHARACTERISTICS OF FRUIT FROM DIFFERENT PROGENIES OF JUJUBE (ZIZYPHUS JUJUBA MILL.)

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The pomological characteristics of five varieties and one wild form of jujube progenies were examined in the experimental orchard of the Institute of Agriculture in Skopje during 2002–2004. The seeds from the varieties: Zu tao czao, Da baj czao, Kitajski 2A, Ja czao, Vahshski 45–2, and Wild midleasiatic jujube type were obtained by open pollination. Some characteristics of the fruits such as: mass of the fruits (g), fruit density (g/cm³), mass of the seed (g), and randman (%) were investigated in 7–9 years old jujube seedlings grown from that seed material and planted in the frames of a breeder field. A dominant mother effect in the inheritance was registered in the mass and density of the fruits. For instance: the variety Kitajski 2A and the types from its progeny were characterized by the greatest mass of the fruits, while, the greatest density was found in the fruits of the variety Da bai czao and the types from its progeny. The greatest mass of the seed was recorded in the variety Kitajski 2A, while the biggest average mass of the seeds was characteristic for the progeny of the Ja czao variety.

Key words: jujube; open pollination; variety; type; fruit

INTRODUCTION

The jujube is a relatively new fruit kind which slowly but surely spreads in the fruit orchards. The characteristics, such as the late start of vegetation (late April), late blooming (the beginning of June) and long period of blooming (1-2 months), as well as the resistance to drought, make this fruit crop very interesting for growing in the present conditions of frequent climate changes. In our decades-long experiences in the breeding of this fruit kind there have been no records of falling or reducing of the yield in some critical years, unlike of some of the traditional fruit kinds as plum, apricot, almond, etc. Because of the genetic predispositions to resist to the external factors, the breeding programmes for this fruit kind are targeted in the direction of increasing of the size and quality of the fruits. There is a limited number of jujube varieties with large fruits. The fruit weight, fruit size and seed weight showed high genetic advance. The genetic advance expressed as a percentage of mean ranged from 12.48 % (fruit length) to 61.16 % (yield/plant) [1]. More and more work is being done to increase the randman and the time of keeping of the fresh fruits. Hence, the main goal of this work is to obtain types with improved fruit characteristics through a mass selection among the jujube varieties progenies.

EXPERIMENTAL SECTION

The investigations were performed under conditions of selection orchard with planting distance of 4×2 m, planted in 1997 with two years old seedlings of jujube varieties. In the investigation period plants were 7–9 years old, which means that they were at the beginning of full fruitfulness The plants were grown without intervened pruning, left to develop naturally shaped crown, specific for each genotype. 131 genotypes (62 % surviving plants, because of difficulties in transplantation) of progenies, obtained by open pollination between six jujube introduced genotypes are examined. 35 seedlings of each mother variety are planted in. As a control 3 trees of six mothers varieties with grafted technique "whip grafting" in 1998 were used. The classification, according to the size of fruits, was performed by Sinko [2] in three groups: with large, medium and small fruits. In the group with large fruits the varieties/types with a mass of fruit greater than 12 g were included. The group with medium fruit size included varieties/types with a mass of fruit from 7 to 10 g. The varieties/types with a mass of fruit below 7 g belonged to the group with small fruit varieties.

The mass of fruits and stones, fruit density, and randman were analyzed. The examined properties varied and were statistically processed according to Mudra [3] in two levels of probability (0.05 and 0.01).

RESULTS AND DISCUSSION

The mass of the fruits unified the total quantitative characteristics of the jujube varieties and types [4]. The fruit weight per fruit had low heritability along with low genetic advance, indicating that dominance of epistatic effect was considerable on these characters, and hence a little improvement was possible through selection [1]. Yield of zizyphus genotypes had a significantly positive correlation with the spread, fruit weight and stone size. The fruit size had a highly positive correlation with the fruit weight and stone size. Fruit weight, fruit size and stone size were the main parameters for a yield which contributed via fruit weight [5]. From the variety Zu tao czao progeny analysis it was determined that the type 37/1 had

the biggest mass of the fruits (13.4 g), which is twice bigger than the average mass of the Zu tao czao progeny fruits (7.6 g) or for about one-third bigger (9.5 g) than the fruit mass of the mother variety (Table 1). Actually, 25 % of the progeny types had bigger mass of the fruits than the variety Zu tao czao. The high heritability of these characteristics, coupled with genetic advance for yield (kg/plant), pulp/stone ratio, indicated that it was probably due to an additive gene effect [6]. Heritability was high for fruit weight (97.2 %) and size (87.9 %), pulp/stone ratio (87.5 %) and seed weight (84.6 %) in jujube at Hisar [1]. There were significant positive correlations between fruit yield, fruit set, and pulp/stone ratio of jujube (cv. Umran), whereas the yield had significant negative correlations with fruit drop and stone weight [1].

There were recorded large differences in fruit density between the types from the Zu tao czao variety. The type 22/1 had the smallest stone from the types in the progeny. Actually, 80 % from the types had the smallest stone, and about 40 % greater randman than the mother variety.

The statistical analysis showed no significant differences between the mother variety and an average of the types. The type 37/1 deviated significantly with regard to all parameters.

With the comparative analysis of the characteristic data (Figure 1) it was determined that minor negative dependence between mass of the fruit and fruit density existed, as well as a slight positive dependence between the mass of the fruit and the mass of the stone (seed).



Figure 1. Dependence between fruit characteristics of all progeny types. The primary y-axis is for values of the mass of fruits, and the secondary *y*-axis is for the mass of stone and density of fruit.

From this figure it can be seen that types with small fruits had stronger variation in the fruit density, while the types with larger fruits had greater variation in the stone (seed) mass. Generally, the types with smaller fruits had higher density of fruit, with rare exceptions. The data analysis showed no statistical difference between the variety and the average of types in relation to stone mass (Table 1). 42–50 % of the types showed a significant difference and 17–20 % of the types had very significantly less mass of the stone than the mother variety.

In the variety Da baj czao the largest density of the fruits had the mother variety Da baj czao (Table 1). The highest mass of the fruits had the type 3/2. This type statistically deviated very significantly from the mother variety in terms of the mass of fruits and from the 24 % of the types in progeny. The type 6/2 had the lowest stone mass (Table 1). The highest randman had the same type 6/2. 40 % of the types had a higher randman than the mother variety. There were no statistically significant differences between the mother variety and an average of the types, and individually among the types in progeny in terms of stone mass. The progeny of the mother variety Da baj czao had on average the smallest mass (6.3 g), but also the biggest density of the fruits (0.600 g/cm³), compared with the other progenies. Because of the small fruits, the progeny of the mother variety Da baj czao, besides the smallest mass of the seed (0.43 g), had almost the lowest percent of the fruit randman, excluding the Ja czao progeny (Table 1)

Table 1. Fruit characteristics of the varieties Zu tao czao, Da baj czao, Ja czao and their progenies

