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A novel approach to green silica nanoparticle synthesis using *Padina tetrastomatica* seaweed extracts against the pest *Spodoptera litura* fabricius

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Abstract
Until now, control strategies for insect pest species have relied on applications of synthetic insecticides. In addition, the effectiveness of these treatments could be reduced due to the resistance of insect populations to insecticides. The irrational use of chemical control strategies also has negative consequences for non-target organisms and threatens human health. A promising alternative to traditional insecticide formulations is the development of nanomaterials for insect control. Especially, silicon (Si) has been shown to be used to control a range of insect pests across taxa in various crops. It has been known to induce the tolerance of anti-xenotic and antibiotic species to insect parasites in various crops. In recent years, silica (SiNPs) has been used as a source of silica for both soil and foliar application to control insect pests. SiNPs are also used as a vehicle for the targeted application of insecticides due to their controlled release efficiency. In this work, we summarized the state of knowledge on synthesized silica nanoparticles (SiNPs) from *Padina Tetrastomatica* Seaweed as used in pest control. In addition, the aspects of their synthesis, the mechanism of action, the use in insect pest control, the potential for use as an insecticide vector, and the environment are described in detail.

Keywords: silica nanoparticles, *Spodoptera litura*, insect pest management, insecticides, green synthesis

Introduction
A macroscopic alga known as kelp is one of the oceans' most vital resources. They stick to the bottom in relatively shallow coastal waters. Algae are a rich source of a large number of various biologically active substances. (Chojnacka *et al*., 2012; Munirasu *et al*., 2013) [4, 16] Chemical pesticides play a significant role in increasing agricultural production as well as protecting crops from damage caused by insect pests. It is estimated that only 0.1% of agrochemicals used for plant protection reach their target pests, while the remaining 99.9% end up in the environment. (Mancini *et al*., 2005) [12] Plant pesticides (Isman, 1994; Mansour *et al*., 2011) [7, 14] are environmentally friendly and safe alternative methods of plant protection. Evaluation of plant extracts for their harmful effects on insects is one of the approaches used to find new plant insecticides (Isman, 1995) [8].

Seaweeds are renewable living resources that are a rich source of active and structurally novel secondary metabolites. It has been proven that algae have an insecticidal effect. (Cetin *et al*., 2010; Sahayaraj and Mary Jeeva, 2012) [3, 22] In addition, algae extract offers a new approach to pest management (Manilal *et al*., 2009; Rajesh *et al*., 2011;) [13, 19]. It is also used as animal feed, fertilizer, and herbicide (Manilal *et al*., 2009) [13].

Spodoptera littoralis is considered one of the most important and destructive pests not only for groundnut, cotton, but also for other vegetables, and field crops in tropical countries. We previously investigated the effects of commercial Silica on second-instar larvae of Spodoptera litura However, the main factors governing the insecticidal efficiency of Si nanostructures have not yet been determined (Osman *et al*., 2015) [17]. The groundnut worm Spodoptera litura can become a major pest of these crops and a threat to other host crops. (Pascal *et al*., 1999) [18].

Several fields have benefited from nanotechnology, including medicine, information communication, chemistry, the environment, defense and security, consumer goods, and agriculture. By controlling and targeting agrochemical delivery and using diagnostic tools for early pest detection, nanotechnology holds significant promise for managing insects and pathogens. In the present study, green silica nanoparticles were tested to determine if they affect Groundnut Crop Spodoptera litura.

Materials and Methods
Collection and extraction of Macroalgae
The selected seaweed was collected from Manora Beach (Latitude: 10.2644°N and Longitude: 79.2874°E) of Thanjavur District, Tamil Nadu, India. In September 2022, sponges were collected at low tide in intertidal and intertidal zones where vegetation is discontinuous and patchy. Also, the pelleted algae were collected in glass bottles and plastic bags with the help of disposable latex gloves. After collection, sponges were washed three times in tap water and once in sterile distilled water to remove salt, sand, and epiphytes. The algae were cleaned with a cloth and air-dried for fifteen days to ensure safe storage. Algal powder materials were extracted by soxolation and cold permeation using polar solvents (methanol-ME).

Pest collection and rearing
During the hatching process, Larval Spodoptera litura larvae collected from groundnut fields in Peravurani village in Thanjavur district in Tamil Nadu were incubated on fresh castor leaves placed in plastic trays. The experiments were

generally conducted using healthy and homogenous Spodoptera litura larvae of the third instar, and the cultures were preserved throughout the study.

