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Defluoridation of Bore Well Water Using Combustion Derived Nanocrystalline Magnesium Oxide

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Nanocrystalline MgO powder has been prepared through combustion route using magnesium nitrate as oxidizer and glycine as fuel; the combustion derived MgO powder has been characterized using XRD, SEM, surface area and porosity measurements. The powder XRD pattern confirms the crystallinity and phase purity of the as-made powder. The scanning electron microscope (SEM) results reveal that the combustion derived powder is an agglomeration of fine particles. The particle size of the powder is found to be in the range of 12-23 nm with a large surface area of 107 m².g⁻¹. The as-made MgO is used for defluoridation of ground water samples with stirring time (5-60 min), adsorbent quantity (0.05-0.3 g.L⁻¹) and pH (5-9) as experimental parameters. It is found that 92% and 97% of fluoride, respectively, could be removed using 0.15 g of MgO from 10 ppm of synthetic fluoride solution and standard sodium fluoride solution. In this technique 90% minimization of sludge could be achieved. It is also observed that the regeneration of adsorbent with NaOH (15 mL of 0.1 N NaOH for 0.5 g of sludge) solution gives better results than HCl (15 mL of 0.1 N HCl for 0.5 g of sludge) solution.

[Keywords : Combustion synthesis, Nanocrystalline MgO, Bore well water, Defluoridation]

Introduction

It is well-known that very small quantities of fluoride ingestion is necessary for bone and teeth developments, but excessive ingestion causes a disease known as fluorosis. The WHO standards¹ and BIS : 10500 1983² permit only 1.5 mg.L⁻¹ as a safe limit of fluoride in drinking water for human consumption. Fluorosis continues to be an endemic problem and more and more areas are being discovered regularly that are affected by fluorosis in different parts of the world. Excess fluoride in ground water has been found in Pakistan, Bangladesh, Argentina, United States of America, Morocco, Middle East countries, Japan, South African countries, New Zealand, Thailand, China, Sri Lanka, Spain, Holland, Italy and Mexico.^{3,4} The problem of fluorosis has thus reached an alarming stage. In India, as many as 200 districts spread across 17 states are suffering from high fluoride concentration. A population of some 25 million has been affected and approximately 66 million people are described as population at risk.⁵ The most affected areas in India as reported³ are:

(a) 50-100% districts in Andhra Pradesh, Tamil Nadu, Uttar Pradesh, Gujarat and Rajasthan.

(b) 30-50% districts in Bihar, Haryana, Karnataka, Maharashtra, Madhya Pradesh, Punjab, Orissa and West Bengal.

(c) < 30% districts in Jammu and Kashmir, Delhi and Kerala.

Fluoride exists fairly abundantly in the earth's crust and can enter ground water by natural processes like weathering and leaching of bedrock. The minerals / rocks rich in fluoride are fluorspar CaF₂ (sedimentary rock, limestones and sandstones), cryolite Na₃AlF₆ (igneous, granite) and fluorapatite Ca₃(PO)₂Ca(FCI)₂. Water is a major source of

fluoride intake. High profile of fluoride in ground water is observed in 4.6% of geographical area (8900 Km²) of Karnataka. The incidence of very high levels of fluoride contamination in the districts has occurred in the eastern and southern belts covering the districts of Gulbarga, Raichur, Bellary, Chitradurga, Tumkur and Kolar and are scattered in the rest of Karnataka. The reported concentration range of fluoride in ground water is 1.55 -7.4 ppm.⁴

Defluoridation of water has been carried out using various methods like coagulation and precipitation process⁶ (named as Nalgonda technique), by the use of locally derived sample of silty clay,⁷ natural materials,⁸ soil sorbent⁹ and oxide minerals.¹⁰ The removal of excessive fluoride (F⁻) from drinking water was also attempted using ion exchange / adsorption process in which commercially available metal oxides like activated alumina, magnesia and other materials were used.^{3,11} In all these cases the adsorbent quantity and fluoride removal capacity have been reported (Table I).

The development of novel methods for dehalogenation of wastes has become a very urgent task due to their high toxicity and the significant ecological hazard. Nanocrystalline alkaline earth metal oxides attract significant attention as effective chemisorbents for toxic gases, HCl, chlorinated and phosphorus containing compounds.¹² Destructive sorption takes place not only on the surface of oxide materials but also in their bulk. Although many metal oxides and metal oxide mixtures prepared and activated in the proper way may be capable of acting as chemical adsorbents, MgO serves as a good model since it possesses a simple (NaCl) crystal structure and can be prepared with widely ranging surface areas.¹³ It must be noted that the efficiency of the destructive sorption increases with a decrease in the size of the MgO crystallites. MgO works

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Table I : Different reported defluoridation techniques.

