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Green Synthesis Of Zinc Ferrite Nanoparticles Using *Tagetes Erecta* Flower (Marigold Yellow) And Their Application As Photocatalysis

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Abstract: -

Zinc ferrite ($ZnFe_2O_4$) nanoparticles (NPs) were significant because of their Photocatalytic activity. In this study, zinc ferrite nanoparticles were synthesized by a green method using *Tagetes erecta* flower (Marigold yellow) extract were studied. The synthesized $ZnFe_2O_4$ samples were characterized by XRD and UV-Visible spectroscopy. X-ray diffraction confirms the formation of $ZnFe_2O_4$ nanoparticles with an average crystallite size of 7.11 nm. The $ZnFe_2O_4$ nanoparticles showed effective photodegradation for Methylene blue under visible light irradiation.

Keywords: $ZnFe_2O_4$, XRD, Methylene blue, UV-Visible

1. Introduction:

Nanocrystalline constituents have fascinated significant attention because of their exclusivity and outstanding properties in numerous fields including physics, chemistry, biology, medicine, materials science, and engineering. Nanomaterials have a particle size in the 1–100 nm range and a high surface-to-volume ratio that determines various or better reactivity, and thermal, mechanical, optical, electrical, and magnetic properties [1]. Although in the case of bulk materials, the chemical composition is the main factor that defines their properties, in the case of nanomaterials, In addition, the chemical composition, particle size, and morphology determine most of their characteristics [1,2]. In addition, these properties can be tuned based on particle size and chemical composition [1,2].

At the present time, basic dyes are preferably used in industries for numerous dyeing or printing purpose owing to their brightness and high color density [28]. The polluted water coming from dye or textile industries is very challenging to handle and resilient to microbial degradation owing to the complex structure of dyes which makes them more stable [29,30]. Besides, the chemicals used in dye preparation are highly poisonous or harmful and cause incalculable health risks to human beings [31,32]. Several methods have been reported for the removal of synthetic dyes such as filtration, ion exchange, photocatalytic degradation, and adsorption [33,34]. Among these methods, photocatalytic degradation is an efficient technique because of affluence in the process, and cost-effectiveness, and it involves the application of very reactive and impressive oxidative free radicals for nonselective mineralization of the dyes [35,36].

Among ferrites, the reverse type is most attractive due to its great magneto crystal-like anisotropy, and high saturation magnetization from a distinctive crystal and magnetic structure. In general, zinc ferrite ($ZnFe_2O_4$) is a well-known reverse spinel ferrite with Zn^{2+} in B (octahedral) sites and Fe^{3+} ions that are separated correspondingly among A (tetrahedral) and B (octahedral) sites. Subsequently, $ZnFe_2O_4$ nanoparticles have been fascinated widely due to their excellent phase stability, high magnetic permeability, high electronic conductivity, low eddy current loss, bandgap (~1.9 eV), low cost of production, and non-toxicity [29]. $ZnFe_2O_4$ nanoparticles have similarly fascinated the attention because of their potential applications in the field of drug delivery, magnetic hyperthermia, sensing applications, magnetic resonance imaging (MRI), antibacterial and photocatalytic activity [30]

In photo-catalysis, a number of heterogeneous photo-catalysts including TiO_2, Fe_3O_4, Cu_2O, ZnO, and WO_3, have been tried by researchers for environmental remediation [37]. Magnetic ferrites have gained received phenomenal attention in photocatalysis due to their compatibility with modern technologies. The zinc ferrites have shown tremendous potential in the field of photo-catalysis due to a narrow bandgap of about 1.9 ev and are capable of absorbing visible light [38].

Greener synthesis of metal nanoparticles offers advancement over other traditional chemical methods due to simple, safe, one-step, and cost-effective practice which eliminates the risk of toxicity in our system and produces stable reproducible materials [39,40]. Generally, the extracts of plant parts [41,42,43], bacterial or fungal biomass [44], and biopolymers [45] are exploited for the production of nanoparticles. The plant extract has a wide variety of aromatics and phenolic species that not only reduces the metal salt but also stabilizes the engineered nanoparticles [46,47,48].