Variety/	Fruit	Density	Stone	Randman	Variety/	Fruit	Density	Stone	Randman	Va	riety/	Fruit	Density	Stone	Randman
Туре	mass		mass		Туре	mass		mass		Тy	pe	mass	(g/cm^3)	mass	
•	(g)	(g/cm ³)	(g)	(%)		(g)	(g/cm^3)	(g)	(%)			(g)		(g)	(%)
Zu tao)				Da baj					Ja					
czao	9.5	0.613	0.61	93.6	czao	4.8	0.814	0.31	93.5	cza	ao	6.0	0.508	0.47	92.2
$\overline{\mathbf{X}}$					$\overline{\mathbf{X}}$					X					
P 10/1	9.4	0.537	0.34	96.4	P 13/2	4.9	0.636	0.59	88.0	Р	12/3	7.9	0.738	0.81	89.7
11/1	3.8	0.691	0.39	89.7	14/2	9.1	0.448	0.39	95.7		13/3	4.5	0.957	0.56	87.6
13/1	4.1	0.539	0.50	87.8	15/2	3.1	0.660	0.32	89.7		14/3	10.0	0.621	0.73	92.7
R 15/1	4.1	0.774	0.35	91.5	R 17/2	5.9	0.602	0.37	93.7	R	15/3	4.7	1.000	0.52	88.9
16/1	7.2	0.571	0.58	91.9	18/2	8.0	0.650	0.55	93.1		16/3	6.4	0.552	0.31	95.2
17/1	10.7	0.669	0.69	93.6	19/2	7.1	0.607	0.45	93.7		17/3	7.4	0.500	0.50	93.2
0 18/1	6.4	0.621	0.53	91.7	0 21/2	7.1	0.634	0.32	95.5	0	19/3	4.5	0.682	0.38	91.6
20/1	12.3	0.515	0.64	94.8	22/2	4.7	0.734	0.35	92.6		20/3	10.7	0.530	0.57	94.7
21/1	8.9	0.622	0.86	90.3	23/2	6.0	0.588	0.34	94.3		21/3	11.5	0.532	0.56	95.1
G 22/1	11.7	0.539	0.33	97.2	G 25/2	7.6	0.598	0.49	93.6	G	22/3	8.2	0.621	0.79	90.4
23/1	10.0	0.546	0.46	95.4	26/2	6.3	0.578	0.43	93.2		23/3	6.2	0.569	0.56	91.0
25/1	9.0	0.621	0.40	95.6	28/2	8.2	0.603	0.45	94.5		24/3	11.4	0.543	0.72	93.7
E 26/1	8.9	0.574	0.50	94.4	E 29/2	4.7	0.553	0.38	91.9	Е	25/3	5.4	0.505	0.39	92.8
27/1	3.5	0.729	0.38	89.1	3/2	10.9	0.548	0.71	93.5		26/3	12.9	0.527	0.96	92.6
28/1	10.2	0.545	0.54	94.7	30/2	5.0	0.658	0.63	87.4		28/3	7.1	0.511	0.56	92.1
N 29/1	4.7	0.566	0.40	91.5	N 31/2	6.7	0.549	0.46	93.1	Ν	30/3	11.4	0.579	0.89	92.2
32/1	7.1	0.573	0.74	89.6	32/2	6.1	0.616	0.39	93.6		31/3	7.5	0.429	0.55	92.7
33/1	8.4	0.542	0.42	95.0	33/2	4.6	0.590	0.46	90.0		32/3	9.7	0.487	0.63	93.5
Y 34/1	7.1	0.483	0.77	89.2	Y 35/2	8.2	0.509	0.45	94.5	Y	33/3	5.7	0.588	0.37	93.5
35/1	8.0	0.650	0.46	94.3	36/2	8.0	0.615	0.42	94.8		37/3	7.0	0.673	0.64	90.9
37/1	13.4	0.472	0.59	95.6	37/2	4.8	0.667	0.45	90.6		38/3	10.4	0.443	0.61	94.1
6/1	5.4	0.581	0.42	92.2	6/2	6.8	0.544	0.25	96.3						
7/1	5.4	0.635	0.39	92.8	7/2	5.4	0.593	0.47	91.3						
8/1	3.1	0.738	0.33	89.4	8/2	4.5	0.703	0.43	90.4						
					9/2	3.1	0.674	0.36	88.4						
Туре	s T	0.5(7	0.50	02.4	Types	· ()	0 (00	0.42	02.2		Types	0.1	0.5(2	0.00	02 (
X	/.0	0.30/	0.50	93.4	$\overline{\mathbf{X}}$	0.3	0.000	0.43	93.2		X	ð.1	0.503	0.00	92.0
CV%	= 39	14	30	2	CV% =	= 30	11	23	3	C١	/% =	32	29	28	2
Lsd 0.05	= 2.9	0.088	0.17		Lsd _{0.05} =	= 2.3	0.328	0.18		Ls	d _{0.05} =	3.5	0.152	0.30	
Lsd 0.01	= 3.8	0.117	0.23		Lsd _{0.01} =	3.1	0.437	0.23		Ls	d _{0.01} =	4.6	0.202	0.39	

A lot of variations existed in ber genotypes for pulp/stone ratio. The pulp/stone ratio was maximum in Sanur-6 and minimum in Punjab Chhuhara [7]. The pulp/stone ratio was the highest in Umran [1]. The same authors reported the highest pulp/stone ratio in HB-2, a wild local strain of *Z. jujuba*.

The mother variety of Ja czao produced progeny that on average insignificantly exceeded the mother statistically in terms of the fruit mass (8.1 g) (Table 1). Actually, as a medium large fruit variety, Ja czao gave progeny with big fruits, similar to the types in the progenies of the varieties Kitajski 2A (8.9 g) and Vahshski 45/2 (8.1 g) which had the largest fruits. As well as in the other investigated progenies, the coefficient of variation in this progeny was the highest regarding the mass of the fruits (32 %).

The type 26/3 was characterized with the largest mass of the fruits (12.9 g), which statistically very significantly deviates in relation to the fruit

mass of the mother variety Ja czao fruits. The type 26/3 statistically showed very significantly larger mass of the fruits than 62 % of the types in progeny. The type 16/3 was characterized with the smallest seeds (0.31 g), and with the highest randman of the fruits (95.2 %) from progeny (Table 1). In the progeny of the variety Ja czao the types 15/3 (1.000 g/cm³) and 13/3 (0.957 g/cm³) were recorded, with significantly bigger fruit density compared to types from all investigated progenies.

In the progeny of the mother form Wild Midleasiatic jujube, which statistically had the significantly biggest fruits, compared to the mother (5.2 g) and the average of the progeny (7.4 g), the type 35/4 (13.3 g) was characterized. The type 14/4 had statistically very significantly larger fruit density (0.909 g/cm³) than the average of the progeny (0.548 g/cm³) (Table 2).

Table 2.	. Fruit char	acteristics	of the	varieties	Wild M.	jujube,	Vahshski 4	45/2,	Kitajski	i 2A an	d their	progenies
						JJ /						1 0

Va	riety/	Fruit	Density	Stone	Randman	Va	riety/	Fruit	Density	Stone	Randman	Variety	/ Fruit	Density	Stone	Randman
Туј	be	mass	(g/cm ³)	mass	(%)	Туј	pe	mass	(g/cm ³)	mass	(%)	Туре	mass	(g/cm ³)	mass	(%)
		(g)		(g)				(g)		(g)			(g)		(g)	
W	ild M.					Va	hshski	i				Kitajsk	i			
jt	ıjube	5.2	0.813	0.32	93.8	4	45/2	11.7	0.534	0.56	95.2	2A	13.1	0.652	1.01	92.3
(X)					($(\overline{\mathbf{X}})$					(X)				
Р	10/4	4.6	0.821	0.45	90.2	Р	11/5	7.1	0.555	0.57	92.0	P 13/	5 3.4	0.618	0.33	90.3
	13/4	5.5	0.611	0.39	92.9		15/5	5.9	0.678	0.44	92.5	14/	5 7.1	0.534	0.54	92.4
	14/4	5.0	0.909	0.28	94.4		16/5	4.9	0.671	0.47	90.4	21/	5 22.2	0.496	0.62	97.2
R	16/4	1.8	0.692	0.18	90.0	R	17/5	12.7	0.474	0.62	95.1	R 22/	5 16.0	0.500	0.84	94.8
	17/4	9.4	0.635	0.59	93.7		18/5	8.0	0.615	0.54	93.3	24/	5 10.5	0.430	0.62	94.1
	18/4	5.6	0.571	0.42	92.5		19/5	7.7	0.527	0.40	94.8	31/	5 5.8	0.537	0.47	91.9
0	19/4	6.0	0.561	0.56	90.7	0	23/5	8.0	0.630	0.42	94.8	0 33/	5 4.6	0.697	0.37	92.0
	21/4	11.1	0.603	0.56	95.0		24/5	8.8	0.583	0.65	92.6	37/	5 3.9	0.780	0.51	86.9
	22/4	6.5	0.556	0.63	90.3		25/5	15.7	0.506	0.55	96.5	40/	5 8.6	0.562	0.49	94.3
G	23/4	8.0	0.556	0.54	93.3	G	26/5	9.4	0.452	0.93	90.1	G 41/	5 10.3	0.493	0.57	94.5
	24/4	8.3	0.447	0.43	94.8		27/5	4.5	0.600	0.37	91.8	9/6	5.8	0.592	0.53	90.9
	25/4	6.0	0.667	0.45	92.5		31/5	6.2	0.466	0.42	93.2					
Е	26/4	7.7	0.503	0.63	91.8	Е	32/5	10.2	0.505	0.37	96.4	Е				
	27/4	9.6	0.397	0.60	93.8		33/5	4.7	0.734	0.49	89.6					
	28/4	7.9	0.485	0.79	90.0		35/5	8.2	0.577	0.57	93.0					
Ν	29/4	11.8	0.509	0.62	94.7	Ν	38/5	6.1	0.610	0.34	94.4	Ν				
	30/4	4.6	0.505	0.51	88.9		6/5	9.2	0.495	0.54	94.1					
	31/4	6.0	0.492	0.33	94.5		7/5	9.3	0.472	0.62	93.3					
Y	32/4	5.9	0.457	0.43	92.7	Y						Y				
	34/4	8.8	0.444	0.32	96.4											
	35/4	13.3	0.591	0.70	94.7											
	37/4	9.1	0.448	0.41	95.5											
-	Types						Types					Тур	es			
	X	7.4	0.548	0.49	93.4		X	8.1	0.523	0.51	93.7	X	8.9	0.511	0.54	93.9
(CV% =	36	25	30	2	С	V% =	35	17	27	2	CV%	= 64	15	25	3
Ι	_sd _{0.05} =	2.3	0.203	0.20		L	.sd _{0.05} =	3.8	0.159	0.27		Lsd 0.05	= 2.9	0.076	0.15	
Ι	.sd _{0.01} =	3.0	0.270	0.26		L	.sd _{0.01} =	5.1	0.212	0.36		Lsd 0.01	= 3.9	0.101	0.19	

In fact, Wild Midleasiatic jujube was characterized with a larger variation of fruit density in the progeny (Table 2), compared with other progenies. Significant genotypic differences were found in the seed characters of *Z. jujuba* varieties [1]. Saran [5] observed variations in the stone size of 35 cultivars of ber and reported that maximum (3.72 cm) was in Ponda and the minimum (0.53 cm) in Illaichi. The type 16/4 had statistically very significantly smaller seed than 59 % of the types in the progeny (0.18 g), showing also significant differences from the average of the types (0.49 g) in the progeny (Table 2).

