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De-fluoridation of Groundwater using Precipitation Method in a Region of Nagaur District, Rajasthan, India

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Abstract Safe drinking water is a human right. Groundwater is polluted by number of chemical elements which includes fluoride too. Deteriorated quality of drinking water due to high concentration of fluoride is one of the major issues in India including in the state of Rajasthan. Although various de-fluoridation methods are available, in Indian context the Nalgonda Technique and Activated Alumina has been mostly adopted. Alum is used as the coagulant in Nalgonda method. However, it has some disadvantages like high sludge volume, residual aluminium and wearing effects on the treatment structure. In Nalgonda Technique, residual Aluminium in treated water is found to be higher than the permissible limit, i.e. 0.2 mg/L. Therefore, an alternative compound other than Alum for de-fluoridation assumes great importance. We aim to reduce the use of Alum in this research work. Ferric Ammonium Sulphate was used as precipitating agent for removal of fluoride ion from groundwater collected from few localities of Nagaur district of Rajasthan State. In order to remove fluoride ion from water, standard solutions of fluoride and Ferric Ammonium Sulphate of different concentrations were prepared and initial and final concentrations of Fluoride solutions were determined by Fluoride ion meter. The affinity of Ferric Ammonium Sulphate for fluoride ion was studied using different solutions as the Sodium fluoride solution containing Fluoride ion. The removal of fluoride ion increases with increasing volume of 2% and 5% solution of Ferric Ammonium Sulphate.

Keywords: fluoride, groundwater, clinical manifestations, coagulation, fluorosis, potash alum; polyelectrolyte; precipitation, reverse osmosis and rainwater harvesting

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1. Introduction

Water is one of the natural resource of paramount importance. It is essential for sustenance of all forms of life and is available in abundance in nature covering approximately three fourths of the surface of the earth. The chemical nature of water is one of the most important criteria that determines its usefulness for a specific need and as such not all the waters are fit for drinking; hence the problems of scarcity of drinking water. High concentration of fluoride in excess of permissible limit, deteriorates quality of groundwater, which may be geogenic as well as anthropogenic in origin. Leaching of Fluoride from the world crust is that the chief source of fluoride content in spring water. However the other sources like food chain also add to increase the overall ingestion of fluoride into the human body. Fluoride in drinking water is known for both beneficial and detrimental effects on human health when it is in deficit as well as in excess concentrations. The fact that the problems associated with the excess fluoride in drinking water is highly endemic and widespread in Rajasthan state especially in Nagaur district. This has prompted many researches to explore quite a good number of materials (both organic and inorganic) for adopting various de-fluoridation processes like coagulation, precipitations, adsorption, Ion exchange etc. [1,2,3] Some processes are good under certain conditions while others are good in other conditions. Current study was carried out in Nagaur district, which is located almost in the middle of the state of Rajasthan. [4]

2. Fluoride in Environment

Fluorine (F₂) is a greenish diatomic gas. Fluorine is so highly reactive that it is never encountered in its elemental gaseous state except in some industrial processes. The fluoride occurs notably as Sellaite, Fluorite or fluorspar (CaF₂), Cryolite (Na₃AlF₆), Fluorapatite, Ca₃(PO₄)₂, Other minerals containing fluoride are given within the Table 1. Fluorspar it is found in sedimentary rocks and Cryolite in igneous rocks. These Fluoride minerals are nearly insoluble in water. [5] Hence fluorides will be present in groundwater only when conditions favors their solution. It is also present in sea water (0.8-1.4 ppm), in mica and in many beverage supplies.

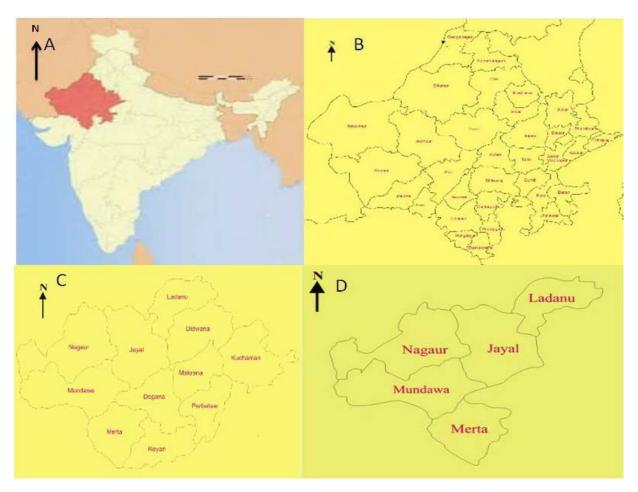


Figure 1. Location map of study area [6]