Zinc Ferrites are substantial and attractive materials, from a practical along with essential point of view. nanosized $ZnFe_2O_4$ have fascinated significant courtesy due to their chemical and thermal constancy and exclusive structural, optical, magnetic, electrical, and dielectric properties and wide potential technological applications in photoluminescence, photocatalysis, humidity sensors, biosensors, catalysis, magnetic drug delivery, permanent magnets, magnetic refrigeration, magnetic liquids, microwave absorbers, water decontamination, ceramics pigment, corrosion protection, antimicrobial agents, and biomedicine (hyperthermia) [**1**,**3**,**4**,**5**,**6**,**7**,**8**].

$ZnFe_2O_4$ has revealed extraordinary applications in magnetic resonance imaging, sensor [9], catalysis [10], pigments [11] and drug delivery [12]. Zinc ferrite nanoparticles have good electrical, optical and magnetic properties, these activities hinge on the composition, size, and shape of the nanoparticles [13]. At the present time, scientists focused on the synthesis of $ZnFe_2O_4$ nanoparticles because properties of ferrite magnetic nanoparticles are diverse from other spinel nanoparticles [14]. Synthesis of nanoparticles is conveyed by various methods such as physical, chemical and biological. In physical, Laser ablation and Evaporationcondensation method are used for the synthesis of thin films and uniform nanoparticles [15]. Sodium citrate, ascorbate, sodium borohydride, Tollen's reagent is used as reducing agents in the chemical method [16]. These methods have some drawbacks such as high cost, usage of toxic chemicals and environmental pollution. To overcome these problems, scientists focused on eco-friendly, cost-effective, non-toxic plant extracts for the synthesis of nanoparticles. Hence the green synthesis is the best method compared to the physical and chemical method synthesis of nanoparticles.

In the last few ages, the number of papers that review data on nanosized ferrites with a focus on diverse ferrites, diverse synthesis methods, or diverse applications suggestively increased. The Core Collection have indexed (**1812)** publication that contains the words "ferrite", Patent **(1,032),** and "review" (**32)** in their title of which were published between 2016 and 2021. Zate et al. [**17**] reviews the mechanical, chemical, spray and electrospinning methods used for the synthesis of different ferrites with magnetic properties. Vedrtnam et al. [**18**] reviews the properties, classification, synthesis, and characterization of hexagonal and spinel ferrites with a focus on the main four synthesis routes (sol-gel, hydrothermal, co-precipitation and solid-state), magnetic properties, and characterization of the ferrites. Vinosha et al. [**19**] review the recent advances of synthesis, magnetic properties, and water treatment applications of cobalt ferrite, while Masunga et al. [**20**] reviews the recent advances in copper ferrite synthesis, magnetic properties and application in water treatment. Kumar et al. [**21**] review magnetic nano ferrites and their composites used in the treatment of pollutants from waste waters, emphasizing pros and cons of several synthetic pathways, the adsorption mechanism, and ferrite regeneration, while Kefeni and Mamba [**22**] review the photocatalytic application of spinel ferrite NPs in pollutant degradation with emphasis on the possible recovery and reuse of NPs. Kharisov et al. [**23**] review the use of cobalt, nickel, copper, and zinc ferrites and their doped derivates as catalysts in organic processes, while Dalawai et al. [**24**] overview the spinel-type ferrite thick films together with preparation strategies and sensors, microwave, magnetic, and advanced applications. Kefeni et al. [**25**] review the ferrite's applications in electronic devices, such as sensors and biosensors, microwave devices, energy storage, electromagnetic interference shielding, and high-density recording media together with the advantages and drawbacks of most important ferrite NPs synthesis methods

2. Practical: -

2.1 Materials and methods: -

Zinc nitrate hexahydrate [Zn $(NO_3)_2 6H_2O$], Ferric nitrate Nona-hydrate [Fe $(NO_3)_3 9H_2O$], Methylene blue are analytical grade chemicals. Indian *Tagetes erecta* flower (Marigold yellow) were taken from the Titwala mandir [Thane, Maharashtra]

2.2. Preparation of plant extracts: -

Tagetes erecta flower (Marigold yellow) were collected, dried in oven (100 ^{0}c) for 48 h, and powdered. 5g of powder was added into the 100 ml of distilled water and kept on heating plate for boiling at 200 °C for 1 h. After boiling extract were formed. The extracts were cooled and filtered using filter paper. The derived extracts were named as, Tagetes erecta flower (Marigold yellow) aqueous extracts.

