

СОДЕРЖАНИЕ ГЛИФОСАТА В ЗЕРНЕ ПРИ ДЕСИКАЦИИ ПОСЕВОВ В ПРИОБЬЕ

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Определены остаточные количества глифосата в зерне пшеницы после десикации посевов. Глифосат – наиболее применяемый в мире гербицид. Уровень его экотоксичности широко обсуждают в научной литературе после отнесения соединения к категории «вероятно канцерогенных» пестицидов. Предполагают, что остаточное количество гербицидов может быть наиболее высоким при десикации посевов перед уборкой. Исследования проведены в 2018 г. (центральная лесостепь Новосибирского Приобья – 54°53'13,5"N, 82°59'36,7"E). В эксперименте десикацию посевов осуществили гербицидом Зеро Супер (содержание изопропиламинной соли глифосата 750 г/кг) в рекомендованной дозе, равной 1,5–2,0 кг/га. Остаточное количество гербицидов в растительной биомассе определяли при помощи тест-системы, основанной на принципе иммуноферментного анализа. Предподготовку растительных проб осуществляли согласно рекомендации фирмы Stylab, проводившей валидацию метода для определения глифосата в зерне. Остаточное количество гербицида обнаружено во всех 37 испытанных образцах. Независимо от срока отбора образцов после десикации показатель в зерне не превышал 4,4 мг/кг. Спустя 14 дней после десикации уровень минимального количества пестицида был ниже (0,5 мг/кг) в сравнении с данными, полученными спустя 7 дней (2,6 мг/кг). Содержание остаточного количества гербицидов в соломе пшеницы оказалось выше, чем в зерне. Наиболее высокие значения показателя обнаружены в биомассе сорной растительности, высохшей при десикации (до 9 мг/кг). В зерне, хранившемся один год, содержание остаточного количества гербицидов не снизилось. Полученные данные сравнили с уровнями ПДК остаточного количества гербицидов в зерне пшеницы, принятыми в мире.

Ключевые слова: глифосат, остаточные количества, зерно пшеницы, Приобье

GLYPHOSATE RESIDUES IN GRAIN AFTER DESICCATION OF CROPS IN THE OB REGION

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The residual amounts of glyphosate in wheat grain after desiccation of crops were determined. Glyphosate is the most widely used herbicide in the world. The level of its ecotoxicity is widely discussed in the scientific literature after the compound was classified as “a likely carcinogenic” pesticide. It is assumed that glyphosate residues can be highest when the crops are desiccated before harvesting. The studies were carried out in 2018 (central forest-steppe of the Novosibirsk Ob region (54°53'13.5"N, 82°59'36.7"E). In the experiment, desiccation of wheat was carried out with the herbicide Zero Super (the content of isopropylamine salt of glyphosate was 750 g/kg) at the recommended dose of 1.5– 2.0 kg/ha. Glyphosate residues in plant biomass were determined using the test system, based on the principle of enzyme immunoassay. Pre-preparation of the samples for the analysis was carried out according to the recommendation of Stylab laboratory, which validated the method for the determination of glyphosate in grain. Glyphosate residues were found in all of the 37 samples tested. Regardless of the sampling period after desiccation, herbicide residues in the grain did not exceed 4.4 mg/kg. In 14 days after desiccation, the minimum pesticide level was lower (0.5 mg/kg) compared to the data obtained in 7 days (2.6 mg/kg). The content of the herbicide residues in wheat straw was higher than in grain. The highest values of glyphosate residues were

found in the dry biomass of weed vegetation (up to 9 mg/kg). In the grain stored for 1 year, the content of GR has not decreased. The data obtained were compared with the MRL for the residual amount of glyphosate in wheat grain accepted in the world.

Keywords: glyphosate, residual amount, wheat grain, the Ob region

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Conflict of interest

The authors declare no conflict of interest.

INTRODUCTION

Obtaining stable output yields to provide the growing population of the planet with a sufficient amount of food and raw materials in the face of dwindling land resources is impossible without the use of plant protection chemicals, which is fraught with environmental pollution with residual amounts of pesticides [1]. To solve this problem, the world community is taking comprehensive measures: it searches for new formulas of chemicals with a low dose of use, improves approaches and methods for controlling the residual amount of pesticides in environmental objects, develops new technologies for cleaning contaminated areas, etc. [2–4].