Sl No.	Name of the material	Adsorbent quantity (g.L ⁻¹)	Initial F ⁻ content (mg.L ⁻¹)	Final F ⁻ content (mg.L ⁻¹)	Defluoridation capacity (% or g.Kg ⁻¹)	Merits/Demerits	Reference
1.	Amended / silty clay	100	25	4.9	80	Sludge generation is high, removal time is 48 h, pH (7-9) dependent.	7
2.	Natural materials : Column study						
	(a) Brick powder	25	10	3.9	39	Removal time 2 h, regeneration is difficult.	8
	(b) Charcoal	25	10	5.0	50		
3.	Soil sorbent	10	10	7	30	Removal time is 48 h.	9
4.	Oxide minerals	30	20	12	60	Sludge generation is high and pH (5-7) dependent.	10
5.	Commercial materials:						
	Activated alumina	5	—	—	0.8 g.Kg ⁻¹	Sludge generation is high and pH dependent. The residual aluminium (2.1-6.8 ppm) is reported. Limit is 0.2 ppm, taste and turbidity are not acceptable.	3
	Activated carbon	—	—	—	0.32 g.Kg ⁻¹	Sludge generation is high and pH (5-7) dependent.	11
	Florex	—	—	—	0.6 g.Kg ⁻¹	It is a mixture of tricalcium phosphate and hydroxy-apatite. High attritional losses.	11
	Lime	—	—	—	1.5 g.Kg ⁻¹	Sludge generation is high and pH (10.5) dependent. Recarbonation required.	11
	Serpentine	80	—	—	69	Sludge generation is high and pH (8-9) dependent.	11
	Precipitation / Nalgonda Technique	Very high dosage	—	—	Limited to <10 ppm of F ⁻	Very large dosages of alkali, chlorine, Al ₂ (SO ₄) ₃ / AlCl ₃ are required. pH (10.5) and residual aluminium (2.1-6.8 ppm) are reported. Limit is 0.2 ppm, taste and turbidity are not acceptable.	11
	MgO coarse material	1.5	—	—	89	Sludge generation is high, pH raises beyond 10, contact period 3 h, acid requirement 300 mg.L ⁻¹ as CaCO ₃ .	11
6.	Combustion derived nanocrystalline MgO	0.15	10	9.2	92	92% removal from synthetic water, 90% of sludge minimization, contact time only 10 min, pH independent.	Present study

most efficiently in the destructive sorption reaction when its particle size is on a nanometer scale. The high efficiency of the nanoparticulate oxides is caused not only by their high surface area but also by the high concentration of low-coordinated sites and structural defects on their surface. Depending on preparation methods MgO exhibits quite different reactivity towards adsorbed chemicals.¹⁴ Therefore, high surface area material, having the most defect sites per unit area, should be of interest as destructive adsorbent.¹⁵ MgO is usually synthesized through hydrothermal,¹⁶ sol-gel,^{17, 18} and thermal decomposition of magnesium hydroxide or carbonate.¹⁹ In this paper we explore the use of the novel combustion technique²⁰ for the synthesis of nanocrystalline MgO and its use as a defluoridation agent (fluoride adsorbent).

Experimental

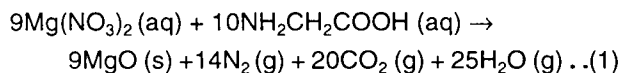
Chemicals

Magnesium nitrate (99%, AR Grade Fischer), glycine (99%, Purified E. Merck), sodium fluoride (99.5%, AR Grade SD fine chemicals), hydrochloric acid (35%, GR Grade, E. Merck), sulphuric acid (98%, Pure E. Merck), sodium hydroxide (Purified, E. Merck) and double distilled water for solution preparation were used in the present investigation.

Synthesis of MgO

MgO of nanodimensions was prepared by solution combustion process.²⁰ An aqueous solution containing stoichiometric amount of magnesium nitrate as oxidizer and glycine as fuel was taken in a pyrex dish (corresponds to an equivalent stoichiometric ratio of fuel to oxidizer $\psi = 1.11$).