The mother variety Vahshski 45/2 belonged to the group of jujube varieties with large fruits. Its progeny was characterized with large fruits (8.1 g) on average, but statistically insignificantly smaller fruits than the mother variety Vahshki 45/2 (11.7 g). According to the dimensions of the fruits in the progeny, the Vahshki 45/2 was just behind the variety Kitajski 2A. The type 25/5 from its progeny was characterized with the largest fruits.

The fruits of this type were statistically very significantly larger (15.7 g) compared with the average of the progeny (8.1 g) and also with the fruits of the mother variety (11.7 g) (Table 2). It can be noted, that the average of the types from this progeny, estimated for all investigated characteristics except fruit mass, was most uniform with the mother variety, unlike other mother varieties (Table 2).

The best *Zizyphus jujuba* varieties for uniformity of fruit size were Ta-Pai and Hsueh-pai [2]. The type 38/5 was characterized with smaller seeds without statistically significant difference in terms to the mother variety and the average of the progeny. The seeds of *Zizyphus mauritiana* trees taken from five districts in Yunnan Province, China and Narkum, Myanmar, were tested for their morphological characteristics, germination characteristics, and the growth patterns of the seedlings and young trees. A close relationship was found between these characters and their geographical distribution and climatic conditions [8].

The progeny of the Kitajski 2A mother variety (although few in number) was characterized with exceptional examples. About 36 % of the types from its progeny had larger fruit mass than 10 g. The type 21/6 was characterized with the largest fruit mass (22.2 g). Its fruits in average were statistically very significantly larger not only from those ones of its mother (Kitajski 2A) which had the largest ones between investigated varieties, but also from all types which were obtained from the progenies. The goal of the investigation was actually accomplished with this type, producing types that would be better than their mother varieties. In this progeny another type 22/6 was obtained with statistically significantly larger fruits (16 g), than the mother variety and in relation to the other mother varieties and types in the progenies.

Pewandi was considered to be the best variety for commercial cultivation, having the largest fruit, a high percentage of edible pulp and good skin colour [1]. There were considerable differences among seven ber cultivars in fruit weight (29.340 to 9.544 g), fruit size (length 3.27-4.33 cm), pulp/seed ratio and fruit quality in Ratta Kulachi, D. I. Khan, Pakistan [1]. Even in an early stadium of hybrid seedling development (in the first or second year) the genotypes with large fruits size could be registered, especially in the third fruit growth period, and that was very useful for an early positive selection of the Chinese jujube perspective types [9].

The type 21/6 was characterized with the highest randman (97.2 %) among the varieties and types (Table 2). The mother variety Kitajski 2A had seeds with statistically very significantly higher mass (1.01 g) compared with other varieties and types in the progenies. The type 37/6 was characterized with statistically very significantly larger fruit density (0.780 g/cm³) than its mother (0.652 g/cm³) and compared with the average of the progeny (0.511 g/cm³). A very significant difference was recorded in the mass of the seeds of the type 13/6 which had the smallest seeds (0.33 g), compared with the mother variety (1.01 g) and the average of the progeny (0.54 g) (Table 2).

The determination of the genetic parameters clarifies the investigated characteristic variation in progenies (Table 3). The genotypic coefficient of variation (GCV) is the highest for fruit and seed mass compared with the other investigated characteristics (fruit density and randman), but the phenotypic coefficient of variation (PCV) for these characteristics was also higher, which indicates the stronger influence of the environmental factors, especially this refers to the characteristics of fruit mass. The range of genetic variability of the seed mass in each of the progenies was higher than GCV of all progenies which indicates a stronger mother influence on inheritance. The progenies of the mother varieties Vahshski 45/2 and Ja czao had the highest GCV for fruit and seed mass than the other varieties (Table 3). The jujube progenies had the lowest GCV for the randman characteristics of the fruit, with an exception to the progeny of the variety Da bai czao. The estimates showed moderate heritability (H²) for all investigated characteristics. The high heritability, coupled with high genetic advance (GA), was noted for progenies' fruit mass. It especially relates to fruit mass of the Kitajski 2A progeny (H² 64.5 % and GA 39.7 %), which means that in this variety it is much easier to obtain inheritance with high fruit mass genotypes through further individual selection. The higher are the values of GCV, heritability and genetic advance (GA) for

some characteristics, the same characteristics can be more improved through the selection process [10]. However, moderate to low heritability and genetic advance suggested a polygenic influence and quite weak additional gene effect (Kitajski 2A) on the creation of the Chinese jujube fruit characteristics (Table 3).

Table 3. G	enetic parameters	s for some	fruit chara	cteristics o	of the	jujube	genotypes
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	Characteristics range	Genetic parameters			
Variety progenies	(min-max)	*GCV (%)	*PCV (%)	*H ² (%)	*GA (% of mean)
Fru	it mass (g)			. ,	
Zu tao czao	2.5-17.2	19.0	38.4	54.0	20.1
Da bai czao	2.1-13.0	18.3	34.2	49.6	16.9
Ja czao	3.5-15.4	21.0	37.0	41.8	15.0
Wild jujube	0.9-15.2	15.4	35.0	57.9	19.7
Vahsh.45/2	4.2-17.1	23.5	44.1	44.3	19.0
Kitajski 2A	2.9-24.0	16.2	63.7	64.5	39.7
All progenies	0.9-24.0	16.6	24.6	57.3	15.0
Fru	it density (g/cm ³)				
Zu tao czao	0.45-0.97	7.3	14.1	51.1	7.0
Da bai czao	0.39-0.98	13.2	22.1	40.0	8.7
Ja czao	0.42 - 1.27	11.7	27.4	58.5	15.6
Wild jujube	0.34-1.34	17.2	30.9	44.7	13.4
Vahsh.45/2	0.42-0.92	13.5	24.6	41.2	9.8
Kitajski 2A	0.44-0.84	6.6	17.9	56.9	9.9
All progenies	0.34-1.34	12.7	17.4	48.0	7.7
Seed mass (g)					
Zu tao czao	0.17-1.05	17.4	32.5	48.8	15.4
Da bai czao	0.18-0.76	19.4	31.4	35.4	11.0
Ja czao	0.17-1.20	24.3	38.0	26.4	9.7
Wild jujube	0.14-0.87	20.2	36.1	43.9	15.4
Vahsh.45/2	0.25-1.17	26.7	42.6	24.7	10.2
Kitajski 2A	0.3-0.95	13.6	32.3	51.3	16.1
All progenies	0.14-1.20	4.3	14.5	62.8	9.2
R	andman (%)				
Zu tao czao	84.5-97.3	1.4	2.3	55.0	1.5
Da bai czao	83.3–97.2	9.2	18.0	52.7	9.2
Ja czao	84.5-97.4	1.8	2.9	33.2	0.9
Wild jujube	82.2-97.4	1.7	3.0	42.4	1.2
Vahsh.45/2	88.7-97.1	1.1	2.3	52.7	1.2
Kitajski 2A	Kitajski 2A 81.4–98.0		3.9	53.6	2.0
All progenies 81.4–98.0		1.6	2.2	46.6	1.0

GCV – Genotypic coefficient of variation, PCV – Phenotypic coefficient of variation,

H² – Heritability, GA – Genetic advance

CONCLUSIONS

The investigated characteristics: fruit density and mass of the seed in the progenies of jujube varieties showed that they were low dependent from the mass of the fruit. Namely, the jujube types with small fruits statistically had insignificantly higher fruit density, but also the higher coefficient of variation of fruit density in relation to the types with large fruits. Also, the jujube types with large fruits had again statistically insignificantly higher mass of the seed, with high coefficient of variation in relation to the types with small fruits. The sublimated investigations showed that the mother varieties with medium to large fruits (Kitajski 2A, Vahshski 45/2, and Zu tao czao) had on average bigger mass of the fruit, mass of the seed and fruit density than the average of the types in their progenies, while the varieties with small fruits (Ja czao, Da baj czao, Wild Middleasiatic jujube) had only bigger fruit density than the average of the types in their own progeny. The investigations showed that within the frames of jujube species it was very difficult to obtain types with very large fruits, but genetic parameters indicated that there were more chances to get such examples from the seeds of varieties and types with large fruits, so I recommend to use open pollination or hybridization between varieties or types with large fruits for obtaining types with larger fruits in the future breeding programs.

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КАРАКТЕРИСТИКИ НА ПЛОДОВИТЕ НА РАЗЛИЧНИ ПОТОМСТВА ОД КИНЕСКА УРМА (*ZIZYPHUS JUJUBA* MILL.)

