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SYNTHESIS OF N-(5-ETHYL-1,3,4-THIADIAZOL-2-YL)BENZAMIDE AND EVALUATION OF THE ANTI-TERMITE ACTIVITY OF 2-AMINO-5-ALKYL-1,3,4-THIADIAZOLE DERIVATIVES

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Abstract. This study investigated the synthesis of N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide and evaluated the anti-termite activity of several 2-amino-5-ethyl-1,3,4-thiadiazole derivatives against worker termites. The biological activity of the compounds was assessed using filter paper treated with 0.02% aqueous solutions of the test substances. Each treatment was tested on 100 worker termites under laboratory conditions, and mortality was recorded from 3 to 14 days. Among the tested compounds, 2-amino-5-ethyl-1,3,4-thiadiazole showed the fastest effect, causing 100% mortality by day 10**,** whereas the other derivatives demonstrated a more prolonged toxic action. The synthesis of **N**-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide was optimized in different solvent media. The highest yield (94.4%**)** was obtained in acetone, while lower yields were recorded in tetrahydrofuran, chloroform, and acetonitrile. The results indicate that thiadiazole derivatives are promising candidates for developing effective and environmentally safer termiticidal agents.

Keywords: 1,3,4-thiadiazole derivatives; termiticidal activity; worker termites; N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide; solvent optimization.

Аннотация. В данном исследовании изучался синтез N-(5-этил-1,3,4-тиадиазол-2-ил)бензамида и оценивалась противотермитная активность нескольких производных 2-амино-5-этил-1,3,4-тиадиазола против рабочих термитов. Биологическая активность соединений оценивалась с помощью фильтровальной бумаги, обработанной 0,02% водными растворами исследуемых веществ. Каждая обработка тестировалась на 100 рабочих термитах в лабораторных условиях, а смертность регистрировалась в течение 3–14 дней. Среди протестированных соединений 2-амино-5-этил-1,3,4-тиадиазол показал наиболее быстрый эффект, вызывая 100% смертность к 10-му дню, в то время как другие производные продемонстрировали более

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длительное токсическое действие. Синтез N-(5-этил-1,3,4-тиадиазол-2-ил)бензамида был оптимизирован в различных растворителях. Наибольший выход (94,4%) был получен в ацетоне, тогда как более низкие выходы были зафиксированы в тетрагидрофуране, хлороформе и ацетонитриле. Результаты показывают, что производные тиадиазола являются перспективными кандидатами для разработки эффективных и экологически безопасных средств против термитов.

Ключевые слова: производные 1,3,4-тиадиазола; термитоцидная активность; рабочие термиты; N-(5-этил-1,3,4-тиадиазол-2-ил)бензамид; оптимизация растворителя.

Annotatsiya. Ushbu tadqiqotda N-(5-etil-1,3,4-tiadiazol-2-il)benzamid sintezi o'rganildi va bir nechta 2-amino-5-etil-1,3,4-tiadiazol hosilalarining ishchi termitlarga qarshi antitermit faolligi baholandi. Birikmalarning biologik faolligi sinov moddalarining 0,02% suvli eritmalari bilan ishlov berilgan filtr qog'ozi yordamida baholandi. Har bir ishlov berish laboratoriya sharoitida 100 ta ishchi termitda sinovdan o'tkazildi va o'lim 3-14 kun ichida qayd etildi. Sinovdan o'tgan birikmalar orasida 2-amino-5-etil-1,3,4-tiadiazol eng tez ta'sir ko'rsatdi, 10-kunga kelib 100% o'limga olib keldi, boshqa hosilalar esa uzoqroq toksik ta'sir ko'rsatdi. N-(5-etil-1,3,4-tiadiazol-2-il)benzamid sintezi turli erituvchilarda optimallashtirildi. Eng yuqori hosil (94,4%) asetonda olingan, tetragidrofuran, xloroform va asetonitrilda esa pastroq hosil qayd etilgan. Natijalar shuni ko'rsatadiki, tiadiazol hosilalari samarali va ekologik toza termitlarga qarshi vositalarni ishlab chiqish uchun istiqbolli nomzodlardir. Ushbu tadqiqotda 2-amino-5-etil-1,3,4-tiadiazol hosilalarining sintezi o'rganildi va ularning ishchi termitlarga qarshi antitermit faolligi baholandi. Birikmalarning biologik faolligi sinov moddalarining 0,02% suvli eritmalari bilan ishlov berilgan filtr qog'ozi yordamida baholandi. Har bir ishlov berish laboratoriya sharoitida 100 ta ishchi termitda sinovdan o'tkazildi va o'lim 3-14 kun ichida qayd etildi. Sinovdan o'tgan birikmalar orasida 2-amino-5-etil-1,3,4-tiadiazol eng tez ta'sir ko'rsatdi, 10-kunga kelib 100% o'limga olib keldi, boshqa hosilalar esa uzoqroq toksik ta'sir ko'rsatdi. N-(5-etil-1,3,4-tiadiazol-2-il)benzamid sintezi turli erituvchilarda optimallashtirildi. Eng yuqori natija atseton (94,4%) muhitida kuzatildi. Tetragidrofuran, xloroform va asetonitrilda esa pastroq unumni qayd etilgan. Natijalar shuni ko'rsatadiki, tiadiazol hosilalari samarali va ekologik toza termitlarga qarshi vositalarni ishlab chiqish uchun istiqbolli nomzodlardir.