Synthesis of Silica Nanoparticles

Silicon nanostructures were synthesized by the modified Stober method. Triton TX-100 was used as a mild surfactant. A mixed solution (50 ml) of TX-100, methanol, NH4OH, and H2O in the final ratio (0.2:4.6:1:4) was stirred at 50 °C for 30 minutes. 3.0 ml of TMOS was introduced drop wise into the mixture at a constant rate of 10 L/min. The final solution was diluted with 100 ml of H2O and continuously stirred for 42 hours. The resulting silicon dioxide sample was filtered, washed several times with a mixed solution of H2O and ethanol, and dried at 60-80°C. Finally, the residues of the surfactant were removed by heat treatment at 550°C for 6 hours.

Antifeedant Activity

The standard concentration (40%) was achieved by dissolving the crude extracts in methanol. The castor leaf discs were given the following treatments: - 5, 10, 20, 30, 40, and 50 mg/ml solvent extract with one absolute control (methanol) and one standard check In accordance with the treatment schedule, fresh castor leaf discs with a diameter of four cm were immersed in the various treatments (for five minutes). The treated leaf discs were air-dried and kept separate in Petri plates with the required moisture (five leaf discs per Petri plate) (wet filter paper). The following procedures were used to investigate the antifeedant action. The test insects were allowed to feed on treated leaves (free choice method) for up to 24 hours in the laboratory. The % antifeedant indicator was computed using Ben Jannet *et al* formula's (2000).

$$\text{Antifeedant Index} = \frac{C - T}{C + T} \times 100$$

where, C and T represent the amount of leaf eaten by the larva on control and treated discs, respectively.

Larvicidal Activity

Larvae fed castor genus Spodoptera litura leaf disc (various compound concentrations) treated for twenty-four h were unceasingly maintained on untreated fresh leaves. The death of larvae was recorded when twenty-four hours after treatment. Replicates with five larvae per replicate (20 people in total) were unbroken for every treatment. Mortality was calculated per Abbott's (1925) formula. Throughout the antifeedant activity study, the conditions within the laboratory were equivalent to those within the present study.

$$\text{Abbott's corrected mortality} = \frac{\%\text{ mortality in treatment} - \%\text{ mortality in control}}{100 - \%\text{mortality in control}} \times 100$$

Results and Discussion

Worldwide, algae are used in agriculture, food, and medicine (Stranska-Zachariasova *et al*., 2017). Various algal secondary metabolites have been reported to have protective properties against invertebrates and especially insects. The chloroform and benzene extracts of Padina pavonica were reported to kill 85% of Dictyota cingulatus nymphs by Sahayaraj and Kalidas (2011) [21]. Osmunda pinnatifida also showed insecticidal properties (Rizvi and Shameel, 2003). A recent study was conducted on the insecticidal properties of plant materials, especially secondary metabolites and essential oils (Senthil Nathan *et al*, 2008) [23], establishing that they were eco-friendly, biodegradable, and species-specific.

Insecticides based on Silica Nanoparticles

Silica Nanoparticles are efficient pesticides which can be used alone or in addition with other commercial pesticides to obtain the desired result, according to studies. Animal-derived SiO2NPs were injected into the plant with garlic essential oil, and the modified hydrophobic Nano silica with a surface charge (3-5 nm) can be successfully used to control agricultural insect pests. SiNPs have been documented in two ways: as field pesticides, to control insects and larvae, or as nanocarriers, to deliver commercial pesticides to increase their effectiveness. SiO2 nanoparticles were reported to be lethal to Callosobruchus maculata (Rouhani *et al*., 2013) [20]. The lethal effect of SiNPs on insects may be due to constriction of the spiracle and trachea, as well as absorption damage and abrasion of the surface protective wax layer. Nano-silica is believed to kill insects by physical adsorption, which disrupts the protective hydrophilic lipid membrane and results in the death of the target organism. SiNPs are adsorbed and degraded, resulting in the death of insects due to desiccation (Ayoub *et al*., 2017; Elnahal *et al*., 2022) [1, 5]. These NPS can also damage the digestive system of herbivorous insects that consume SiNP-treated plants (Thabet *et al*., 2021) [24]. The direct effects of SiNPs on a wide range of insects have been studied on a laboratory scale (Mousa *et al*., 2014) [15]. Field exposure to SiNPs was evaluated on an insufficient number of insects; they are harmful and dose-dependent. Species counted included biting insects (moth: Spodoptera littoralis), burrowing insects (aphids: Aphis granivore), and internal feeders (miner leaf fly: Liriomyza trifolii) (Thabet *et al*., 2021) [24]. Hormonal signaling was detected in rice-shaped SiNPs (absorbing Cnaphalocris medinalis leaf folder against Trathalaflavor orbitalis parasite and Microplitis mediator) (J. Liu *et al*., 2017) [10].