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Во периодот 2002–2004 во опитните насади на Земјодлескиот институт во Скопје се испитувани помолошките карактеристики на пет сорти и еден див тип на кинеска урма. Семето од сортите: Жу тао цзао, Да бај цзао, Китајски 2А, Ја цзао, Вахшски 45–2 и од типот Дива средноазиска е добиено по пат на слободно опрашување. Кај 7–9 годишните самоници кинеска урма добиени од тоа семе, кои се посадени во селекционо поле, се испитувани некои карактеристики на плодовите: маса на плодовите (g), густина на плодовите (g/cm³), маса на семето (g) и рандманот (%). Доминантен мајчин ефект во наследувањето е забележан кај масата и густината на плодовите. На пример: со најголема маса на плодовите се карактеризира сортата Китајски 2А и типовите од нејзиното потомство, додека со најголема густина на плодовите се карактеризира мајчинската сорта Да бај цзао и типовите од нејзиното потомство. Најголема маса на семето е забележана кај сортата Китајски 2А, додека со просечно најголема маса на семето се карактеризира лотомството од сортата Китајски 2А, додека со просечно најголема маса на семето се карактеризира лотомството од сортата Китајски 2А, и цзао.

Клучни зборови: Кинеска урма; слободно опрашување; сорта; тип; плод

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Review

REVIEW OF THE RESEARCH IN CROP WATER REQUIREMENT AND ITS USE IN THE REPUBLIC OF MACEDONIA

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The paper presents a review of the research activities dealing with evapotranspiration (ET), crop water requirement (CWR) and the use of these parameters in other research, particularly in climate change. The first part discusses the development of these research methodologies globally. Later the achievements of the national research are discussed. Macedonia did not developed capacities for measuring of the ET and the crop water requirement. There is only one practice which is followed – field experiments for the assessment of the water balance with bucket approach. The estimation of evapotranspiration is based mainly on the Penman – Monteith FAO 56 procedure. Due to the lack of input data the less data intensive Thornthwhaite methodology is applied. In the country the ET and the crop water requirement are used in irrigation projects, in research activities and in the assessment of the effects of water limitation on crop yield, particularly in climate change. Recently some activities dealing with the use of crop biophysical models WOFOST and CropSyst are taken.

Key word: evapotranspiration, crop water requirement, climate change, research, Republic of Macedonia

INTRODUCTION

The climate determines agricultural productivity. The agricultural production is inextricably linked to climate that makes agriculture the most sensitive economic sector to climate and weather variations, and consequently to climate change.

The temperature drives the crop growth. The global warming in the last century was almost 1°C, and the first decade of the 21^{st} century was the warmest recorded in history. The global warming is evident. One of the factors that elevates global temperature is the increase of the atmospheric CO₂ concentration. The atmospheric CO₂ concentration rose from pre-industrial 280 ppm to 402.6 ppm in January 2016 (reported as global average by Earth System Research Laboratory of the National Oceanic and Atmospheric Administration – NOAA). In the last year the increase of the CO₂ concentration was for 3 ppm, and it is expected that the rise of the global temperature will continue. However, the

global warming will affect the precipitation as well. The changes in precipitation are observed and rainfall has increased in the mild latitudes of the North hemisphere. Most of the scenarios for the Republic of Macedonia show a decrease of the annual rainfall. According to the A1B scenario of the Geophysical Fluid Dynamics Laboratory Climate Model 2.1 (US) the slight decrease of the rainfall is expected in the period 2010–2020. After that a period with more significant drop of the rainfall is expected (Sutton *et al.* [1]).

The changing climate influences the crop and livestock production. Nevertheless, the effects of these biophysical changes and particularly the human response to these changes are very complex and uncertain. Besides the increased temperature, the elevated CO₂, fertilization effect promotes the crop growth. But it is important to take Liebig's law of the minimum into consideration to understand the effect of the global changes. These changes should be analyzed from the point of the most limiting factor in the production and in Macedonia this is the water limitation. The yield in most of the agricultural areas in the country is limited by water. Due to this limitation about ¼ of the cultivated land is equipped for irrigation. Unfortunately the irrigated area dropped from more than 80 thousand hectares in the 1980s to about 30 thousands ha nowadays. The climate change will also cause more frequent appearance of extreme events as drought, floods, heat waves, etc. It is expected that these extreme events, in combination with present water limitation, will further increase the negative effects on the agricultural productivity in the country.

Due to this the researchers in the country focus their attention to the effect of water limitation on the crop yield in present and future climatic conditions. This paper aims to present the current research efforts in Macedonia to address the ET, crop water requirement and crop yields in present and future climatic conditions.

Reference evapotranspiration and crop water requirement

Basically crop water requirement is ET adjusted for crop species and growth stages. Various methods for estimation of evapotranspiration are in use. Direct measurement is considered as one of the best practices, particularly the use of weighted lysimeters (Mustonen and McGuinness [2], Hargreave [3]; Pruitt and Lourence [4], Howell *et al.* [5]; Liu *et al.* [6]; López-Urrea *et al.* [7]; Toyin *et al.* [8]). The weighted lysimeters are in use for a long period of time and still considered to be the most accurate way to estimate crop water use and develop crop specific coefficients. Therefore they are regarded as a standard for determination of the ET. The weighted lysimeters measure the changes in mass of a soil container planted with investigated crop and these changes are representing water used in a certain period. Even though the weighted lysimeters are in use since the late 1930s, they are still used for testing, and comparing of the results from the new practices as Bowen ratio, Eddy covariance, remote sensing, etc. (Dugas et al. [9]; Barr et al. [10]; Wolf et al. [11]; Gebler et al. [12]; Reginato et al. [13]; Chávez et al. [14]; Cruz-Blanco et al. [15]). Even more, the lysimeters are used for development of new empirical methods for estimation of ET and calibration of the empirical and biophysical models for estimating of the ET (Abtew and Obeysekera et al. [16]; Kashyap and Panda et al. [17]; Marsal et al. [18]; Reddy [19]). Finally lysimeters are used for operational irrigation scheduling.

The various authors were developing empirical methods for estimation of ET (Thornthwaite [20]; Penman [21]; Blaney et al. [22]; Blaney and Criddle [23]; Harbeck [24]; Priestley and Taylor [25]; Hargreaves [26]; Hargreaves and Samani [27], etc.). The existing methods for the estimation of potential ET are based on high correlation of ET with some measurable parameters as temperature (Thornthwaite [20]; Blaney and Criddle [22]; Hargreaves [26]; Hargreaves and Samani [27]), radiation (Priestley and Taylor [25]; Jensen and Haise [28]), mass-transfer (Harbeck [24]) or combination of some of these parameters (Penman [21]).

Table 1. The methods for estimation of the ET and measurable parameter correlated with ET

Mathed for activation of FT	Method based on correlation with			
Method for estimation of E1	temperature	radiation		
Penman-Monteith	Х	Х		
Hargreaves/Hargreaves Samani	Х			
Thornthwaite Method	Х			
Blaney-Criddle Method	Х			
Priestley-Taylor	Х	Х		
Makkink Method		Х		
Turc Method		Х		
Jensen and Heise		Х		

The Penman method modified by Monteith [29] became a standard for estimating of reference evapotranspiration (ETo) particularly due to the work of Doorenbos and Pruitt [30] when they proposed the Penman-Monteith procedure as one of the 4 standard methods for estimation of reference evapotranspiration (defined as ET rate from a reference surface, not short of water). The reference surface is a hypothetical grass reference crop with specific characteristics. The reference evapotranspiration is considered as a climatic parameter and can be computed from weather data, and only weather data can affect it. Moreover, the FAO consultation process resulted in a proposal for revision of the methodology proposed by Doorenbos and Pruitt [30] which led to the publication of the FAO Irrigation and drainage paper 56 entitled "Crop Evapotranspiration - Guidelines for computing crop water requirements" (Allen et al. [31]). With these guidelines a hypothetical reference crop was defined with assumed crop height of 0.12 m, a fixed surface resistance of 70 s m⁻¹ and an albedo of 0.23. The FAO Penman-Monteith method is selected as a reference method by which the ET of this reference surface (ETo) can be unambiguously determined, and as a method which provides consistent ETo values in all regions and climates. Moreover, the American Society of Civil Engineers (ASCE) conducted extensive consultations on standardization of the procedures for ET estimates. As a result of this process Walter et al. [32] recommended two reference crops (short similar to grass and high similar to alfalfa) and ASCE Penman-Monteith (ASCE-PM) equation with some simplifications as a standard method. Later an extensive comparison of the number of methods for estimation of the ETo was conducted by Itenfisu et al. [33]. The researchers used hourly and daily weather data from 49 geographically diverse sites in the United States. Calculations were performed for both grass and alfalfa reference crops in a consistent manner, using weather data that passed integrity and quality assessment checks. Comparisons were made between ETo computed by various methods and the ASCE-PM equation for a daily calculation time step. Results showed that the ASCE standardized equation agreed best with the full form of ASCE-PM and provided a basis for an objective assessment of the relative performance of reference ET equations in a variety of climates and supported adoption of a standardized equation.

