ISSN : 0025-0422

JOURNAL OF THE MAHARAJA SAYAJIRAO UNIVERSITY OF BARODA



सत्यं शिवं सुन्दरम् Estd. 1949 Accredited Grade 'A' by NAAC

VOL. 53 (2019) NO. 1 (Science & Technology) Vadodara 2020

JOURNAL OF THE MAHARAJA SAYAJIRAO UNIVERSITY OF BARODA

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VOL. 53 (2019) NO.1 (SCIENCE & TECHNOLOGY) VADODARA 2020 ISSN : 0025-0422

Printed by Shri Jatin H. Somani, Manager, The Maharaja Sayajirao University of Baroda Press (Sadhana Press), Near Palace Gate, Palace Road, Vadodara, and Edited by **Prof. C. N. Murthy,** Editor (Science & Technology) at the Maharaja Sayajirao University of Baroda, Vadodara - 390 002, (India), February 2020.

CONTENTS

Sr.	No	Торіс	Page No.
1.		RAPID INTEGRATED WATER QUALITY EVALUATION OF THOL WETLAND USING BENTHIC MACROINVERTEBRATES	1-13
		Bhadrecha Mahendra H., Mankodi Pradeep C.	
2.		BIOSYNTHESIS OF NATURAL POLYMER BASED NANO CELLULOSE FIBRE : TODAY'S TREND	15-26
		S. B. Chaudhari, Tasnim N. Shaikh, B. H. Patel, Pooja Dadhaniya	
3		COMPARATIVE STUDY OF SHAFT SURDORTED AND	27-35
5.		FRAME SUPPORTED INTZE WATER TANK USING LIMIT STATE METHOD	27-35
		D. R. Panchal, B. A. Shah, C. B. Maradiya	
Л		REVENW ON ERACTURE PROCESS ZONE OF CONCRETE	37-48
4.		Deeprai Patadiva. Krishna Nair	57-40

RAPID INTEGRATED WATER QUALITY EVALUATION OF THOL WETLAND USING BENTHIC MACROINVERTEBRATES

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Abstract

An integrated water quality of Thol Wetland, in Mehsana District of Gujarat, India was assessed by studying physico-chemical parameters as well as Benthic Macroinvertebrates. Physico-chemical parameters analysed were pH, Colour, Turbidity, Conductivity, Total Solids, Total Dissolved Solids, Total Suspended Solids, Ammonical Nitrogen, Nitrate, Phosphate, Alkalinity, Total hardness, Sodium, Potassium, Calcium, Magnesium, % Sodium, Sodium Absorption Ratio, Dissolved Oxygen, Chemical Oxygen Demand, Biochemical Oxygen Demand, Chloride and Sulphate. The biological parameters calculated were BMWP (Bio Monitoring Working Party) Score or Saprobic Score and Sequential Comparison Index or Diversity Score. In total 11 families were encountered. The findings indicate that the water quality of Thol Wetland is 'Moderately Polluted' with comparatively high organic content mainly of natural origin and suitable for irrigation purpose.

Key words: Benthic Macroinvertebrates, BMWP Score, Diversity Score, Biological Water Quality Criteria,

Introduction

Wetlands are highly productive ecosystems that provide food and habitat to aquatic and amphibious organisms. It also provides multifarious ecosystem services that contribute to sustenance of human life on earth [1]. It is thus necessary to periodically monitor the ecological health of wetlands especially the water quality. Mostly, water quality is assessed by studying various physico-chemical parameters such as pH, Biochemical Oxygen Demand, Chemical Oxygen Demand, Dissolved Oxygen etc. However, for an overall assessment of an ecosystem, physico-chemical studies have to be supplemented with biological assessments [2,3]. Central Pollution Control Board, Delhi has also established the role of biological parameters particularly Biomonitoring using Benthic Macroinvertebrates [4,5,6]. They are indicators of water quality as these groups exhibit varying sensitivity to different pollution levels. This study is an attempt to integrate Bio-monitoring and Physicochemical monitoring for effective assessment of overall water quality of Thol Wetland, Gujarat.