Table 1. Fluoride bearing minerals

	e	
Mineral	Chemical Formula	% Fluorine
Sellaite	MgF_2	61%
Villianmite	NaF	55%
Fluorite (Fluorspar)	CaF_2	49%
Cryolite	Na_3AlF_6	45%
Bastnaesite	(Ce,La) (CO ₃)F	9%
Fluorapatite	Ca ₃ (PO ₄)3F	3-4%

It is evident from the available information that a particular quantity of fluorine is important for the formation of caries-resistant dental enamel and for the traditional process of mineralization in hard tissues. The element is metabolized from both electrovalent and covalent compounds. Low Fluoride concentrations stabilize the skeletal systems by increasing the dimensions of the apatite crystals and reducing their solubility. About 95% of the fluoride in the body is deposited in hard tissues and it continues to be deposited in calcified structure even after other bone constituents (Ca, P, Mg, CO₃ and citrate) have reached a steady state. Age is an important factor in deciding to what extent fluorine is incorporated into the skeleton. The uptake almost ceases in dental enamel after the age of about 30 years.

3. Hydrogeology

Hydro geologically the whole district can be classified into three formations viz. consolidated formation,

formation semi-consolidated and unconsolidated formation. The consolidated formations comprise of metamorphics like schists, gneisses, quartzites and phyllites of Precambrian age and limestone and sandstone of Marwar Supergroup belongs to upper Precambrians. Metamorphics are normally impervious except in the presence of a few weak planes, joints, weathered zones and kinks which contain moderate and limited quantity of groundwater [7]. These are basically phreatic aquifers and availability of groundwater depends on good amount of precipitation. Such aquifers are mainly confined to eastern part of Riyan and Parbatsar blocks, central part of Makarana block, eastern part of Ladnun block and northern part of Didwana block. Jodhpur sandstone mainly consists of medium to coarse grained sand, cemented with silica and ferruginous matrix. The sandstone is intercalated with siltstone and shale. The sandstone is hard, compact and forms medium potential aquifer. Wherever, groundwater occurs, it mainly occupies either void space between the adjacent grains (primary porosity) and in the secondary porosity zones. Jodhpur sandstone mainly occurs in south-western part of Mundwa block and central part of Ladnun block. Groundwater in this formation occurs under semi-confined to unconfined conditions. Thickness of sandstone varies from 100-250m. Bilara limestone forms the most important and potential aquifer comprising limestone, dolomite and shale. The limestone is white to grey in colour, hard and compact, cherty and dolomitic in nature. However, it is cavernous at places and susceptible to solution activity which gives rise to high discharge in wells. This formation covers western

and north-central parts of Nagaur block, central part of Mundwa block, west central & eastern parts of Jayal block and part of Ladnun block. Thickness of limestone varies from 100- 300m. Nagaur sandstone is coarse to fine grained, loosely cemented with gravel at basal part which acts as good aquifer and occupies mainly parts of Nagaur, Jayal, Mundwa and Merta blocks. The associated rocks are siltstone and shale. Its thickness varies from 140-240m. Semi-consolidated formation includes only Palana sandstone consisting of very coarse grained, gravelly sand with intercalations of clay with kankar and lignite. Groundwater occurs under phreatic to confined conditions and saturated thickness of 40m constitutes a potential aguifer. This mainly occurs in parts of Merta, Mundwa and Jayal blocks. Its thickness varies from 100-250m. Unconsolidated Quaternary alluvium is the main aquifer which is comprised of unconsolidated to loosely consolidated fine to coarse grained sand having intercalations and intermixing with silt, clay with 'kankar'. Groundwater occurs under unconfined to semi-confined conditions. The Quaternary alluvium covers parts of Rivan, Merta, Degana, Parbatsar, Makarana, Kuchaman, Didwana, Ladnun and Jayal blocks. Its thickness is limited to 200 m. [8]