From reference evapotranspiration to crop water requirement

The reference evapotranspiration is exclusively a climatic parameter and should be converted into the crop evapotranspiration (ETcrop). The FAO 24 methodology (Doorenbos and Pruitt [30]) recommended the use of the growth stage specific crop coefficient (kc) to relate ETo to ETcrop in order to account for the effect of the crop characteristics on crop water requirements. Besides climate

and climate induced variations of the reference evapotranspiration, the ETcrop is affected by numerous factors that further influence crop water requirement, such as: crop phenology, cultural practices, irrigation method, soil water availability, etc. Therefore the FAO organized an expert consultation that was held in May 1990 in Rome and established a working group for revision of the FAO 24 methodology. This working group revised the methodology (Allen et al. [34]; Allen et al. [31]). This revision included modified procedures for estimating crop coefficients (kc). The single and dual crop coefficients were introduced for estimation of crop water requirement in standard condition. Moreover, the detailed procedures were provided for non-standard condition through adjusting of the crop coefficient. This procedure became a standard for estimation of the ET and crop water requirement for irrigation projects and many others applications. The work on calibration of the FAO 56 and growth stage specific crop coefficients for various crop in different climatic conditions is still ongoing in many environments (Yang et al. [35]; Tian et al. [36]; Aamlid et al. [37]; Muniandy et al. [38]; Campos et al. [39]; Paparrizos et al. [40]).

RESEARCH IN EVAPOTRANSPIRATION AND CROP WATER REQUIREMENT IN THE REPUBLIC OF MACEDONIA

Determination of the evapotranspiration and crop water requirement

The measurement of the ET and crop water use in Macedonia is still far away from modern scientific achievements. Unfortunately in the Republic of Macedonia there are not any weighted lysimeters installed. Also there is not any other lysimetric type used in practice. The cost of installation of the weighted lysimeter field is overcoming the capacities in the country. Unfortunately, after the transition period it was quite difficult for the scientific community to keep pace with modern technologies used for determination of ET such as remote sensing, Bowen ratio, Eddy covariance, etc. Nevertheless, the Macedonian researchers are doing their best to determine ET and get information on crop water requirement of various crops in the country. The first paper on measurement of ET and crop water requirements was published by Kosevski [41]. This paper introduced the experimental determination of the ETcrop using the soil water budget practice, particularly using the bucket approach (assuming that there is not flux of water from and into the root zone). This practice still remains as a standard method to determine ET in the country. The paper presented data on crop water requirement of maize planted in two regions (Skopsko Pole and Polog). Further this practice was used by Iljovski [42] when he determined crop water requirement and water use of hop in various regimes of water supply in Pelagonia. Furthermore Iljovski et al. [43] presented the effect of irrigation technique on crop water use. Iljovski and Chukaliev [44] presented crop water requirement for sunflower cultivated as second crop using the same experimental method for determination of the ET, crop water requirement and irrigation water requirement. The similar research was carried out by Chukaliev and Iljovski [45] that presented results on crop water use of maize cultivated as second crop. Iljovski et al. [46] conducted research on the determination of the crop water use for sugar beet cultivated as second crop, using same methods. Furthermore Chukaliev and Iljovski [47] published a work on a three-year experiment for determination of water consumption of sugar beet irrigated by micro-sprinkler irrigation, sprinkler irrigation and furrow irrigation. Their results demonstrated that similar ET could result in different yields if water was applied with different irrigation techniques. Therefore the micro-sprinkler irrigation that increased air humidity alongside of soil moisture, achieved higher yield even though the ET was similar to sprinkler irrigation one. The authors concluded that if water was applied in small portions several times per day, the water use efficiency of the sugar beet could be increased. Based on previous work, Iljovski and Chukaliev [48] presented initial results on crop water requirement and water use of sugar beet by applying different irrigation techniques (pulse irrigation, sprinkler and furrow, compared with non-irrigated treatment). Later, Chukaliev [49] presented final results on the crop water requirement, water use and water use efficiency of sugar beet planted in Skopsko Pole with particular emphasis on pulse irrigation (application of fine drops of water in very small portions several times per day). This work proved that even though ET was determined by meteorological conditions, irrigation could change some of these parameters within the crop canopy (decrease of temperature, increase of air humidity) and reduce crop water requirement. The pulse irrigation resulted in higher yield and sugar content with smaller/similar use of water as sprinkler and furrow irrigation. Iljovski and Chukaliev [50] conducted research on the crop water requirement for the young apple orchard and reported much lower crop water requirement than in full developed orchards. Iljovski et al. [51] conducted research on comparison of crop water use

with application of furrow and drip irrigation and confirmed that tomato crop under drip irrigation used water more efficiently in comparison with furrow irrigation. Furthermore, Iljovski and Chukaliev [52] presented results on crop water use for tomato crop irrigated by drip irrigation, by combination of micro-sprinkler and drip irrigation and by furrow irrigation. The tomato irrigated by drip irrigation used just two thirds of the water used in furrow irrigation and achieved 30% higher yield. The sporadic intervention with micro-sprinklers did not increase the yield, but had an effect on earlier maturity. The very similar concept for the determination of the crop water use was used in the research of Iljovski and Chukaliev [53]. They presented results for ET and crop water use for alfalfa irrigated by pulse irrigation and by sprinkler irrigation and concluded that pulse irrigation used less water (by almost 30%) and achieved higher yield (by around 20%). Moreover, the similar type of experiment was conducted in the research on the effects of water conservation on crop water use. Iljovski et al. [54] presented results that different types of soil mulch (polyethylene and straw) affected crop water use. The highest water use was observed in not mulched treatment, while the lowest in the treatment with polyethylene mulch. The authors reported that in the case of polyethylene mulch higher root density was recorded in the top soil layers that resulted in over extraction of water from these layers while straw mulch resulted in higher water consumption from deeper soil layers. The mulching conserved water in the soil and resulted in lower crop water use. Chukaliev and Iljovski [55] published their new results on comparative research of water use of tomato crop using drip and furrow irrigation. Once again they recorded higher water use efficiency of tomato when drip irrigation was used.

Later Jankulovski et al. [56] presented results from the field experiments they carried out for determination of ET under different irrigation regimes. Their research was particularly oriented to water use efficiency. The highest water use efficiency was determined when irrigation water was applied at depletion level of 50% of total available water (TAW) of 66 l/kg. Similar result of 68 l/kg was achieved when irrigation water was applied according to the soil water balance as recommended by Iljovski [57], Cukaliev [58], Cukaliev and Iljovski [59] and Chukaliev and Iljovski [60]. Other treatments used the water less efficiently. Jankulovski [61] presented additional results of the effect of irrigation regime and amount of fertilizers on sugar beet yield. The highest yield was achieved when water was applied at 50% of TAW followed by irrigation according to

the water balance with yield lower by 7%. Also they proved that the increase in the quantity of fertilizers in these treatments resulted in higher yield and increased water use efficiency.

Tanasković V. [62] continued the practice to use field experiments for determination of ET by application of the water balance method. Unlike previous authors his experiments were based on the use of drip irrigation resulting in substantially improved control of applied water. Moreover, he applied same amount of water for each treatment based on daily ET. The author used different frequency of irrigation/fertigation (every 2 days, every 4 days and every 6 days). The comparison of the achieved results was done with treatment irrigated every 4 days, but fertilizers were applied in granular form and incorporated into the soil. All fertigation treatments compared with classical fertilization practice achieved higher yield. Also treatments of the applying water and fertilizers every 2 and 4 days achieved significantly higher yield compared with 6 days frequency of fertigation. The yields in fertigation treatments were very high, about 120-150 t/ha. The same water amount (same ET) yielded in different productivity of the tomato crop, just due to the frequency of application of water which should be considered as one of the factors that affected crop water requirement and water use efficiency. Later, work of Tanaskovik V et al. [63] and [64] presented additional explanation of the findings of Tanasković V. [62].

Chukaliev O *et al.* [65] presented results on nitrogen use efficiency under different irrigation regimes and found that irrigation regime affected the nitrogen uptake by the tomato crop. They used N¹⁵ labelled nitrogen fertilizer and provided data that increased water use efficiency reflected on increased nitrogen use efficiency. The work of Jankulovski *et al.* [61], Tanasković [62], Tanasković *et al.* [63] and [64] and Chukaliev *et al.* [65], provided valuable data for future work on calibration of ET based on fertilization level and fertilizers and water use efficiency.

Tanasković [66] presented results on use of soil water balance for determination of evapotranspiration on pepper crop. The basis for irrigation scheduling was the daily evapotranspiration calculated by the FAO 56 procedure. He used 3 drip fertigation treatments (every 2 days, every 4 days and irrigation based on tensiometric measurement of soil water potential). The comparison was done with furrow irrigated pepper crop. The crop evapotranspiration in his research was about 490 mm when using drip irrigation scheduled according to daily evapotranspiration, 510 mm when using tensiometers and 590 mm when using furrow irrigation. The highest yield was recorded in 2 days fertigation scheduling of about 71 t/ha that was significantly higher than in both treatments using higher amount of water (scheduling by tensiometers and furrow irrigation).

Next several papers published by Tanasković *et al.* [67], [68], [69] and [70] and Chukaliev *et al.* [71] contributed to the work of Tanasković [62] and [66] and presented a valuable source of data for calibration and validation of crop models, but did not bring any new research on the use of evapotranspiration and crop water requirement.