Study Area and General Habitat

Thol Bird Sanctuary is located at about 22 km away from Kadi town of Mehsana district of Gujarat State, India. It has a total area of 699 sq. km. and the periphery is 5.62 km [1, 7]. There is a continuous earthen bund on its western, southern and eastern periphery, which helps in collection of water that flows into it during the monsoon from the catchment area (Fig.1). Thol water body also supports a canal based irrigation system. It remains covered with water in the rainy season. During winter it begins to dry and by summer the wetland is separated into water bodies of varying in size, the biggest being towards the western side. Physico-chemical Study was carried out at sampling location no.1 (23°07' 53.9" N, 72° 24' 46.7"E) and Location no.2 (23° 08' 01.8" N, 72° 24' 01.3" E). Location 1 is characterized with almost no human intervention, profuse growth of water lilies in surrounding waters and where as location 2 is characterized by comparatively more human intervention with visitors camping and bird watching. Cattle wading, water pumping for irrigation was found in some spots of the Wetland. The surrounding land use mainly falls into arable, grazing and forest type. There was no discharge of water outside the wetland during the field visit.

Materials and Methods

Ample care was taken to ensure that all indicator families of Benthic Macroinvertebrates present are actually encountered. This was accomplished by sampling all different (micro) habitats in a sizeable stretch of the water body [6,8]. At each location; the water sampling point (GPS location) was fixed as reference point and keeping 250 m stretch on either side of it of the wetland bank; a total of 500 m X 2 m width of wetland stretch was covered for Benthic Macroinvertebrate collection. This 500 m stretch was divided into 5 sub stretches of 100 m length and in each sub stretch, Benthic Macroinvertebrate collection was carried out.

All possible microhabitats such as aquatic vegetation on edges of the wetland banks, algae, pebbles, wetland bed, detritus, submerged and floating vegetations etc. were explored for collection of Benthic Macroinvertebrates by using hand net, sieve and hand picking with forceps [5,6,8] (Fig.2). Collection procedure was repeated five times in each sub stretch. Five grab samples of silt was picked from

the wetland bed by the plastic scoop/shovel and washed by wetland water in a sieve with mesh size 0.6 mm. The animals were then picked by forceps from the sieve and transferred into the tray. Net was also moved all along the edges of grass/emergent aquatic vegetation all along the wetland bank and the animals were collected and transferred into the tray with forceps. The water plants/floating plants present in the sampling area was uprooted and washed directly into the net or into the white tray so as to detach the animals. All animals thus collected were preserved in 4% formalin for further identification in laboratory.

Grab water samples were collected from both the locations for the retrieval of physico-chemical parameters [9] in the lab. Temperature was measured at the site. Dissolved Oxygen and COD were preserved at the sites itself using Winkler's reagent and Sulphuric acid respectively. The samples were then brought to laboratory for further analysis. Benthic Macroinvertebrates were identified using 'Appendix: 6 Taxonomic Key for Biological Water Quality Evaluation of the Manual on Integrated Water Quality Evaluation' [3]. The abundance of each animal observed during sampling was also noted down.

The physico-chemical parameters of water samples were assessed as per standard Methods for Examination of Water and Waste Water (APHA, 2005) [10]. Physicochemical parameters retrieved were pH, Colour, Turbidity, Conductivity, Total Solids, Total dissolved Solids, Total Suspended Solids, Ammonical Nitrogen, Nitrate (NO₃-N), Phosphate, Alkalinity - as CaCO₃, Total Hardness as CaCO₃, Sodium, Potassium, Calcium, Magnesium, % Sodium, Sodium Absorption Ratio (SAR), Dissolved Oxygen, Chemical Oxygen Demand, Biochemical Oxygen Demand (3 days 27°C), Chloride as Cl⁻ and Sulphate. The biological parameters using Benthic Macroinvertebrates analyzed were BMWP (Bio Monitoring Working Party) Score or Saprobic Score and Sequential Comparison Index or Diversity Score [4,5,6].

Diversity Score (Sequential Comparison): For calculation of Diversity Score, Benthic Macroinvertebrate specimens were randomly dispersed on a plastic tray marked with a grid of 1 inch x 1 inch (Fig 3) to facilitate the sequential comparision.

A sequential comparison of organisms in each square of the gridded tray was carried out from left to right direction and starting from the uppermost extreme left grid and ending at lowermost extreme right grid. While comparing, the organism is assigned a mark (run) of 1 if it is different from the just previous organism and a mark of 0 if it is same. The Diversity Score is calculated as:

Diversity Score = Number of Runs ÷ Total Number of Organisms.