The pyrex dish was introduced into a muffle furnace (PSM Instruments, Bangalore) maintained at 400°C. The redox mixture boiled and formed a white residue. Then burning appeared at one end and propagated throughout the mass within a minute. A fine powder with black colour was obtained. It turned to white MgO after allowing it at the same temperature in the muffle furnace for 30 min. The theoretical equation assuming complete combustion of the redox mixture used for the synthesis of MgO may be written as:



Characterization of MgO

The X-ray powder diffraction pattern of the as-prepared MgO was obtained on a Phillips X-pert PRO diffractometer operating with Cu K α ($\lambda = 1.54056 \text{ \AA}$). The surface morphology and distribution of particles of the as-formed powder were examined using JEOL (JSM-840 A) scanning electron microscope (SEM). The surface area and porosity of the as-prepared MgO were determined by QuantaChrome Corporation NOVA 1000 Gas Sorption Analyzer.

Estimation of Fluoride from Bore Well Water

Reagents

A stock solution of fluoride (1000 ppm) was prepared by dissolving 2.2111 g of anhydrous sodium fluoride, NaF, in 1000 mL double distilled water. Standard fluoride solutions of 5 and 10 ppm were prepared by successive dilution of the stock solution and used for the calibration of Fluoride ion meter. Solutions of HCl (0.1 and 0.2 N), H₂SO₄ (0.02 N) and NaOH (0.1 N) were prepared by dissolving the required quantity of the respective chemicals in double distilled water and standardized.²¹ Synthetic water,²² containing 10 ppm of fluoride was prepared by spiking 10 mL of stock fluoride solution into 1000 mL of tap water.

Materials and Equipment

For the estimation of fluoride present in bore well water, the analyses were carried out for various physico-chemical parameters according to APHA 1998 standard methods²³ and the results are shown in Table II. Fluoride content was measured using Fluoride ion meter (Orion, Model 290A). The pH, conductivity and turbidity were measured using pH-meter (Orion, Model 290A), conductivity meter (Systronics) and Nephelometer (Systronics), respectively. The estimation of sodium and potassium by flame photometric method (Systronics), nitrate by UV-screening method (UV-Visible Spectrometer, Lambda 20, Perkin Elmer) and sulphate by turbidimetric method (Nephelometer, Systronics) were carried out. Estimations were made of total dissolved and suspended solids by gravimetric method, total hardness and calcium hardness by EDTA titration, total alkalinity, carbonate, and bicarbonate by acid titration method, chloride by argentometric method and magnesium by calculation.

Bore Well Water Sampling

The bore wells situated in Raichur, Davanagere, Chitradurga, Pavagada (Tumkur dist) and Kolar were

identified as sources of samples from fluoride prone areas. Two litres of samples each from five bore wells were collected in cleaned plastic containers. These samples were preserved in an ice box and refrigerated as per the standard procedure.²³

Procedure for Defluoridation

A fluoride ion meter (calibrated using standard 5 and 10 ppm of fluoride solution in distilled water) was used for estimation of the fluoride content present in synthetic as well as bore well water samples. The defluoridation experiments were performed with 10 ppm of fluoride solution (synthetic water), utilizing batch shaking process on magnetic stirrer. In each experiment, 100 mL of synthetic water containing 10 ppm of fluoride was taken in 250 mL beaker and to this, a known quantity (0.15 g) of adsorbent (as-made MgO) was added. In a batch of experiments, the optimum values of variables like pH (the process is independent of pH), stirring time (10 min), and sedimentation time (20 min) were maintained. The fluoride content left out in water after the treatment with MgO was measured. The fluoride adsorbed MgO settled at the bottom of the beaker was separated by decantation; this is termed "sludge". This standard procedure was extended for the defluoridation of bore well water samples with the same experimental parameters. The results obtained from the experiments are reported in Table II.

Regeneration of Sludge

The sludge after separation was dried in air and 0.5 g of it was washed thrice with 15 mL portion of 0.1 N NaOH followed by 45 mL double distilled water. The washings were separated by filtration and the filtrate was made up to 100 mL with double distilled water. The fluoride content was measured using Fluoride ion meter and it was found to be 4.5 ppm. The same procedure was repeated for the regeneration of sludge with 15 mL portion of 0.1 N HCl instead of NaOH and the fluoride content was estimated. With acid washing, it was found that only 2 ppm of fluoride could be removed from the sludge. From this it is possible to conclude that the sludge can be regenerated completely from fluoride with NaOH treatment.