Kalit so'zlar: 1,3,4-tiadiazol hosilalari; termitsid faolligi; ishchi termitlar; N-(5-etil-1,3,4-tiadiazol-2-il)benzamid; erituvchini optimallashtirish.

INTRODUCTION.

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Termites are among the most destructive cellulose-feeding insects in natural, agricultural, and urban environments, where they damage wood, plant-derived materials, and other cellulose-containing substrates [1, 10-15,16]. Modern termite management increasingly emphasizes bait-based strategies, because slow-acting toxicants may be transferred among workers through social interactions and feeding[8], thereby contributing to colony-level suppression rather than only rapid mortality of exposed individuals. Recent termite-control literature continues to describe baits as a major targeted control approach[5,6,8]. In the search for new biologically active agents, heterocyclic compounds occupy a prominent place because relatively small structural modifications can markedly alter their physicochemical and biological properties. Among them, 1,3,4-thiadiazole derivatives have attracted considerable attention due to their broad spectrum of biological activities [3], including pesticidal and insecticidal potential [2,4,7,9]. Reviews of this scaffold describe it as a versatile platform for the design of biologically active compounds and note its relevance in pesticide development. In the present study, attention was focused on 2-amino-5-ethyl-1,3,4-thiadiazole derivatives as potential anti-termite agents. According to the our experiments, several compounds were evaluated against worker termites using filter paper treated with 0.02% aqueous solutions, with mortality recorded over 3–14 days and corrected using Abbott's formula. The same study also examined the synthesis of N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide in different solvent media in order to determine the most effective reaction conditions. Our data show that 2-amino-5-ethyl-1,3,4-thiadiazole produced the fastest mortality response, whereas the benzamide, toluenesulfonamide, and monochloroacetamide derivatives showed a more prolonged effect; in the synthesis section, acetone gave the highest product yield. Therefore, the aim of this work was to synthesize N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide under different solvent conditions and to evaluate the anti-termite activity of selected 2-amino-5-ethyl-1,3,4-thiadiazole derivatives under laboratory conditions. The obtained results may contribute to the development of new thiadiazole-based compounds as promising candidates for environmentally safer termite management. 1,3,4-Thiadiazole derivatives are considered promising biologically active compounds due to their wide range of pesticidal and insecticidal properties. The search for new termiticidal agents based on heterocyclic compounds remains an important direction in the development of environmentally safer termite control strategies. In this context, 2-amino-5-ethyl-1,3,4-thiadiazole derivatives are of particular interest because structural modification of the amino group may influence both their biological activity and physicochemical properties. The present study was aimed at two main objectives:

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first, to investigate the synthesis of N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide under different solvent conditions; and second, to evaluate the anti-termite activity of several 2-amino-5-ethyl-1,3,4-thiadiazole derivatives against worker termites.

MATERIALS AND METHODS.

Evaluation of anti-termite activity

The anti-termite activity of 2-amino-5-ethyl-1,3,4-thiadiazole derivatives was evaluated by determining the amount of filter paper consumed by termites when the compounds were offered as food. Filter paper pieces (2 × 2 cm) were soaked either in solutions of the test compounds or in distilled water in the control treatment. The treated filter papers were then placed into sterilized Petri dishes containing 100 worker termites. During the experiments, the Petri dishes were kept in a dark place. At scheduled observation times, the dishes were removed for recording the condition of the termites, the amount of filter paper consumed, and the numbers of live, paralyzed, and dead individuals. Throughout the experiment, the termites were supplied with distilled water. The tests lasted from 3 to 14 days. Both experimental and control treatments were performed in five replicates. Mortality percentages were calculated using Abbott's formula, taking into account mortality in the control group.

Test compounds and concentration

To study the termiticidal properties of the synthesized compounds, 100 termite individuals were used in each experiment. The compounds were dissolved in water to prepare 0.02% solutions, and their biological efficacy was then assessed under the same experimental conditions.

Synthesis of N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide

Within the scope of the study, the acylation of the amino group of 2-amino-5-ethyl-1,3,4-thiadiazole was investigated in different solvent media. Benzoyl chloride was used as the acylating agent. The reaction was carried out in tetrahydrofuran, chloroform, acetonitrile, and acetone, and the effect of the reaction medium on product yield was comparatively analyzed.

RESULTS AND DISCUSSION.