Block	Village Site	Sample Source	
Nagaur	Chenar, Basra, Nagaur City	Hand Pump, Tube Well	
Ladnu	Kasumbi, Rodu, Dholiya, Sardi	Hand Pump, Tube Well	
Jayal	Deh, Soneli, Rajod, Kusumbi	Hand Pump, Tube Well	
Merta	Lampolai, Dangawas, Merta City	Hand Pump, Tube Well	
Mundwa	Kharnal, Panchla Sidha, Khajwana, Kurchi, Roon	Hand Pump, Tube Well	

Quality of groundwater in phreatic aquifer varies widely from saline to fresh. Electrical Conductivity (EC) ranges between 1380 to 16240 µS/ cm at 25°C. It has been observed that by and large, EC conforms broadly to chloride concentration. In greater part of the area, it is within 5000 μS/cm at 25°C. Higher values of EC have been observed in the west central part of the district and also in depressions in the vicinity of the saline lakes. The chloride content ranges from 50 to 5069 ppm in phreatic aquifer. Fluoride in the ground water ranges between traces and 11.20 mg/l. [9] Fluoride concentration in excess of maximum permissible limit of 1.5 mg/l has been noticed in central and northeastern parts of the district. Nitrate concentration in ground water varies widely. Its concentration ranges between traces to as high as 1000 ppm. [10]

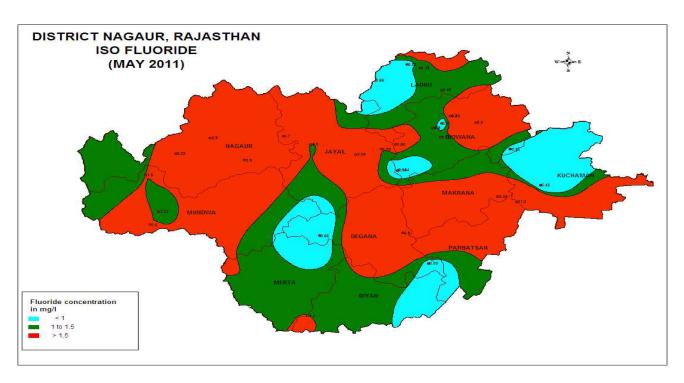


Figure 2. Location map of Fluoride affected area of study region

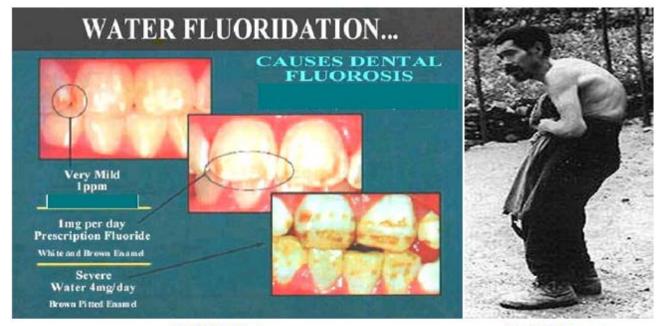
4. Clinical Manifestations of Fluoride Toxicity

Long term exposure to fluoride through various fluoride containing water and other products leads to development of fluorosis, which is also known as a crippling and painful disease. Fluoride predominantly affects the teeth, skeletal systems and also the structure and function of skeletal muscle, brain and spinal cord. [11] Dental fluorosis occurs during the period of enamel formation. It is linked to excessive incorporation of fluoride into dental

enamel and dentine, which prevents normal maturation of enamel. Prolonged intake of high fluoride in drinking water leads to staining of teeth, which became yellow, brown or black in colour. General symptoms of acute fluoride poisoning include non-skeletal fluorosis viz. nausea, salivation, vomiting, diarrhoea and abdominal pain. [12] Fluoride is also found to be involved in the alteration of metabolism of some essential nutrients which leads to hyperlemia, hypocalcemia, hypomagnesemia, hypopophosphatemia. Persistent fluoride serum level leads to mineral homeostatis which ultimately causes cellular damage. Chronic fluoride toxicity occurs after the