Results and Discussion

A powder X-ray diffraction pattern of combustion derived MgO powder is shown in Fig. 1. The as-formed MgO powder exhibits peaks corresponding to the impurity-free nanocrystalline cubic phase which is in good agreement with the literature data.²⁴ The particle size of MgO powder was estimated from the full width at half-maximum (FWHM) of the diffraction peaks of the powder from the line broadening Scherrer's formula.²⁵ The average particle size of the powder was in the range of 12-23 nm.

The SEM image (Fig. 2) shows that the as-formed powder is agglomerated and highly porous having pore diameters in the range of 1-11 nm. Representative nitrogen adsorption / desorption isotherms and the corresponding pore size distribution curve (inset) of the obtained MgO product are shown in Fig. 3. The BET surface area²⁶ calculated from

Table II : Analysis report of bore well water samples collected from different parts of Karnataka

Parameters	Units	Name of sampling points					Name of sampling points				
		Analysis of water samples as such (Untreated)					Analysis of water samples after defluoridation (Treated with MgO)				
		1	2	3	4	5	1*	2*	3*	4*	5*
pH	—	8.5	8.6	8.4	7.5	8.3	10.8	10.1	10	9.8	10.1
Conductivity	ms.cm ⁻¹	4	2.5	0.8	2.1	0.6	4.1	2.6	0.9	2.2	0.7
Turbidity	NTU	ND	ND	ND	ND	ND	0.6	1	2.6	0.2	15
Total dissolved solids	mg.L ⁻¹	2680	1675	536	1407	402	2747	1742	603	1474	469
Sodium	mg.L ⁻¹	525	415	116	129	80	525	410	112	128	82
Potassium	mg.L ⁻¹	3	6	3	3	3	3	6	3	3	3
Total hardness (as CaCO ₃)	mg.L ⁻¹	758	380	108	613.6	128	772	390	120	633	124
Calcium (as Ca)	mg.L ⁻¹	135	126	25.6	418.6	70	128	76	24.8	402.4	68
Magnesium (as Mg)	mg.L ⁻¹	623	254	82.4	195	58	644	314	95.2	230.6	56
Total Alkalinity	mg.L ⁻¹	280	516	280	364	200	260	676	380	292	272
Carbonates	mg.L ⁻¹	25.6	136	67.2	8	16	200	664	280	184	142
Bicarbonates	mg.L ⁻¹	254.4	380	212.8	356	184	60	0	100	108	130
Chlorides (as Cl)	mg.L ⁻¹	1012	424	76	384	84	1008	412	76	380	80
Nitrates (as NO ₃)	mg.L ⁻¹	3.8	19	4.5	5	1.22	3	10.5	4.13	4.7	ND
Sulphates (as SO ₄)	mg.L ⁻¹	83.5	69.6	27.6	71.1	24.7	84	67.68	27.6	70.64	23
Fluoride (as F)	mg.L ⁻¹	1.8	2.32	1.65	2.1	2	0.9	0.75	0.8	0.65	0.5

1. Bore well water samples (untreated) : (1) Raichur, (2) Davanagere, (3) Chitradurga, (4) Pavagada (town in Tumkur dist) and (5) Kolar

2. Bore well water samples (treated) : 1*, 2*, 3*, 4* and 5*; 3. NTU : Nephelometric turbidity units; 4. ND : Not Detectable;

5. Highlighted results are exceeding the standard limits.

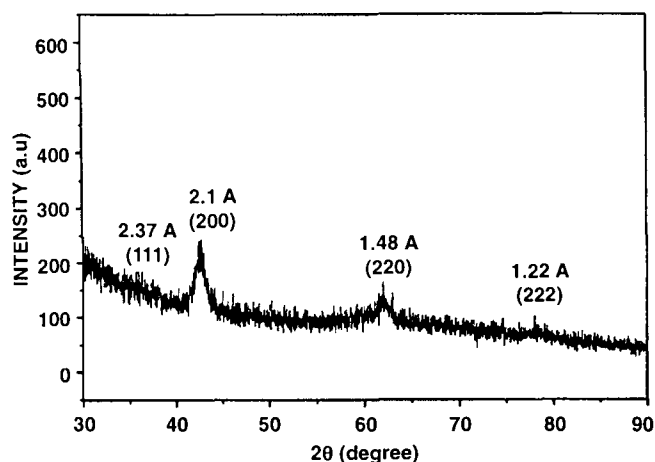


Fig. 1- Powder XRD pattern of as-prepared nanocrystalline MgO.