2.3. Synthesis of Zinc ferrite: -

Zinc nitrate (3.00 g) and Ferric nitrate (8.00 g) were taken in the ratio of 1:2 dissolved distinctly in 10 ml of normal water, both the solution was mixed in one beaker and 10 ml of plant extracts was added. Stirred continuously for 5 h at 100°C. The synthesized nanoparticles were dried at 180 °C for 5 h and calcinated at 600 °C for 4 h in Muffle furnace. The attained $ZnFe_2O_4$ powder sample was characterized and its photo catalytic activity were studied.

2.4. Characterization: -

The physical characterization of the synthesized nanoparticles was examined using X-ray diffraction (XRD) [BRUKER D2 Phaser], UV–visible Diffuse Reflectance Spectroscopy [Systronics Double beam spectrophotometer 2203]

2.5. Photocatalytic Activity: -

Photodegradation studies of green synthesis of $ZnFe_2O_4$ nanoparticles were evaluated under visible light irradiation for Methylene blue (MB) dyes. The photodegradation process was carried out in a photoreactor. The $ZnFe_2O_4$ photocatalyst (50 mg) was added to a quartz tube containing 5 ppm of dye in 250 mL aqueous solution. To attain the adsorption/desorption equilibrium the reaction mixture was bubbled in dark for 30 min. The reaction mixture was irradiated at room temperature under 300 W tungsten visible light lamp. Furthermore, the aliquots were collected at an interval of 15 min first then after 10 min to find the degradation percentage of the dye solution. The dye residuals were tested by examining the absorbance at 670 MB. The photodegradation of dyes were measured by the following relation

$$percentage\ of\ degradation = \frac{Co - Ct}{C_o} \times 100$$

Where Co is the initial absorbance of the dye solution and Ct is the absorbance of the dye solution after time t in min, respectively.

3. Results and Discussion: -

3.1 X-ray diffraction (XRD) Analysis: -

The XRD pattern of the sample showed prominent diffraction peaks at 2θ values of 29.99°, 31.82°, 34.52°, 35.37°, 36.29°, 56.68°, 62.26°, 64.05°, 68.02°, corresponding to the (220), (311), (222), (400), (422), (511), (440), (620), and (533) respectively crystallographic planes of the cubic phase of the material. The relative intensities of the peaks indicated a high degree of crystallinity, while the broad peak widths suggested the presence of small crystallites with a mean size of approximately 7.11 nm. The crystal structure was confirmed to be consistent with the standard reference pattern for the cubic phase, and no evidence of preferred crystallographic orientation or texture was observed." This confirms the cubic structure of $ZnFe_2O_4$ nanoparticles according to the JCPDS No: 01079150. Figure shows $ZnFe_2O_4$ nanoparticles formed at 600 °C.