Probably the research of Tanasković [66] was the last attempt for direct determination of the crop water use because in the last period of time there was almost no financing of the national research. This type of research activities is quite obsolete and it is very difficult to get international grants that will support projects based on this methodology. It is even harder to get financial support for constructing weighted lysimeters or equipment for more advanced measurement of evapotranspiration and crop water requirement. Building of lysimeters is very costly. Even when they are constructed, researchers interested in this issue can not provide sustainability of the lysimetric fields, particularly owing to the lack of national financing of the research activities. The problems are accelerated due to the fact that during the privatization the experimental fields of the scientific institutions became private entities, and got more interested in commercial production than in non-commercial use of their land in research purposes. Therefore researchers in the country are moving their interest to other fields, even though it is essential to have data on measured evapotranspiration and crop water requirement, particularly related to the changing climate. Although in the country there is a lack of recent experimental data on calibrating and validating models and other advanced research techniques, we can say that lately there has not been any research conducted in this issue as well.

Estimation of evapotranspiration and crop water requirement

The estimation of evapotranspiration was mainly used in the design purposes, and most of designers used the consumptive use equation developed by Blaney and Criddle [23]. This topic was not attractive for research during that period, and designers were using the coefficients derived from other countries. The research on the estimation of evapotranspiration and crop water requirement started in the early 1960s, when Petrovski [72] published his paper on water regime in Skopsko Pole. Later the use of estimation of the evapotranspiration and crop water requirement gradually improved. Cosevski and Popov [73] and Iljovski et al. [74] were trying to derive some calibration and to correlate temperature with water use, but these attempts were limited to one location. The first paper on the use of temperature correlated with crop water use was published by Iljovski [57]. He established correlation coefficients between crop water requirement and temperature for hop based on the extensive field experiments. Later, the same author (Iljovski [75]) presented the needs of estimating crop water requirement with methods that were calibrated for a given condition, particularly in designing larger scale accumulation for irrigation. The first attempt to develop model of the crop water requirement based on reduced input was done by Chukaliev [58]. He developed empirical model for balancing the soil water content based on daily values of the temperature and precipitation and applied it for simulation of irrigation of grain maize. Chukaliev and Iljovski [59] implemented similar model for simulating the crop water requirement for apricot grown in the area of the irrigation system Lisiche in Veles municipality in order to predict number of irrigations and application rate. Due to unavailability of meteorological data in the country, the authors used a reduced input dataset with just few parameters (temperature, rainfall and soil water properties). The authors recommended the use of such model for irrigation scheduling when only temperature and precipitation data are available. Chukaliev and Iljovski [60] used the same model as in their previous work on apricot for tomato crop and reported that models with reduced input of meteorological data can be used for irrigation scheduling if carefully calibrated with measured data on crop water requirement from experimental fields.

This early work did not take into consideration the various developed practices for estimation of the evapotranspiration. While the FAO 24 methodology was widely used in the world, the research in Macedonia was not able to keep pace with this processes mainly due to lack of measured data on wind and solar radiation. The other disadvantage was that this research was very site specific (conducted for one meteorological station and for crops that were in experimental fields and where data for calibration was available). One of the first attempts to estimate evapotranspiration and water requirement on the larger scale (country level) was conducted by Iljovski and Chukaliev [77]. Unfortunately this work was using aggregated data on evapotranspiration and rainfall on the country scale and can be used therefore only as orientation, because it did not provide proper spatial analysis of these important parameters. The more complex work on estimation of evapotranspiration all over the country was conducted by Filipovski *et al.* [78]. In this work the authors presented data for evapotranspiration and water availability for all main meteorological stations in the country. The method of choice was Thornthwaite [20] due to reduced data availability, but also this method is good for description of the climatic water balance for climatological work. Moreover, this work was conducted for determination of soil-climate-vegetation zones and therefore did not intend to be the source of data for crop water requirement for use in an irrigation project.

The first attempt to use the FAO 24 methodology and Penman-Monteith equation was made by Iljovski and Chukaliev [79]. Next year the same authors (Iljovski and Chukaliev [80]) presented the paper where they discussed methods for estimation of evapotranspiration and crop water requirement. They compared the methods for estimation of the evapotranspiration and recommend the FAO 24 Penman-Monteith procedure for use in the irrigation projects. This happened very late, in the same year when Allen et al. [31] proposed revision of the FAO24 procedures for estimation of evapotranspiration, and new procedures published in the FAO 56 guidelines for estimation of the evapotranspiration (Allen et al. [34]) were already appreciated. The late adoption of FAO 24 procedures was due to crisis in Former Yugoslavia and the long transition period after the independence of the Republic of Macedonia. In that period the access to new scientific literature was almost impossible and the financing of the research required for calibration of the crop specific coefficient was not available. In addition, the use of the procedures which are intensive in requirement of various meteorological data (as Penman-Monteith procedure is) was limited to the monthly step for several main meteorological stations where all required data was monitored. Unfortunately, the problem of transparency of meteorological data remains major problem in development of more advanced research in crop water requirement particularly in the use of Penman-Monteith procedures and in application of the crop empirical and biophysical models.

Furthermore Chukaliev *et al.* [81] presented research in the effects of evapotranspiration and water deficit on the yield of winter wheat planted in Pelagonia area. The agricultural production in the country is water limited, so authors tried to determine the yield reduction as a result of water limitation. They considered the winter wheat grown in Pelagonia region as usually non-irrigated crop, therefore very prone to reduced yield as a result of water limitation.



Figure 1. Potential evaporation (annual and for growing season) in period 1961–1990 and 1971–2000, rainfall and water deficit in the growing season for period 1971–2000 (Chukaliev [87])

The methodology applied was FAO Crop yield response to water deficit (Doorenbos and Kassam [82]), known as FAO Irrigation and Drainage Paper No. 33 or FAO 33. The 5-year period was analyzed and authors reported water deficiency of 19% in average that resulted in yield decrease by 28%, that was unexpected result and pointed to appearance of water deficit in the very sensitive stage for water deficit. This was the first paper published on the use of this methodology, almost 20 years

after it was introduced. Nevertheless, this methodology would be of great use in the future research conducted on effects of climate change on crop yield in the country. This methodology was long lasting and its update was presented in Steduto *et al.* [83].

Ančev *et al.* [84] presented their work on effect of the drought on the crop yield. They presented potential and real evapotranspiration calculated by Thornthwaite [20] methodology for several agricultural regions in the country and determined the

climatic water balance. The climatic water balance for all regions was negative; water deficit is therefore an important factor that limits crop yield in the country. Ančev *et al.* [85] provided extensive summary on the effect of drought on agricultural production and on the environment. They provided data for potential evapotranspiration for the period 1951–1990. Moreover, the paper compared the yield of the agricultural crops in years with different drought intensity and provided beneficial results for further elaboration of the crop yield response to water deficit.

Iljovski *et al.* [86] presented their research in the importance of accurate determination of irrigation water requirement in preparation of the Water Master Plan of the country. They presented their results on the use of Penman–Monteith FAO56 procedure and estimated that amount of water for irrigation of the potential 390 000 ha was about 2 billion m³.

More complex work on estimating evapotranspiration was done by Chukaliev [87] for preparation of the First National Communication of the Republic of Macedonia to UNFCCC [87]. The maps of the spatial distribution of the evapotranspiration were prepared for the two 30-year periods (1961-1990 and 1971-2000). The maps for both periods were compared and showed an increase of evapotranspiration in major agricultural regions in the country. The method of choice was Thornthwhaite [20], due to the low level of availability of the data required to use the FAO 56 Penman-Monteith procedure. Moreover, the maps of the efficient rainfall and water deficit were produced for the same period. With this work the geospatial distribution of the evapotranspiration in the country was provided for the first time.

Tanasković *et al.* [88] and [89] gave their contribution to the calibration of the methods for estimation of the evapotranspiration and crop water requirement for pepper crop, providing data for different crop growth stages and different irrigation techniques and irrigation practices.

Research in climate change vulnerability assessment and adaptation measures based on evapotranspiration and crop water requirement

The first paper on the effect of climate change on agricultural sector was published by Chukaliev *et al.* [90]. The authors discussed the effect of the increased temperature on evapotranspiration using FAO 56 procedure and provided data for two important agricultural regions (Bitola and Štip) on the average values of the referent evapotranspiration, for the period 1961–1998 and in two

climate change cases (increase of temperature for 1 °C and for 2 °C). In order to determine the irrigation water requirement they applied different procedures in estimating the efficient rain as recommended in the FAO Irrigation and drainage paper No 25 (Dastane [91]). The simulation was done by using FAO CROPWAT software. In this work authors concluded that using of this procedure for estimating the effect of climate change on irrigation water requirement was very risky because the choice of the method for estimation of the effective rainfall was making much bigger difference than the effect of increased temperature itself.