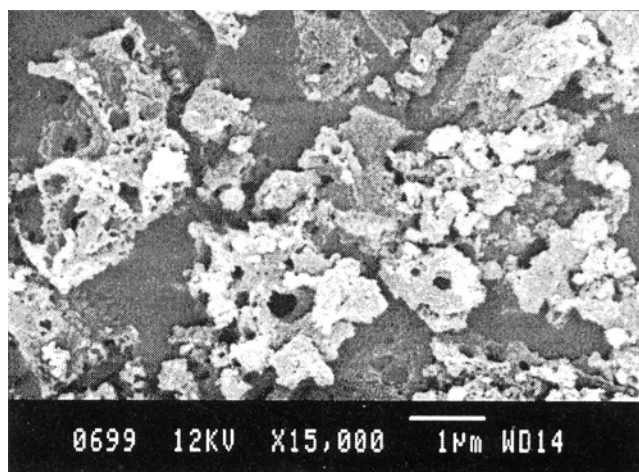


Fig. 2 – SEM image of as-made MgO powder.

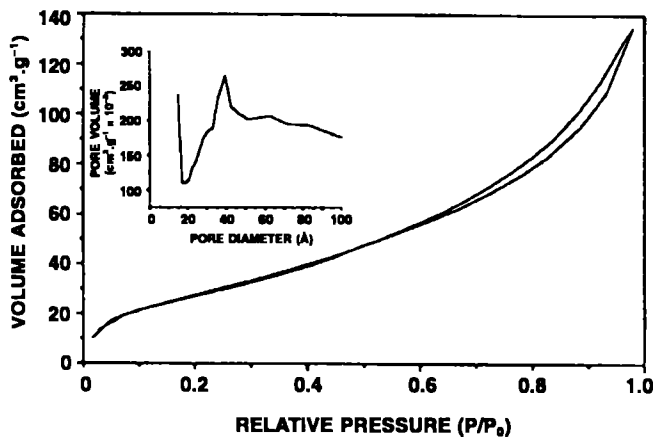


Fig. 3 – Nitrogen adsorption-desorption isotherms for the obtained MgO. Inset : corresponding pore size distribution curve determined from the N₂-desorption isotherm.

isotherm of adsorption / desorption curve is 107.24 m².g⁻¹. The average pore diameter obtained from desorption is found to be 7.8 nm and this value is in the range of pore diameter observed in SEM images. The large surface area of the sample might be due to the porous nature of the powder and uniform distribution of nanosized particles which are agglomerated as observed in SEM image.

Effect of Adsorbent Quantity

Different quantities (0.05-0.3 g) of MgO as adsorbent were taken separately in 100 mL of 10 ppm fluoride solutions (synthetic and standard) and the batch was kept shaking for 1 h. Thereafter the analysis of residual fluoride in the treated water was performed and a relationship between fluoride removal capacity (percentage) and adsorbent quantity was established, as shown in Figs. 4 and 5. From the trend it can be seen that a maximum level of fluoride removal (in synthetic water 90-92% and in standard sodium fluoride solution 97%) occurs in the presence of 0.1-0.15 g of as-prepared MgO. After this, the removal

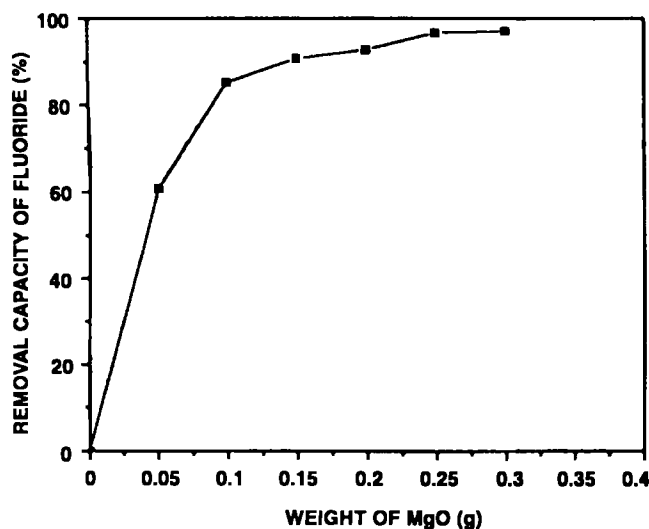


Fig. 4 – Fluoride removal capacity versus amount of adsorbent (using 10 ppm of synthetic water).