Anti-termite activity of 2-amino-5-ethyl-1,3,4-thiadiazole derivatives

The results on the effect of 2-amino-5-ethyl-1,3,4-thiadiazole derivatives on the viability of worker termites are presented in Table 1. The compounds showed different levels of biological activity at a concentration of 0.02%. A 0.02% solution of compound I (2-amino-5-ethyl-1,3,4-thiadiazole) caused termite mortality of 59.9% on day 3, 90.3% on day 7, and 100.0% on day 10. A 0.02% solution of compound II, N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide, caused termite mortality of 54.8% on day 3, 75.0% on day 7, 88.1% on day 10, and 97.9% on day

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14. A 0.02% solution of compound III, N-(5-ethyl-[1,3,4]thiadiazol-2-yl)-toluenesulfonamide, caused termite mortality of 49.2% on day 3, 70.4% on day 7, 82.5% on day 10, and 93.2% on day 14. A 0.02% solution of compound IV, N-(5-ethyl-1,3,4-thiadiazol-2-yl)monochloroacetamide, caused termite mortality of 35.0% on day 3, 73.0% on day 7, 85.9% on day 10, and 89.1% on day 14. Overall, the results show that all tested compounds exhibited anti-termite activity, although the rate and duration of their effects differed. Compound I acted faster and reached complete mortality by day 10, while compounds II, III, and IV showed prolonged effects over 14 days. According to your draft, this longer action may be important because a slower-acting bait has a greater chance of being distributed within the termite colony and eventually reaching the queen, thereby contributing to colony suppression.

Synthesis of N-(5-ethyl-1,3,4-thiadiazol-2-yl) benzamide in different solvents The solvent had a substantial effect on reaction efficiency.

In tetrahydrofuran, the reaction of 2-amino-5-ethyl-1,3,4-thiadiazole with equimolar benzoyl chloride in the presence of pyridine was first performed at room temperature for 1 hour and then at 55–65 °C for 2.5–3 hours. The product yield was 60%, indicating relatively low efficiency. In chloroform, the reaction was initially carried out at 0 °C for 1 hour and then at 55–60 °C for 2–3 hours. Under these conditions, the yield increased to 78.2%, although side reactions may still have occurred. In acetonitrile, reducing the reaction temperature to 75–80 °C and shortening the reaction time to 2–3 hours gave a yield of 86%, suggesting moderate efficiency but no clear specific advantage for this system. In acetone, the highest yield was obtained. The reaction started at 50–56 °C and was completed in a water bath over 2–3 hours. Under these conditions, the product yield reached 94.4%, making acetone the most effective medium among those tested.

Table-1.

Anti-termite activity of 2-amino-5-ethyl-1,3,4-thiadiazole derivatives at 0.02% concentration against worker termites

Compound	Chemical name	Mortality on day 3 (%)	Mortality on day 7 (%)	Mortality on day 10 (%)	Mortality on day 14 (%)
I	2-amino-5-ethyl-1,3,4-thiadiazole	59.9	90.3	100.0	—
II	N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide	54.8	75.0	88.1	97.9

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III	N-(5-ethyl-[1,3,4]thiadiazol-2-yl)-toluenesulfonamide	49.2	70.4	82.5	93.2
IV	N-(5-ethyl-1,3,4-thiadiazol-2-yl)monochloroacetamide	35.0	73.0	85.9	89.1

Note. Each treatment was tested on 100 worker termites using 0.02% aqueous solutions; mortality was recorded over 3–14 days.

Table 2.

Effect of solvent on the synthesis yield of N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide

Solvent	Reaction conditions	Yield (%)
Tetrahydrofuran	Room temperature, 1 h; then 55–65 °C for 2.5–3 h	60.0
Chloroform	0 °C, 1 h; then 55–60 °C for 2–3 h	78.2
Acetonitrile	75–80 °C for 2–3 h	86.0
Acetone	50–56 °C, water bath, 2–3 h	94.4

Identification and purity of the synthesized product

Thin-layer chromatography analysis of the products obtained by all methods showed the same retention factor, Rf = 0.51 in a benzene: acetone (2:1) solvent system, indicating that the products were chemically identical. Recrystallization from alcohol provided a highly pure material. In addition, IR spectroscopy revealed characteristic absorption bands of the amide group, confirming the expected monomeric structure of the synthesized compound.

CONCLUSION

The results demonstrated that 2-amino-5-ethyl-1,3,4-thiadiazole derivatives possess significant anti-termite activity against worker termites. At a concentration of 0.02%, all tested compounds caused substantial termite mortality, although their activity profiles differed in speed and persistence. Compound I produced the fastest mortality response, while compounds II, III, and IV showed longer-lasting effects, which may be advantageous for bait-based colony suppression. The solvent optimization study for the synthesis of N-(5-ethyl-1,3,4-thiadiazol-2-yl)benzamide showed that reaction yield depended strongly on the solvent medium. Among the solvents tested, acetone provided the best result, with a yield of 94.4%, and was therefore considered the most economically and technologically effective medium

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for synthesis. Further studies on structure–activity relationships are needed to clarify the mechanism of action of these compounds and to support the development of new, environmentally friendly termiticides based on thiadiazole derivatives.

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