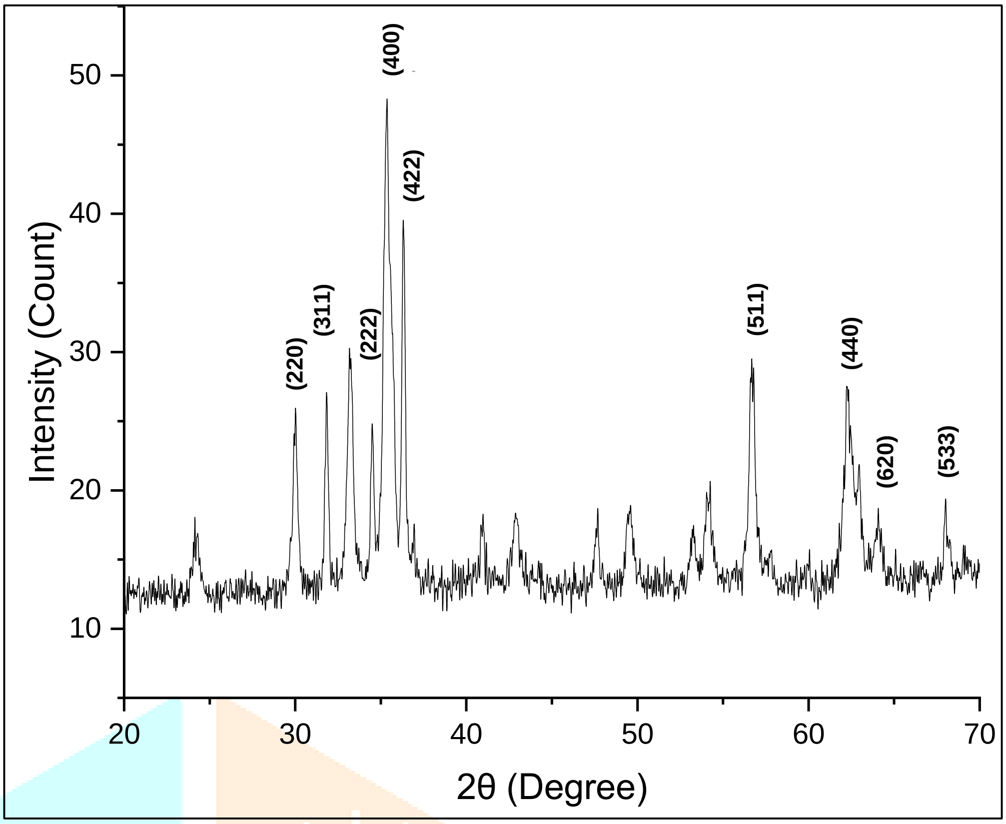

Fig 1. XRD pattern of ZF-NPs.

3.2. UV-Visible analysis: -

Figure 2 shows the UV-Visible spectrum of *Tagetes erecta* flower (Marigold yellow) extracts synthesised $ZnFe_2O_4$ nanoparticles in the wavelength range from 350–800 nm. The UV-VISIBLE spectrum of the sample showed a broad absorption band centered around 402 nm, which can be attributed to the π-π* transition of the conjugated system in the molecule. Synthesised zinc ferrite nanoparticles using green synthesis method and the absorption peak was noted around 385–415 nm. This could be due to the excitation of electron from O-2p level to Fe-3d in spinel type compounds. The optical band gap of the prepared samples was determined using diffused reflectance spectra obtained from a UV-Visible spectrophotometer and is shown in the figure 2. The incorporation of $ZnFe_2O_4$ conveys important development in the optical response of the compound. The significant decrease in the optical band gap $ZnFe_2O_4$ (i.e., 2.95 eV) makes it a more responsive photocatalyst, which supports the generation of electron-hole pair and resultantly the advanced hydroxyl radicals.

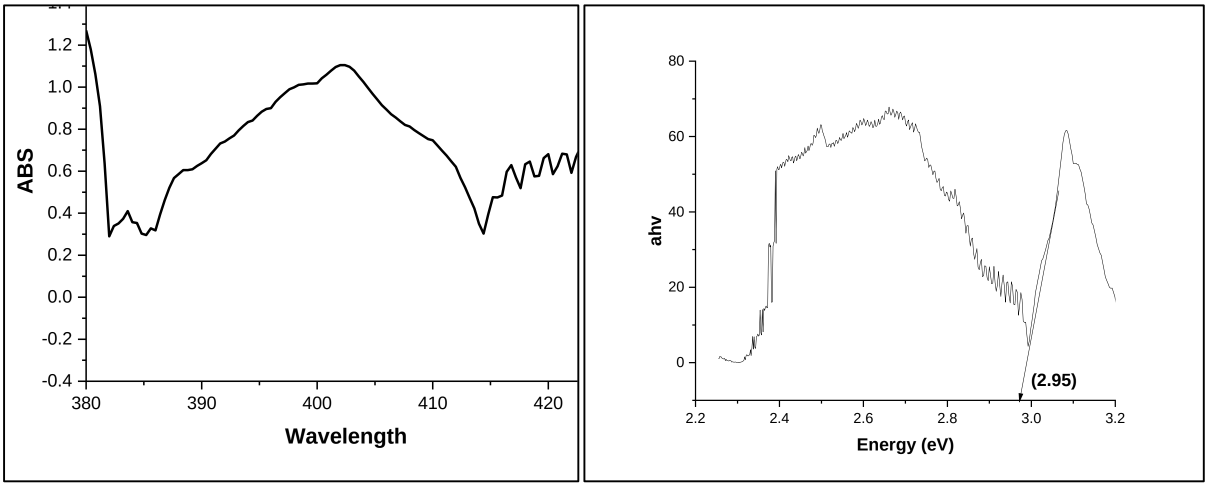

Fig 2. Uv- Visible spectrum and Band gap of *Tagetes erecta* flower (Marigold yellow) extracts mediated synthesis of ZnFe2O4.