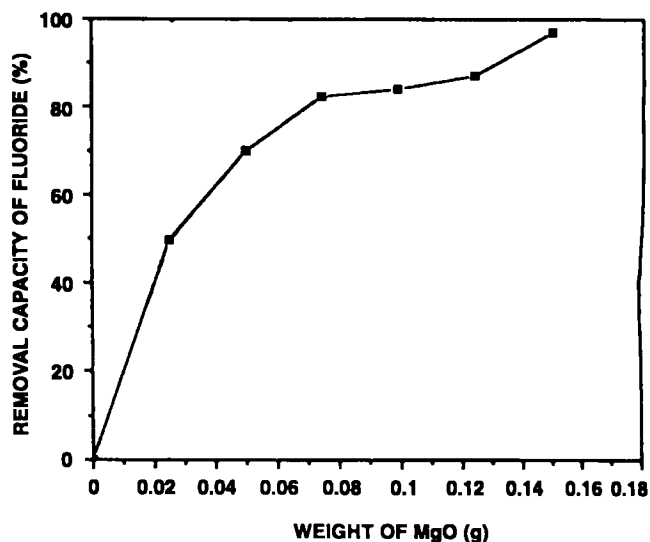


Fig. 5 – Fluoride removal capacity versus amount of adsorbent (using 10 ppm of standard sodium fluoride solution).

rate becomes almost constant and therefore, 0.1 g can be considered as the optimum dose.

Effect of Stirring Time

The effect of contact time on fluoride removal rate of the adsorbent was determined by using an optimum dosage of 0.1 g MgO powder (see above). The batch experiments for 10 ppm of fluoride were conducted at different intervals of stirring time in 100 mL synthetic solutions. After every 10 min of stirring, the residual fluoride in treated water was analysed. The fluoride removal gradually increased with time and attained a maximum value after 10 min shaking (Fig. 6) and this duration could be considered as the optimum time for maximum level of fluoride removal. This stirring time was 6 times less than the methods^{7, 8} reported so far.

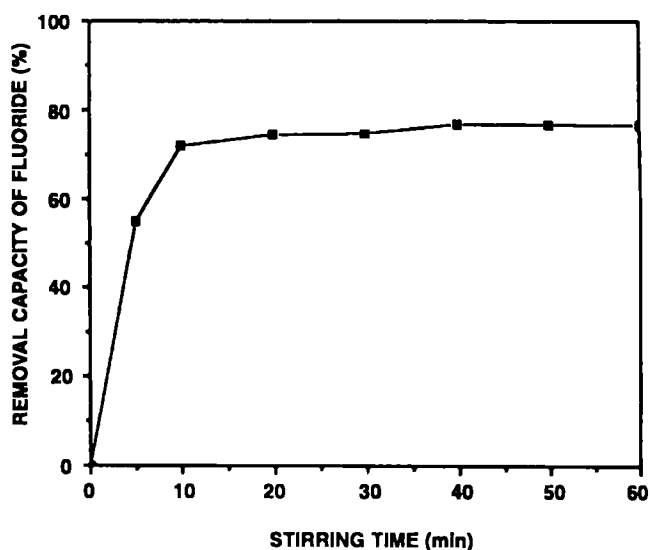


Fig. 6 – Fluoride removal capacity versus stirring time (using 10 ppm of synthetic water).

Effect of pH on Fluoride Removal

The effect of pH (5-9) on fluoride removal was examined using an optimum dosage (0.1 g) of adsorbent and stirring time (10 min). The results are shown in Fig. 7. It is evident from the curve that the fluoride removal is independent of pH.

Estimation of Fluoride from Bore Well Water

The physico-chemical parameters of untreated and treated water samples are listed in Table II. The results show that, out of the five bore wells none are fit for drinking purposes as per the Indian drinking water standard.² The pH (sample 2), chloride content (sample 1), total hardness (samples 1 and 4) and fluoride level (sample 1-5) are beyond the limits (Table III) of Indian drinking water standards.² The results of defluoridation of bore well water samples show that the fluoride level decreased by 50 to 75% in all the water samples after treatment with 0.1 g of MgO. It is also interesting to observe that the nitrate content along with fluoride in bore well water samples decreased by 6 to 44%. However, there was an increase in pH²⁷ and hardness (Table II) after the treatment, and increase of these is due to the partial dissolution of adsorbent MgO in water followed by the formation of MgCO₃ which was confirmed by the experiment.