long-term ingestion of even small amount of fluoride beyond permissible limit. It inhibits the synthesis of DNA (Deoxyribose Nucleic Acid), protein and inhibits cell proliferation and cytotoxic at the high doses. [13] Symptoms of long term fluoride toxicity include emaciation, stiffness of joints, hyper salivation, tremors, tetanic contractions and abnormal teeth and bones. Other effects include lowered milk production and detrimental effects on reproduction. Fluoride is known to cross the blood brain barrier and accumulate in the brain of animals exposed to high fluoride levels. [14] Recent studies have

shown accumulation of fluoride within the hippocampus of the brain causing degeneration of neurons, decreased aerobic metabolism and altered free-radical metabolism in liver, kidney, and heart. [15] Skeletal fluorosis is a pathological condition which includes inhibition of bone hardening (mineralization), causing the bones to become brittle and their tensile strength may be reduced. Symptoms include limited movement of joints, skeletal deformities, and intense calcification of ligaments, muscle wasting and neurological defects .The disease fluorosis is irreversible in nature. [16]



Dental Fluorosis

Skeletal Fluorosis

Figure 3. Dental & Skeletal Fluorosis

5. Mechanism of Toxicity

High concentration of Fluoride in drinking water leads to toxicity as follows:

- Binds calcium ions and may lead to hypocalcemia which could further lead to osteoid formation.
- Disrupts oxidative phosphorylation, glycolysis, coagulation, and neurotransmission (by binding calcium).
- Inhibits Na⁺/K⁺-ATPase (Adenosine Tri Phosphotase), which could lead by hyperkalemia by extracellular release of potassium.
- Inhibits acetyl cholinesterase, which may be partly responsible for hyper salivation, vomiting, and diarrhea (cholinergic signs). [17,18]

6. De-fluoridation of Water

De-fluoridation of drinking water is considered as the only practicable option to overcome the problem of excessive fluoride in drinking water, where alternate source is not available. During the years following the discovery of fluoride as the cause of fluorosis, extensive research has been done on various methods for removal of fluoride from water and wastewater. These methods are

based on the principal of adsorption, ion-exchange, precipitation -coagulation, membrane separation process, electrolytic de-fluoridation, electro dialysis etc. In-situ de-fluoridation with high fluoride litho-formations is a difficult task and has not been so far tried in field or laboratory. [19,20]

6.1. Methods and Materials for Present Studies

6.1.1. Preparation of Solutions

All the reagents used for present de-fluoridation studies were of commercially available high purity grade (Hi media, E-merck, sd-fine, India). Stock solution of fluoride were prepared from sodium fluoride by dissolving 2.210 gm into double distilled water and then diluting to 1L.

- a. Preparation of 2% Ferric Ammonium Sulphate:
 2.00 gm of FAS dissolve into 100 ml of double distilled water.
- b. Preparation of 5% Ferric Ammonium Sulphate: 5.00 gm of FAS dissolve into 100 ml of double distilled water.
- c. The chemical processes, though admittedly are not fully understood, can be seen below:
 - d. $Al_2(SO_4)_318H_2O => 2Al + 3SO_4 + 18H_2O$

 $2AL + 6H_2O => 2Al(OH)_3 + 6H^+$ F' + Al(OH)_3 => Al-F Complex +undefined product $6Ca(OH)_2 + 12H^+ => 6Ca^{2+} + 12H_2O$

e. The amount of $Al_2(SO_4)_3$ added must be carefully monitored as both left over aluminum can cause significant health problems including neurological, cardiovascular, and respiratory problems among others and must be kept under $0.2 mg/L^{\#2}$

f. The dose of aluminium salt increases with increase in the fluoride and alkalinity levels, which are manipulated by adding lime, the raw water. The selection of either aluminium sulphate or aluminium chloride also depends on sulphate and chloride contents of the raw water to avoid them exceeding their permissible limits. The dose of lime is empirically 1/20th that of the dose of aluminium salt, but also depends on the alkalinity of water. Lime facilitates forming dense floc for rapid settling and also, neutralization of H^{\dagger} ions released during the coagulation process. Bleaching powder is added to the raw water at the rate of 3 mg/L for disinfection $^{\sharp 1}$

6.1.2. Determination of Fluoride Concentration

The concentration of fluoride ion in the different solutions was determined using a fluoride ion selective electrode,

which measures concentrations from 10⁻⁶ M to saturated solution. TISAB-III (Total Ionic Strength Adjustment Buffer) was added to the solutions to reduce the variation in the ionic strength in the samples. This buffer contains a chelate, which forms complexes with other ions, such as iron, aluminum that could interface in the determinations. A calibration curve was obtained using NaF standard solutions with different fluoride concentrations from 1 to 10 mg/L. The results were plotted as fluoride concentration (mg/L) versus potential (mV).