The agrarian sector of the Siberian Federal District is in line with the global trend towards the built-up of crop protection chemicals. At the same time, the assessment of the safety of the pesticides used is based on the recommendations of the manufacturers, developed for areas with more favorable climatic conditions in comparison with Siberia. In this regard, it becomes necessary to study the features of the degradation of pesticides in crop products and environmental objects in the specific conditions of a short, often dry growing season in Siberia. The Novosibirsk Region is one of the largest grain producers in the Siberian Federal District. A significant part of the production on farms is obtained with the help of intensive technologies for growing crops, and this tendency in the de-

velopment of agriculture will only intensify. In addition, in recent years, agricultural technologies have been increasingly actively mastered, implying a partial or complete abandonment of mechanical soil cultivation (direct sowing system, no-till, etc.), which requires an increased use of pesticides. One of the most widely used pesticides in such agricultural technologies are continuous herbicides based on glyphosate. In the Russian Federation, herbicides based on this compound account for 1/3 of the volume of applied herbicides and are represented by more than 40 names [5].

Glyphosate is the active ingredient in a spectrum of non-selective systemic herbicides, first patented as commercial products such as Roundup® (Monsanto Co) or Touchdown® (Syngenta Co) in 1974. Since then, these herbicides have become one of the most widely used in the world and their consumption rates are increasing [6, 7]. An increase in the sowing of transgenic crops resistant to this group of herbicides and an increase in the resistance of weeds contribute to the expansion of the market for herbicides based on glyphosate [8, 9].

The high ecotoxicity of most of the pesticides has been experimentally proven. Glyphosate (N- (phosphonomethyl) glycine) is probably no exception. In 2015, the WHO Agency for Research on Cancer (IARC) classified glyphosate as a “possibly carcinogenic” pesti-

cide¹. The IARC announcement attracted public and scientific attention around the world. The European Union has recommended that the use of glyphosate as a desiccant be restricted and its use in public gardens and parks must be stopped². Public movements have formed for a complete ban on the use of the drug in the agricultural sector. Since the publication of the document (see footnote 1), a lot of research has been done on various aspects of the problem. A great deal of very contradictory data has been obtained. In general, the authors believe that the number, duration and the design of studies are insufficient for final conclusions so far [8, 9]. To date, the use of glyphosate in the EU is allowed only until 2022, a number of countries (Belgium, Colombia, the Netherlands, Sri Lanka, France, many countries of the Middle East) have legally restricted the use of glyphosate in agriculture [10, 11].

However, the appearance of glyphosate at one time made it possible to move to a new stage in the development of soil-protective and resource-saving agriculture in the world. At present, it is difficult to imagine the agricultural sector without this drug. In recent years, possible parameters for food production have been studied if the use of glyphosate is prohibited. Calculations have shown that in this case, the gross harvest of soybeans, corn, rapeseed in the world may decrease by 18.6; 3.1; 1.5 million tons per year, respectively. To compensate for these losses, an increase in the arable land area by 0.76 million hectares will be required. At the same time, the cost of weed control will increase by \$ 20-30. US / ha. The use of alternative herbicides will not lead to a decrease in the pesticide load on agrocenoses [12, 13].

As our brief review shows, despite the ecotoxicity, agriculture is unlikely to be able to stop using glyphosate. There is only one way out of the situation - strict control of the resid-

ual amount of the pesticide in the environment, primarily in products, in order to minimize its entry into the food chain.

The aim of the study is to determine the value of the residual amount of glyphosate in grain and wheat straw after desiccation of crops in the recommended doses and timing of harvesting in the Ob region, to clarify the degree of its decomposition during grain storage throughout the year.