Later Chukaliev and his team started the work on vulnerability assessment and adaptation measures of the agricultural sector for the purpose of preparation of the First National Communication to the United Nation Framework convention on Climate Change (UNFCCC) (Group of authors, [92]). This work was the first attempt for larger scale addressing of climate change in agricultural sector. The extensive literature review was conducted and team faced with a serious challenge to conduct this research based on very limited data (average monthly temperatures and precipitation for the period 1961-90 for the major meteorological stations in the country). The previous experience with water limitation and the use of drought indices for assessment of drought served as a starting point and the most vulnerable regions were determined as regions with the highest increase of dryness as compared to the reference period. The most vulnerable zone was Povardarie region, especially the area of the conjunction of Crna and Bregalnica River with Vardar River. Very vulnerable zones were: the southeastern part of the country (Strumica), Southern Vardar valley (Gevgelija), Skopje-Kumanovo Valley and Ovche Pole. The most vulnerable crops were determined as the most important crops in the vulnerable regions as follow: vine grape in Povardarie region; tomato in South and South Eastern part of the country; winter wheat in Skopje -Kumanovo and Ovche Pole area: apple in big lakes region, particularly Resen and alfalfa as crop with very high water demand and huge importance in livestock sector in all agricultural regions in the country. Having in mind that even at present the most important limiting factor of crop production is water deficit, the authors applied FAO Crop Yield Response to Water Deficit Approach. The crop evapotranspiration in reference period was used as potential, and crop evapotranspiration in climate change case was used as actual evapotranspiration. The reference period crop yield was used as potential, and the yield with climate change was the actual one. This was done for all of the most vulnerable crops and regions using the FAO 56 methodology, but the evapotranspiration calculated by Thornthwhaite [20] was assumed as reference transpiration. The biggest limitation of this approach was that it was not possible to evaluate the effect of the proposed adaptation measures. This was a very rough approach, but due to the limited data availability it was impossible to conduct more precise research in that period.

The same team worked on the vulnerability and adaptation of agricultural sector report for the preparation of the Second national communication to UNFCCC (Group of authors, [87]). Again very limited data set was available, monthly values for temperature and precipitation for two periods 1961-1990 and 1971-2000. The team faced the same problems particularly with the evaluation of the yield response to climate change and the adaptation measures. The insufficient data, made almost impossible the application of some of the existing crop models. There was a substantial improvement in biophysical crop modelling in the period between two national communications and several models were extensively used in climate change studies as WOFOST (Diepen et al. [93]), CropSyst (Stöckle et al. [94]), DSSAT (Jones et al. [95]), STICS (Brisson et al. [96]), etc. Moreover, the problem was that there was no financing of national research that would enable the development of capacities for using biophysical crop models. On the other hand, the research team developed new capacities for using GIS and spatial numerical modelling. So the vulnerability assessment was conducted in GIS, and various thematic maps were developed for drought indices, evapotranspiration (estimated with Thorntwaite method), effective rainfall, climatic water deficit, etc. These maps were developed for the both investigated periods and provided an excellent overview of the vulnerable regions in the country. The most vulnerable region appeared to be the central part of the Vardar River catchment. The effect of the changed climate on the crop yield was again predicted using FAO Crop Yield Response to water deficit as previously explained. In this research there was much better addressing to the climate change with an evaluation of the periods of the years 2025, 2050 and 2100. But there was still a similar weakness as in the previous communication.

For the preparation of the third national communication to UNFCCC (Group of authors [97]) an extensive cooperation with the Joint Research Center of the European Commission from Ispra, Italy, was established. The most advanced methods were used as biophysical model applica-

tions - BioMA (BioMA [98]). The BioMA is a modelling platform that combines several models. In the preparation of the third national assessment of vulnerability and adaptation of the agricultural sector two models were used: ClimIndices model for assessment of the vulnerability to climate change and CropSyst model for assessment of the impact of the adaptation measures to climate change. Moreover, the data set used for historical weather, as well as for climate change weather, were sourced from JRC (MARS weather database for historical data and climate change weather data generated with a stochastic weather generator trained over RCM-GCM downscaled simulation from the ENSEMBLES project). The weather data used were grid weather with the grid size 25×25 km. The report addressed the South Eastern part of the country distributed in 7 grids. This report is the most extensive work on the climate change and its effect on agriculture, and probably the best source of evapotranspiration calculated by means of the biophysical model. A similar approach was used by Mukaetov et al. [99] for assessment of the climate change impact on viticulture in Povardarie region. The adaptation practices tested in this work showed a need for dislocation of the grape on higher elevation and increased crop water requirement.

The use of crop water requirement data for estimation of the economical losses caused by the climate change and of the costs of the adaptation measures in the country was conducted by Callaway [100]. The crop water requirement was determined for the reference case and for climate change case using FAO 56 Penman-Monteith procedure. The damages of the climate change were based on yield reduction. Further, two adaptation options were applied. The first adaptation case was supplemental irrigation (to irrigate rain-fed area with amount of water required to maintain base case yield). The second case was using full irrigation. The research was conducted for the irrigation system of Streževo and the authors used two time frames (years 2050 and 2100). The climate change scenario was developed by Bergant [101] and 3 levels of impact were used (low, medium and high). The researchers found that by 2050, the climate change damages in the rain-fed part of the region for the most severe climate change scenario roughly equalled the net income in the reference case. For the irrigated part of the agricultural sector in this region, the magnitude of the climate change induced damages reached the level of net income in the reference case by 2100 under the medium climate change scenario - and far surpassed the reference case net income levels under the high climate change scenario. The net adaptation benefits were positive in all cases that showed that the economic benefits of these measures would be greater than their costs.

Buzarovska [102] used the same data set as Callaway [100] in her research on the optimization of agricultural production under climate change for Pelagonia Region. Her research aimed to illustrate the variation in crop area in Pelagonia region in 2050 due to the increased irrigation requirements of crops. The allocation of the crop area depended on the net return per unit of crop area. On the other hand, the net return was strongly related to the irrigation water requirements. Both, rain-fed and irrigated crops were expected to have higher water demand driven by the higher temperatures and reduced soil moisture because of the decreased precipitation and runoffs. The author used three climate scenarios for 2050 (low, medium and high impact). The climate change scenarios were subject to comparison with the Base case scenario. The technique of linear optimization was used to identify the best cropping pattern under given constraints. The findings of the study showed that due to climate divergences in 2050, the crop structure differed in various climate scenarios. In general, the more severe climate in 2050 will cause decrease in net returns by 11% in the most optimistic scenario (2050 Low) and 22% in the pessimistic scenario (2050 High), if no adaptation measures are applied. The production of the low profitable crops (cereals, industrial and fodder crops) will be reduced to their minimal levels, while the production of high profitable crops such as vegetable, especially green pepper, tobacco and other crops that increase net return per crop area would be intensified.

Even though the following research was conducted on European scale, it is presented here because the national researchers participated in these activities, so we can consider it as a national achievement. The research of the Duvellier et al. [103] analyzed the change of inter-annual variability of agro-climatic indices calculated for the major environmental zones in Europe from a baseline climate in 2000 to a projected climate in 2030. It leveraged on a future daily weather dataset based on 2 contrasting realizations of scenario A1B by global circulation models (GCMs), dynamically downscaled with regional climate models (RCM) that have been bias-corrected. Agro-climatic indices were calculated using the ClimIndices software package. Although more than 100 indices were systematically calculated only 4 were presented (Growing Season Start, Growing Season Length, Last Air Frost Spring and Dry Spell). Both climate

projections showed an increase in the growing season length for all zones without any considerable increase in variability. This would be partly caused by an earlier start of the season. The changes in late frost dates were not apparent, warning that although the earlier sowing of crops could be beneficial to have a longer growth cycle, the crops might potentially be exposed more to frost damage. A clear pattern of longer and more variable (from year-toyear) periods of consecutive dry days was determined in the Mediterranean and Pannonian regions

Ceglar et al. [104] used the Bio-physical Model Applications framework (BioMA) to simulate the maize yield response to water availability in current and future climatic conditions. Two different realizations of the A1B scenario from dynamically downscaled global circulation models within the ENSEMBLES project, which capture the most contrasting situations with respect to changes in precipitation and temperature, have been selected for this purpose. The CropSyst crop model was used to simulate the water-limited and potential maize yield, as well as total crop water requirement and total water consumption. The water deficit productivity index was introduced for the purpose of the study, describing the gain in crop yield when water deficit was reduced. The results showed that the maize yield was expected to decrease in Southern Europe as well as in the regions around the Black Sea during the 2030s. The water could become more productive in Central and Western Europe and slightly less productive in the Southern Europe.

Ceglar et al. [105] analyzed the spatial distribution of water demand for irrigation as a prerequisite to devise an appropriate water management strategies, which could stabilize crop production. In order to assess the effect of irrigation on crop yield, the experiment was conducted on grain maize, well-known as a crop sensitive to water deficit and drought. The spatial distribution of water deficit and maize yield deficit across Europe was simulated with the WOFOST model and compared between current and expected climatic conditions in 2030s. In our study, the priority has been given to future projections of the A1B emission scenario given by two contrasting regional climate model runs (in terms of projected air temperature change) within the ENSEMBLES project. The effectiveness of three irrigation strategies was compared, which could potentially be applied to offset the adverse climate change impact on grain maize yield in Europe: full, deficit and supplemental irrigation. The results showed that similar yields could be achieved using deficit irrigation strategy, when compared to full irrigation, thereby saving at least 30% of irrigation water in the current and future climate conditions.