Processes like electro-dialysis, ion exchange and reverse-osmosis require special equipment, power, especially trained person to operate and require maintenance and are expensive.

7. Result and Discussion

7.1. Variation between Alkanity & Fluoride Samples

 $\label{eq:local_local_local} Initial \ Concentration \ of \ standard \ solution \ of \ Fluoride \ (mg/l) \\ = 10 \ mg/L$

Initial pH of standard solution of Fluoride = 6.0.

Table 2. Approximate Alum dose (mg/L) required to obtain acceptable quality (~1.0 mg/L) of drinking water from raw water at various alkalinity and fluoride levels**1 *To be treated after increasing the alkalinity with lime.

Test Water Fluoride (mg/L)	Test water alkalinity (ppm)							
	150	250	350	400	500	600	800	1000
2	165	220	265	305	340	405	470	520
3	205	300	335	375	505	520	585	765
4	*	400	405	460	560	600	690	935
5	*	*	510	595	690	715	885	1010
6	*	*	610	705	780	935	1065	1210
8	*	*	*	*	990	1120	1300	1430
10	*	*	*	*	*	*	1510	1690

7.2. Removal of Fluoride by Ferric Ammonium Sulphate Solution

Initial concentration of standard solution of Fluoride (mg/l) = 10 mg/LInitial pH of standard solution of Fluoride = 6.0

Initial pH of Ferric Ammonium Sulphate solution = 6.5

Table 3. Removal of Fluoride by 5% Ferric Ammonium Sulphate solution

Volume of standard solution of Fluoride (ml)	Volume of 5% solution of Ferric ammonium sulphate (ml)	Final Concentration of standard solution of Fluoride (mg/l)	Final pH of standard solution of Fluoride	Removal of Fluoride %
10	0.0	10	6.0	0 %
10	1.0	6.95	5.17	30.5 %
10	1.5	4.7	4.9	53 %
10	2.0	3.4	4.5	66 %
10	2.5	2.5	4.3	75 %
10	3.0	1.7	4.1	83 %
10	3.5	1.25	3.9	87.5 %
10	4.0	0.765	3.5	99.3 %

7.3. Removal of Fluoride by 5% Ferric Ammonium Sulphate Solution

Initial concentration of standard solution of Fluoride (mg/l) = 10 mg/L

Initial pH of standard solution of Fluoride = 6.0

Initial pH of Ferric Ammonium Sulphate solution = 6.5

Table 4. Removal	of Fluoride b	by 2% Ferric A	Ammonium Sul	phate solution

Volume of standard solution of Fluoride (ml)	Volume of Ferric ammonium sulphate (ml)	Final Concentration of standard solution of Fluoride (mg/l)	Final pH of standard solution of Fluoride	% Removal of Fluoride %
10	0.0	10	6.0	0 %
10	0.3	8.8	5.8	12 %
10	0.6	7.19	5.5	28.1 %
10	0.9	6.12	5.3	38.8 %
10	1.2	5.43	5.2	45.7 %
10	1.5	3.78	4.9	62.2 %
10	1.8	2.26	4.6	77.4 %
10	2.1	1.37	4.3	86.3 %
10	2.5	0.75	4.2	99.25 %

Table 5. Comparison between removals of fluoride by different concentration of solution of Ferric Ammonium Sulphate

Initial Concentration of standard solution of Fluoride (mg/l)	Volume of 5% solution of Ferric ammonium sulphate (ml)	Final Concentration of standard solution of Fluoride after adding 5 % FAS(mg/l)	Volume of 2% solution of Ferric ammonium sulphate (ml)	Final Concentration of standard solution of Fluoride after adding 2 % FAS(mg/l)
10	0.0	10	0.0	10
10	1.0	6.95	0.3	8.8
10	1.5	4.7	0.6	7.19
10	2.0	3.4	0.9	6.12
10	2.5	2.5	1.2	5.43
10	3.0	1.7	1.5	3.78
10	3.5	1.25	1.8	2.26
10	4.0	0.765	2.1	1.37
10	-	-	2.5	0.75