MATERIALS AND METHODS

The studies were carried out in 2018 on the territory of the scientific and experimental base of the Siberian Research Institute of Agriculture and Chemicalization of the Siberian Federal Scientific Center of the Russian Academy of Sciences (central forest-steppe of the Novosibirsk Ob region, 54° 53'13.5 "N, 82° 59'36.7" E). Desiccation of spring wheat crops was carried out on September 3 with the herbicide Zero Super (content of isopropylamine salt of glyphosate 750 g / kg) at the recommended dose of 1.5–2 kg / ha³. The wheat was Novosibirskaya 31. The moisture content of grain before desiccation was 25–28%. The sprayer OP-2000. The consumption rate of the working solution is 270 l / ha. Harvesting took place 21 and 25 days (in 2017 and 2018, respectively) after desiccation. The sampling was carried out by the sheaf method (1 m²) in triplicate on the plot and three repetitions of the experiment (9 sheaves). In the period after desiccation, the air temperature was close to the average multiyear norm. Precipitation in 2017 (harvesting 09.09.2017) 20 mm with an average annual rate of 33 mm, in 2018 (harvesting 09.13.2018) - 60 mm with a rate of 40 mm.

Grain, straw and biomass of weeds dried up under the influence of the preparation were analyzed. To determine the possibility of glyphosate decomposition during storage of

¹IARC Monograph on Glyphosate (https://www.iarc.fr/en/.../iarcnews/.../glyphosate_IARC2016).

²Directive 2009/128/EC, 2009b (Article 4) of the European Parliament and of the Council of 21 October 2009. Regulation (EC) No 1107/2009 of the European Parliament and of the council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC <https://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ: L: 2009:309:0001:0050: EN:PDF>.

³State catalog of pesticides and agrochemicals approved for use on the territory of the Russian Federation.

grain products, grain of the 2017 harvest was analyzed, which was stored for a year in a cool and dry room. The residual amount of glyphosate was determined using the Abraxis Glyphosate Plate ELISA test system. The preparation of plant samples was carried out according to the recommendation (procedure R091313) of the Stylab company (Moscow), which validated the method for the determination of glyphosate in grain (www.Stylab.ru). Samples of the samples were derivatized, then added together with antibodies specific to glyphosate into the wells of a microtiter plate, coated with anti-species antibodies to antibodies to glyphosate, and incubated for 30 min. The enzyme conjugate glyphosate was then added. At this point, a competitive reaction occurs between glyphosate, which may be contained in the sample, and the labeled glyphosate enzyme for binding sites with antibodies on the plate. After washing and adding the substrate (color solution), a colored signal (blue color) develops. The presence of glyphosate is detected by the addition of a colored solution that contains an enzyme substrate (hydrogen peroxide) and a chromogen (3,3', 5,5'-tetramethylbenzidine). The antibody-bound glyphosate-enzyme conjugate catalyzes the conversion of the substrate / chromogen mixture into blue-colored reaction products. After incubation, the reaction was stopped and stabilized by adding dilute acid (stop solution). Since there was competition between labeled glyphosate (conjugate) and unlabeled (sample) for binding to antibodies, the

color intensity is inversely proportional to the concentration of glyphosate in the sample.

RESULTS AND DISCUSSION

Residual amounts of glyphosate were found in all 37 samples tested. Regardless of the time of sampling after desiccation in grain, it did not exceed 4.4 mg / kg (see Table 1). At 14 days after desiccation, the minimum amount level was lower (0.5 mg / kg) compared to the data obtained after 7 days (2.6 mg / kg). The residual amount of glyphosate in wheat straw was found to be higher than in grain. The highest values of the indicator were found in the biomass of weeds dried up during desiccation.

In grain stored for a year in a cool dry room, the residual amount of glyphosate was slightly higher compared to the data obtained in the analysis of grain from the 2018 harvest.

For the first time for Siberia, data were obtained on the content of the residual amount of herbicides in wheat grain after desiccation with the recommended doses of herbicide.