Mortality of *Spodoptera Litura* caused by silica nanoparticles

Spodoptera litura third-instar larvae were tested for their antifeedant activity by methanol extracts of Padina Tetrastomatica. Among the treatments, metallic Silica nanoparticles @ 50 mg/ml had the highest mortality rate at 97.56%, which was significantly higher than other treatments. 84.98 percent of mortality was achieved with green silica nanoparticles @ 40 mg/ml. In the control group, no mortality of S.litura appeared with only survivals being observed in treated groups. The tested concentrations of green Silica nanoparticle 5 mg/ml had the highest survival rate of 29.90% while the lowest was 26.33%. The results supported that the extracted extracts effectively induced larval mortality against the third instar of Spodoptera litura. The most death tolls for gemcitabine were recorded in silica nanoparticles @ 50mg/ml and it was remarkably better than the other treatment groups. Considering that there are no available reviews on the effect of silica green nanoparticles on other insects the effects of silica green nanoparticles on these insects are discussed here. Groundnut crop Spodoptera litura was effectively managed with green silica nanoparticles at 50 mg/ml. Results suggest the possibility of applying green silica nanoparticles to eradicate pests and can be used as valuable tools in pest management. Although silica nanoparticles as insecticides have yet to be studied for their environmental impacts, one clear advantage of their use is the low likelihood of insect resistance developing after long-term use.

Table 1: Antifeedant and Larvicidal Activity of synthesized Si NPs against *Spodoptera Litura*

Sample Extract	**Concentrations (mg/ml)**	**%Mortality (mg/ml)± SD**	
		Antifeedent	**larvicidal**
Synthesized Si NPs	5	26.33±4.89	29.90±2.12
	10	32.22±5.13	34.89±3.22
	20	36.74±5.24	39.76±4.12
	30	54.33±5.68	68.54±4.98
	40	84.98±5.90	89.22±4.21
	50	97.56±5.62	109.92±0.00

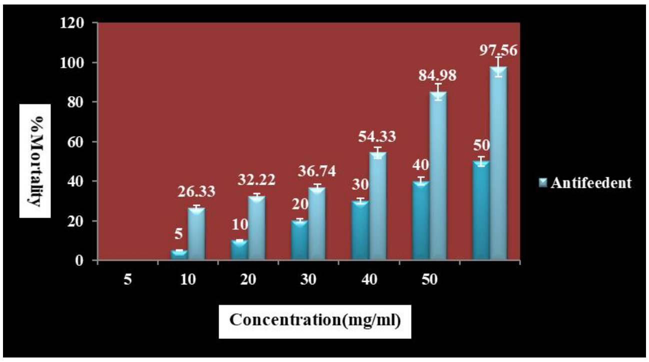

Fig 1: Antifeedant activity of synthesized Si NPs against *Spodoptera Litura*

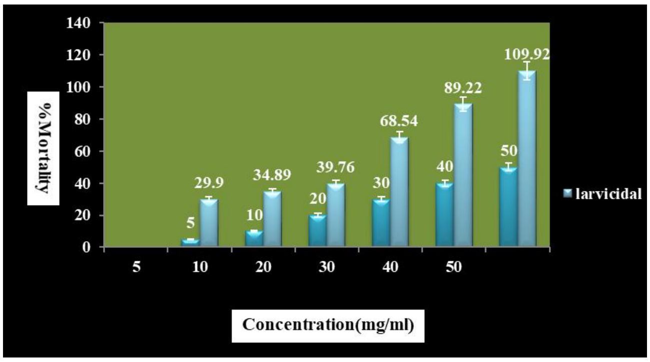

Fig 2: Larvicidal activity of synthesized Si NPs against *Spodoptera Litura*

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