3.3. Photocatalytic degradation of MB: -

The photocatalytic efficiency of $ZnFe_2O_4$ nanoparticles was studied using specific decolourization of MB into inorganic species (CO_2 and H_2O) as a model reaction in the presence of visible light. The main reason for its selection is that its absorption peaks fall in the visible region, therefore, its decolourization could be easily observed through UV-VISIBLE spectrophotometer by measuring a reduction in absorbance value with time at the λmax (670 nm) along with its less definite peak at 670 nm. The photocatalytic degradation of MB is of exact importance because it is a toxic pollutant and can cause vomiting, nausea, cyanosis, mental confusion, increase heart rate, jaundice, tissue necrosis and can induce methemoglobinemia problems when ingested through the mouth [49,50]. Figure 3 shows time-dependent UV-Visible spectra of MB decolourization that display reaction completion within 75 min. Additionally, the spectra were not seemed to be affected by surface Plasmon resonance peak because the catalyst is used in a very small amount. The parameters affecting the photo catalysis of MB, possible degradation mechanism, and reuse of ZF-NPs also has been discussed in Supporting Information (SI). The complete disappearance of the blue colour confirms the completion of the reaction. The photocatalytic decolourization of MB in the presence of ZF-NPs displays excellent degradation efficiency due to the presence of photocatalyst that makes reaction feasible to cross the activation energy barrier.