This review will end with the last published paper on the use of evapotranspiration and crop water requirement for assessment of the impact of climate change and determination of the best adaptation options. Dimov et al. [106] presented the research in the changes in productivity of the winter wheat and sunflower as a result of changing climate. The biomass yield of the winter wheat without adaptation will decrease by 23% in 2025 and by 27% in 2050. Very similar reduction is expected for grain yield. The grain yield of the sunflower will decrease by 30% in year 2025 and by 40% in year 2050 without adaptation. The adaptation strategy for winter wheat composed of later sowing (mid to end of November) and sprinkler irrigation will reduce the impact of climate change. The adaptation strategy of 4 irrigations of sunflower by sprinkler irrigation showed the best effect on the sunflower yield and reduced the negative impact of climate change.

CONCLUSION

The national capacities for measuring the evapotranspiration and crop water requirement are extremely limited. There are not installed lysimeters. In addition, there is no equipment for use of other advanced technologies for measurement of the evapotranspiration and crop water requirement. For more than 50 years the scientific community in the country has been using the same approach for determination of the ET: the field experiments for determination of the water balance with bucket approach. Even though this is an old-fashioned technology, the extensive research activities were conducted and there were significant data accumulated for use in calibration and validation of more advanced technologies.

The capacities for assessment of the evapotranspiration and crop water requirement using the procedures that are highly appreciated by international scientific community (FAO 56 and ASCE-PM) exists, but the national researchers do not have experience with the ASCE-PM procedure and appreciate FAO56 and the use of the FAO CROP-WAT software. The problem is that climate data in the country are hardly available and very expensive, so that a limited number of research activities is conducted. Due to the lack of climate and meteorological data a lot of efforts were made to calibrate simple temperature based procedures for estimation of the evapotranspiration and crop water requirement and to recommend these procedures for use in irrigation scheduling.

The crop yield response to deficit water is a well established methodology in the country because there are no capacities to use more complex biophysical models that operate not only with water deficit, but also take into consideration many other parameters affecting crop yield. Even though certain attempts on the use of CropSyst and WOFOST were made in the last several years, a trained researcher can not continue with this activities due to the shortage of data required (meteorological and phenological data are almost not available).

The capacity to assess the vulnerability to climate change is well developed. Unfortunately capacities for modelling the crop response to changing climate and to the adaptation practices need a serious reconsideration. In the last period national scientists gain some experience in using of WOFOST and CropSyst biophysical models, but it is quite difficult to provide sustainable use of these models due to the lack of data required to run these models. The use of the biophysical models should be increased, but also the use of the empirical models should be initiated, and probably these models with lower need for data not available in the country can be a solution for the operational activities. The research should be aimed towards filling of the gaps and using of Earth Observation and other advanced practices.

Having in mind the transition period, data transparency and the poor national investment in research, we can conclude that the national research develops better than expected, but still a lot of efforts should be put forth to keep pace with the international scientific community.

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ПРЕГЛЕД НА ИСТРАЖУВАЊАТА НА ПОТРЕБАТА ОД ВОДА ЗА ЗЕМЈОДЕЛСКИТЕ КУЛТУРИ И НИВНА ПРИМЕНА ВО РЕПУБЛИКА МАКЕДОНИЈА

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Трудот дава преглед на истражувачките активности врз евапотранспирацијата (ЕТ), потребата од вода за земјоделските култури (ПВЗК) и употребата на овие параметри во други истражувања, пред се истра/увањата на климатските промени. Во првиот дел на трудот се разгледуваат за достигнувањата во овие истражувања на светското ниво. Потоа се дискутира за постигнувањата на домашните научноистражувачки активности. Македонија нема развиено капацитети за мерење на ЕТ и ПВЗК. Се применува само една практика – полски опити за определување на водниот биланс под претпоставка дека нема движење на вода во зоната на ризосферата. Определувањето на ЕТ пред сè е базирано на примена на процедурата ФАО 56 (Пенман-Монтеит процедура). Поради недостиг на податоци потребни за помасовна примена на оваа методологија се применува методологијата на Торнтвајт, која има помалку интензивни барања за влезни податоци. ЕТ и ПВЗК во земјата се користат при изработка на проекти за наводнување , во научноистражувачките активности и при определување на ефект од недостигот на вода врз приносите на земјоделските култури, пред сè во услови на климатски промени. Во последно време се изведени активности за примена на некои биофизички модели како што се WOFOST и CropSyst.

Клучни зборови: евапотранспирација; потреба за вода на земјоделките култури; климатски промени; научни истражувања; Република Македонија

Erratum

At the request of Ramachandran Vembu, autor of the Original scientific paper "Topomorphism - A new approach to identify topologies", published in *Contributions, Sec. Nat. Math. Biotech. Sci., MASA*, Vol. **36**, No. 2, pp. 145–152 (2015), the following corrigendum is given:

	Page	Col	Line No.	Error	Corrections to be carried out	
1.	145	2	5 from top	denote the set $A - B^c$.	denote the set $A \cap B^c$.	
2.	145	2	8 from top	For any function $F: X \to Y$	For any function $f: X \to Y$	
3.	145	2	11 from top	These sets are denoted by	These sets are usually denoted by	
4.	146	1	6 in Def 3.1	collection A_{α} of	collection $\{A_{\alpha}\}$ of	
5.	146	1	5 in Thm3.3	then $F(A) \subseteq f(B)$ and	then $F(A) \subseteq F(B)$ and	
6.	146	2	10 from top	For $i = 1$, let	For $i = 1, 2$, let	
7.	146	2	9 from bottom	$B_1 \cup B_2 = Y - F(X)$	$B_1 \cup B_2 = Y = F(X)$	
8.	146	2	6 from bottom	$B_1 \cap B_2 = \emptyset - F(\emptyset)$	$B_1 \cap B_2 = \emptyset = F(\emptyset)$	
9.	147	2	3 from top	function from $f: X \to Y$ as	Function $f: X \to Y$ as	
10.	147	2	7 from top	$y \in F(X - \{a\})$	$y \notin F(X - \{a\})$	
11.	147	2	9 from top	$F(X) - \{a\}$	$F(X-\{a\})$	
12.	147	2	26 from top	$F(X) - F((X - \{a_1\}) \cup$	$F(X) = F((X - \{a_1\}) \cup$	
13.	147	2	13 from bottom	Then $W - f^{\rightarrow}(U)$	Then $W = f^{\rightarrow}(U)$	
14.	148	1	12 from bottom	from (X, \mathcal{T}) to (Y, \mathcal{T}^*) .	from (X, \mathcal{T}) to (Y^*, \mathcal{T}^*) .	
15.	148	2	7 from bottom	$C = [0,1] \cup B \text{ and}$	$C = [0,1] \cap B$ and	
				$D = [0,2] \cup B$	$D = [0,2] \cap B$	
16.	149	1	15 from top	spaces [0,1] and [0,2], with	spaces [0,1] and (0,2), with	
17.	149	1	11 from bottom	$F^{-1}(B) = f^{\rightarrow}(B)$ for every	$F^{-1}(B) = f^{\leftarrow}(B)$ for every	
18.	149	2	Thm 5.1, 6(iii)	open in (Y, τ') with	open in (Y, \mathcal{T}') with	
19.	150	1	1 from top	$F_c(A) \subseteq F_c(B)$	$F_c(A) \subseteq F_c(B)$	
20.	150	1	4 from top	closed in (X, τ)	closed in (X, \mathcal{T})	
21.	150	1	5 from top	Since $\subseteq B$,	Since $A \subseteq B$,	
22.	150	2	16 from top	$\bar{A} = F_c^{-1}(F_c(\bar{A}))$	$\bar{A} = F_c^{-1}(F_c(\bar{A}))$	
23.	150	2	10 from bottom	\ldots open set in (X, τ)	\ldots open set in (X, \mathcal{T})	
24.	151	1	15 from top	$G(V) = F(B_{\alpha})$	$G(V) = \cup F(B_{\alpha})$	
25.	151	1	18 from top	Since $B_{\alpha} = \bigcup_{\beta \in \Lambda'} B_{\beta}$,	Since $B_{\alpha} \subseteq \bigcup_{\beta \in \Lambda'} B_{\beta}$,	
26.	151	1	20 from top	$F(B_{\alpha}) = \cup_{\beta \in \Lambda'} F(B_{\beta})$	$F(B_{\alpha}) \subseteq \cup_{\beta \in \Lambda'} F(B_{\beta})$	
27.	151	2	7 from top	Let $W \in \mathcal{T}$.	Let $W \in \mathcal{T}'$.	
28.	151	2	17 from top	$=\cup_{\beta\in\Lambda'}B_\beta.$	$= \cup_{\beta \in \Lambda'} F(B_{\beta}) .$	

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- [2] G. Jovanovski, P. Makreski, B. Šoptrajanov, B. Kaitner, B. Boev, Minerals from Macedonia, *Contributions, Sec. Math. Tech. Sci.*, MANU, XXVI, 1 (2005), pp. 7–84.
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Books:

- [1] J. A. Roels, *Energetics and Kinetics in Biotechnology*, Elsevier Biomedical Press, Amsterdam, New York, Oxford, 1983.
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Scientific meetings:

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