It should be noted that the MRL (maximum residual level) or MRL for the residual amount of glyphosate in unprocessed wheat grain, varies significantly across the world. In the USA this value is taken equal to 30 mg / kg, in the EU - 10, in Canada - 5 mg / kg⁴. This is probably due to the different background value of the content of the residual amount of pesticide in the product. As noted by the authors of the review [9], these limits are constantly growing,

Табл. 1. Содержание остаточного количества глифосата в надземной биомассе зернового агроценоза после десикации посевов

Table 1. Concentration of glyphosate residues in the aboveground biomass of grain agrocenosis after desiccation of crops

Sample selection year	n	The day after desiccation	Herbicide dose, kg / ha	Substrate	Glyphosate content, mg / kg	
					lim	average
2018	7	7	1,5	Grain	2,6–4,4	3,6
	12	14	1,5	»	0,5–3,9	2,5
	6	14	1,5	Straw	5,5–6,0	5,7
	6	14	1,5	Weeds	8–9	8,5
2017	6	After a year of storage	2	Grain	4,1–5,4	4,2

⁴BCGlobal Pesticide MRL Database <https://www.bryantchristie.com/BCGlobal-Subscriptions/Pesticide-MRLs>

apparently following an increase in the content of contaminants in products. In the Russian Federation, the MRL for herbicide residues is 20 mg / kg of grain⁵. It is difficult to understand where this figure came from. This is probably the calculated average MRL value adopted in the USA and the EU, since we did not find published information on the content of the residual amount of glyphosate in grain for the Russian Federation in the available literature. According to the above criteria, the content of the residual amount of herbicides in the grain in our experience is within acceptable limits. Based on the decrease in the minimum detectable amount of glyphosate residues in the grain, the waiting time of 14 days after desiccation is significant.

As noted by the authors of a review published in 2019 [11], there is very little documented information in the scientific literature on herbicide residues in cereal products, particularly wheat grain, after desiccation. The authors found only four sources published between 2013 and 2018 (see Table 2). From these data, it appears that herbicide residue levels in grain after desiccation exceeded the MRL adopted in Canada in one case out of four (the study was conducted in Canada). For the territory of the former USSR, data for Ukraine are available [14]. In Kiev region in 2002-2011, residual amounts of herbicides in grains of three wheat varieties during desiccation of crops with 3 l / ha of isopropyl salt of glyphosate (480 g/l r.a.) were determined. Depending on the wheat variety the indicator in the grain was 0.5-2.5 mg/kg. In Table 2 we gave data on changes in the content of herbicide residue in processed grain products. The bulk of it remains in bran. In a special experiment by Canadian researchers [15], the proportion of herbicide residue in bran was 81% of the original content in the grain. Nutritionists usually suggest bran as a component of a healthy diet. It follows that this side of the issue should be kept in mind.

On the whole, the impression is created that the situation with the studied indicator in

grain after desiccation is quite favorable. However, the question arises why the FAO / WHO MRL for glyphosate residues in wheat grain is 30 mg / kg.

If there is a lot of data on the dynamics of herbicide residues during grain processing, then there is very little information on the change in the pesticide content during grain storage [11]. We have received information on this issue. Our conclusion is based on the assumption that strict adherence to the same experimental conditions in both years of research resulted in an approximately similar level of herbicide residues in the grain before harvesting. When storing grain during the year, this indicator did not decrease. This is probably due to the well-known fact that glyphosate is well fixed in plant biomass, as a result of which the rate of its metabolism decreases [16]. Information on the herbicide residues in straw and weeds is important, since, as follows from our data, these components of the biomass can serve as a significant concentrator of pesticides in the agrocenosis.

CONCLUSIONS

1. Residual amounts of glyphosate (up to 4.4 mg / kg) were found in all samples of wheat from crops subjected to desiccation.
2. When storing contaminated grain for a year in a dry and cool room, there was no decrease in the residual amount of glyphosate in the grain.

Табл. 2. Содержание глифосата в зерне пшеницы до и после переработки [11]

Table 2. Concentration of glyphosate in wheat grain before and after processing [11]

Glyphosate content in the source grain, mg / kg	Grain processing products	
	Product type	Glyphosate content, mg / kg
Less than 0,13	Bran	0,7
0,67	Flour	0,02
1,07–1,13	Bread	0,001–0,0458
6,1–11,1	Baked goods	0,001–0,0179

⁵Hygienic standards for pesticide content in environmental objects (list) GN 1.2.3538-18 // Bulletin of regulatory and methodological documents of RSES. 2019. Issue 3 (77). Pp. 7-103.

3. A significantly higher content of the residual amount of glyphosate in straw and weed biomass (up to 9 mg / kg) was found in comparison with grain. These plant residues can be a significant source of pesticide pollution.

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