Maintenance of pH of treated water in the neutral range is important. Hence, it is necessary to bring down the pH to 7 by dilute mineral acids. The experiment was conducted to determine the neutralization point of treated water with

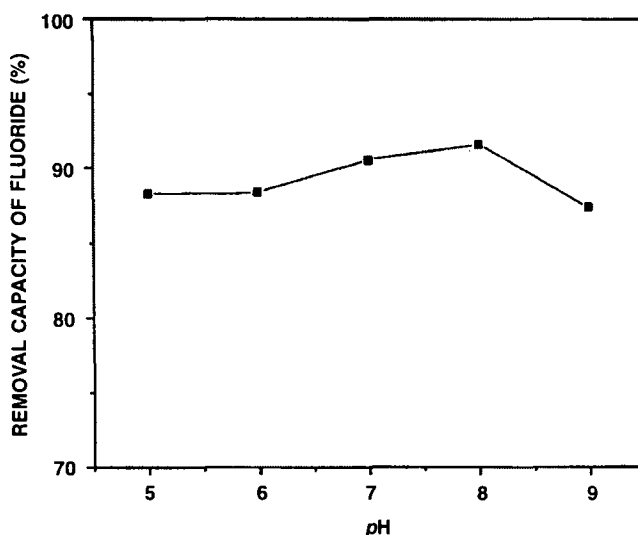


Fig. 7 – Fluoride removal capacity versus pH (using 10 ppm of synthetic water).

0.02 N H₂SO₄ and 0.02 N HCl. It was found that about 88 mL of H₂SO₄ or 108 mL of HCl, respectively, were required to neutralize 1 L of treated water. From these experiments, it was noticed that 153 mg.L⁻¹ of sulphate or 74.5 mg.L⁻¹ of chloride were increased for H₂SO₄ and HCl, respectively in the treated water. So, the contents of chloride and sulphate in all the samples except sample 1 (chloride content) are within the limits of Indian standards of water quality even after the neutralization process with acids.

Table III : Quality of synthetic water

Sl No.	Parameter	Units	IS-10500-1983		Tap water as such	Synthetic water
			Desirable limit	Permissible limit		
1.	pH	—	6.5-8.5	6.5-8.5	8.1	8.1
2.	Conductivity	ms.cm ⁻¹	—	—	0.39	0.39
3.	Turbidity	NTU	5	10	1	1
4.	Total dissolved solids	mg.L ⁻¹	500	2000	268	268
5.	Sodium	mg.L ⁻¹	—	—	36	36
6.	Potassium	mg.L ⁻¹	—	—	ND	ND
7.	Total Hardness (as CaCO ₃)	mg.L ⁻¹	300	600	158	158
8.	Calcium (as Ca)	mg.L ⁻¹	75	200	19.6	19.6
9.	Magnesium (as Mg)	mg.L ⁻¹	30	100	25.92	25.92
10.	Total Alkalinity	mg.L ⁻¹	200	600	182	182
11.	Carbonates	mg.L ⁻¹	—	—	36	36
12.	Bicarbonates	mg.L ⁻¹	—	—	146	146
13.	Chlorides (as Cl)	mg.L ⁻¹	250	1000	18	18
14.	Nitrates (as NO ₃)	mg.L ⁻¹	—	50	0.8	0.8
15.	Sulphates (as SO ₄)	mg.L ⁻¹	200	400	12	12
16.	Fluoride (as F)	mg.L ⁻¹	1	1.5	0.3	10.2

NTU : Nephelometric turbidity units; ND : Not Detectable

Conclusions

The present study clearly highlights the significant potential of nanodimensional MgO for fluoride removal in the bore well water. The MgO obtained through combustion synthesis is used as adsorbent in the present investigation which is eco-friendly and non-toxic. The fluoride removal capacity of MgO is very high compared to that shown in the reported methods. In this process, waste (sludge) generation can be avoided up to 90% because of the nanosize and large surface area of MgO used as adsorbent when compared with the commercially available coarse materials. The generated sludge is a value added product and can be regenerated and the same can also be used in the pharmaceutical and chemical industries. This simple and novel method can be easily implemented at household level in community water supply schemes in rural India.

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