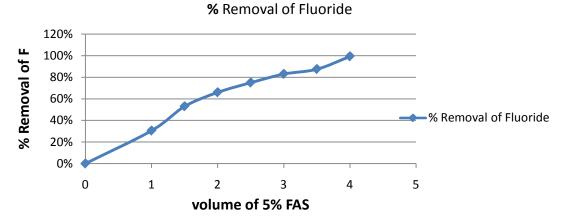


Figure 4. Graph between % removal v/s volume of 5% FAS

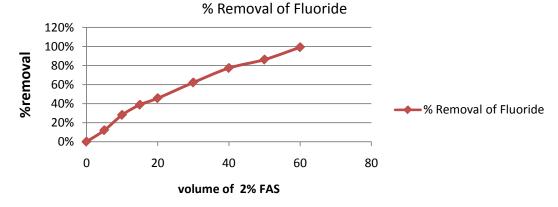


Figure 5. Graph between % removal v/s volume of 2% FAS

Based on the above results, it is observed that this process also results in high residual Al in output water ranging from 0.16ppm to 0.45ppm. Also, reactivation of

filter material is cumbersome and it can be done only with the help of trained persons generally not available in most of our villages. People with exposure and those with clinical and sub clinical symptoms, the only available measure as of today is eliminating the intake of high fluorides in drinking water and all other sources of fluoride in day to day life. No chemical till date is capable of extracting fluoride absorbed in the body.

8. Other Management Options

In addition to above de-fluoridation mechanism/devices, the following remedial measures are also suggested:

8.1. Dietary Changes

The basic diet during earlier days in the arid district was milk and milk products, which were taking care of the fluoride, affected mass population to greater extent. However, present modern time has changed the dietary habits. Therefore, dietary interventions are need of the hour. The mass population should be made aware to reduce as much as possible the fluoride intake through water and food. Practice consumption of diet rich in calcium, vitamin C (ascorbic acid), vitamin E and anti-oxidants. A properly designed nutritional regimen can beneficially interfere with the toxic effects of fluoride. Vitamin C, vitamin E and anti-oxidants, which are beneficial and are not very expensive, can be produced in rural areas without much investment. All these items have antagonistic effect; thereby play the prophylactic role in preventing fluorosis. Sources of various required nutrients are:

- Calcium: Milk, Curd, Yoghurt, Green leafy vegetables, Jaggery, Drumstick, Sesameseeds
- Vitamin C: Aaonla, Lemon, Orange, Tomato, Sprouted cereals/pulses and Dhania leaves
- Vitamin E: Vegetable oil, Cumin seeds, Nuts, White grain cereals, Green vegetables and Dried beans
- Anti-oxidants: Garlic, Ginger, Carrot, White onion, Papaya, Pumpkin and Green leafy vegetables. [21,22,23]

8.2. Rainwater Harvesting and Artificial Recharge to Ground Water

In areas having high concentration of fluoride, rainwater may be stored and preserved for drinking purposes only. Large scale artificial recharge to potential aquifers is another management option, which will minimize fluoride contents in ground water by dilution and flushing action. Surface and shallow ground water recharge techniques will cause evaporation losses, less effective due to deep groundwater levels and most importantly chemical reactions with soil and litho-formations of zone of aeration, thereby further enhancing fluoride concentration. Therefore, direct and deep recharge techniques like dug wells, tube wells, hand pumps, injection wells etc. are advisable. [24]

9. Result & Conclusion

The present study showed that the Ferric Ammonium Sulphate is an effective precipitating agent for the removal

of fluoride ions from groundwater of Nagaur district. The removal of fluoride ion depends on the concentration of Ferric Ammonium Sulphate solution. The removal of fluoride ion increases with increasing concentration and volume of Ferric Ammonium Sulphate Solution. The study indicated that the Ferric Ammonium Sulphate was found to be a favourable precipitating agent for the removal of fluoride ion,Improvement in dietary habits, rainwater harvesting and artificial recharge to ground water etc. are still tested fluoride management options in the study area.

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