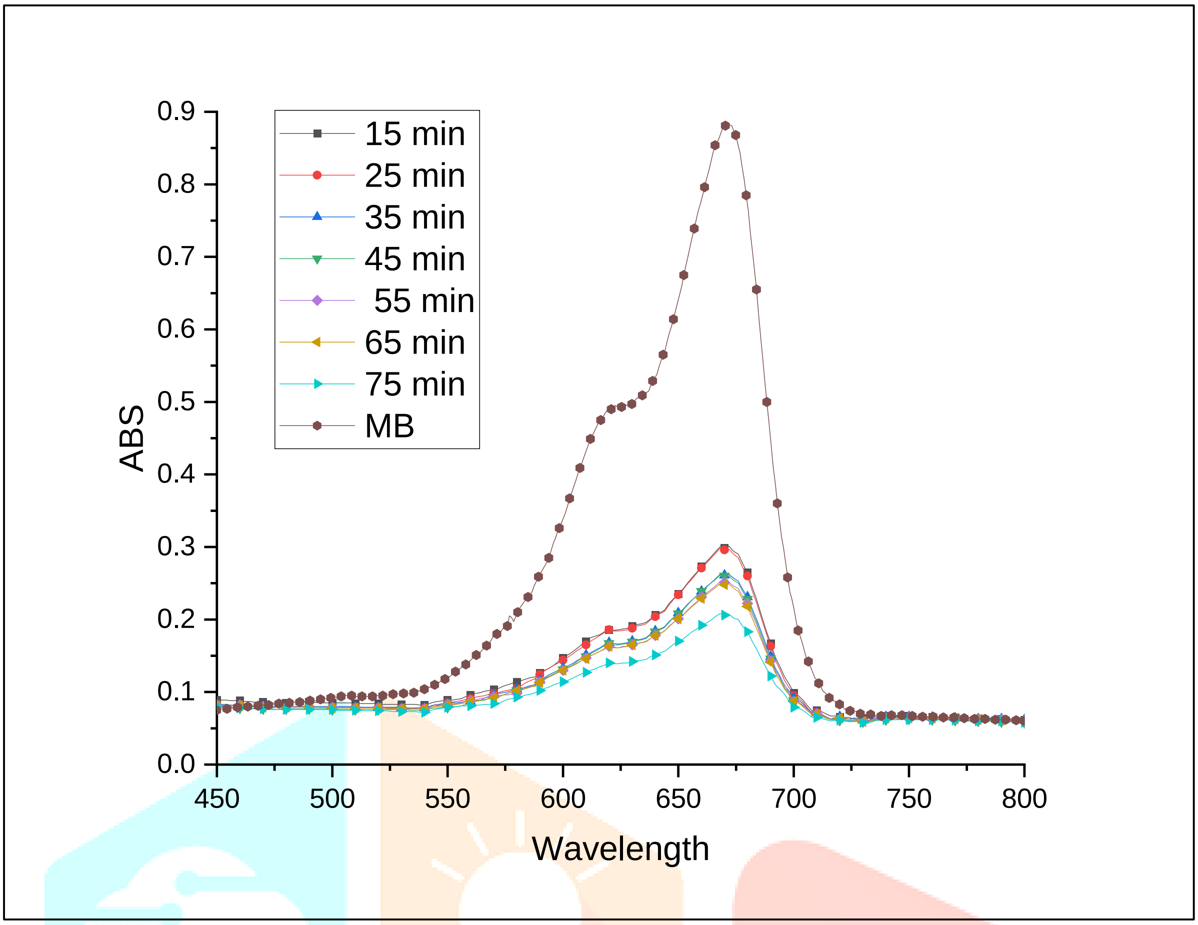

Fig 3. MB degradation at various time intervals (reaction conditions): [ZF-NPs] =0.5 g; [MB]=5 ppm; visible light;

The above figure 3 shows the degradation of Methylene blue dye with respect to time. Methylene blue was degraded under visible light up to 76% when kept for 75 min using catalyst $ZnFe_2O_4$ nanoparticles. Less amount (0.5) of $ZnFe_2O_4$ nanoparticles and less time was required to degrade mb these factors increased the efficiency of degradation process. the np can be filtered and reused for further degradation process

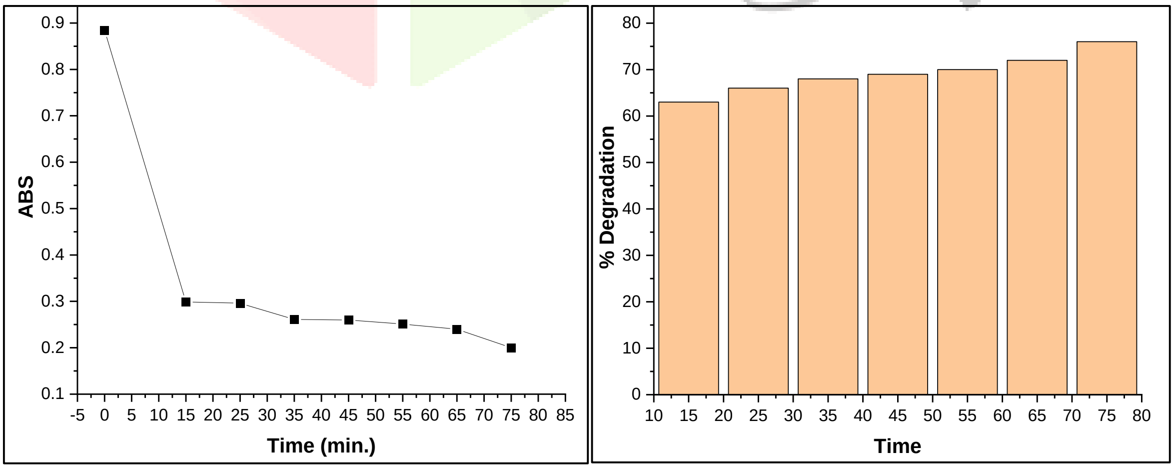

Fig 4. Absorbance Vs Time in Min. And Percentage Degradation Vs Time in Min

Conclusion: -

In this research work we synthesize zinc ferrite nanoparticles. Because of its good photocatalytic activity. For synthesis of $ZnFe_2O_4$ nanoparticles we use green method i.e., Plant sources *Tagetes erecta* flower (Marigold yellow) using calcination at 600°C.We obtain particle size using XRD is 7.11 nm. Synthesised $ZnFe_2O_4$ nanoparticles were non-hazardous and eco-friendly. The prepared $ZnFe_2O_4$ nanoparticles show substantial photocatalytic activity for Methylene blue dyes.

We perform photocatalytic activity of above synthesized nanoparticles are shown in below table.

Sr. No.	Source	% Degradation	Time (min)	Size (nm)	Band Gap (e.V)
1.	*Tagetes erecta flower* (Yellow)	76%	75	7.11	2.95

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