Biological Monitoring Working Party (BMWP) Score: A quantitative inventory up to 'family' level of taxonomic precision was carried out for Benthic Macroinvertebrates collected from different microhabitats of the study area (Fig 4) by using BMWP score card designed by Central Pollution Control Board. All possible families having saprobic indicator value are classified on a score scale of 1 to 10. The families which are most sensitive to pollution are on the top of the list and are getting a score of 10 while the most pollution tolerant families are getting a score of 1 and 2. The other intermediately sensitive families are placed in between the scoring scale of 10 to 1. The saprobic scores of all the families were registered based on BMWP score. It was then averaged to produce Total BMWP Score. Abundance Scale of Families, A = Single (1 Individual), B = Scarce(2-10 Individuals), C = Common (10-50 Individuals), D = Abundant (50-100Individuals), E = Excessive (more than 100 individuals) is also noted based onfield observation.

Results and Discussions

Biological Assessments: Total number of organisms collected at Location no. 1 and Location no. 2 were 201 and 163 respectively. Therefore the Diversity Score at Location no. 1 and Location no. 2 are found to be 105/201 = 0.52 and 81/163 = 0.49 respectively. At both Locations, 9 different families falling across 5 different taxonomic orders were found. However, in all 11 families falling across 5 taxonomic orders were encountered (Table 1 and Table 2). The number of families found in each taxonomic order is depicted in the following graph (Fig.3).

The BMWP Score was found to be 5.33 and 4.77 at location 1 and 2 respectively and the Diversity Score is found to be 0.52 and 0.49 at location 1 and 2 respectively. These values when compared with the BWQC (Biological Water Quality Criteria) developed by Central Pollution Control Board [3,4,5] (Table 3), collectively indicate that the water quality of Thol wetland at both the locations during the study is 'Moderately Polluted'.

Physico-chemical assessments : The water samples were analyzed for pH, Colour, Conductivity, Total Solids, Total Suspended Solids, Total Dissolved Solids, Chlorides, Total Hardness, Calcium Hardness, Magnesium Hardness, Alkalinity, Turbidity, Ammoniacal Nitrogen, Chemical Oxygen Demand, Biochemical Oxygen Demand, Dissolved Oxygen, Sulphates and Nitrates. The values of physico-chemical parameters monitored are presented in Table 4.

The obtained average values of physico-chemical parameters are compared with IS:10500 Drinking Water Specifications [11,12,13] and it is evident that the water quality is within the permissible limits. These obtained values are also within the usual range prescribed in FAO guidelines for irrigation water [14]. However, the values of Chemical Oxygen Demand (COD) and Biochemical Oxygen Demand (BOD) are found to be comparatively high indicating accumulation of organic matter in wetland water (Table 4). This is mainly imparted by organic detritus in the form of dried leaves, twigs, flowers etc. falling from the surrounding trees and shrubs into the wetland water. The water column also had a greenish hue due to phytoplankton growth indicating organic content.

Conclusions

The Integrated water quality of Thol wetland is observed to be 'Moderately Polluted' owing to comparatively high organic content. The results of Physico-chemical analysis are in consonance with the Biological Water Quality Criteria developed by Central Pollution Control Board. Further, the use of Biomonitoring for water quality assessment using Benthic Macroinvertebrates can be used as a complementary method along with the regular physico-chemical analysis for comprehensive water quality monitoring and thus turning out to be an effective tool for water quality management.

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Fable 1. Calculation of BMWP Score	(Saprobic Score) for Loca	ation no. 1
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TAXONOMICAL GROUP	TAXONOMICAL FAMILIES	MARK ENCOUNTERED FAMILIES WITH ABUNDANCYAS A, B, C, D, E	TOTAL FAMILIES/ SPECIES ENCOUNTERED	BMWP SCORE	MULTI- PLIED SCORE
Odonata	Lestidae	В			
	Gomphidae C				
TOTAL FAMILI SCORE	ES ENCOUNTERED &	2	X 8	16	
Mollusca	Mollusca Bithynidae C				
Crustacea	Crustacea Atycke C				
TOTAL FAMILIE	S ENCOUNTERED & TO	TAL MULTIPLIED SCORE	2	X 6	12
Hemiptera	Hemiptera Hydrometridae C				
	Gerridae	D			
	Nepidae	В			
TOTAL FAMILIE SCORE	S ENCOUNIERED & TO	TAL MULTIPLIED	3	X 5	15
Mollusca	Planorbidae	C			
TOTAL FAMILI SCORE	ES ENCOUNTERED &	1	X 3	3	
Diptera	Chironomidae	D			
TOTAL FAMILI SCORE	ES ENCOUNTERED &	1	X2	2	
GRAND TOTAL F GRAND TOTAL M	AMILIES ENCOUNIER IULTIPLIED SCORE	ED &	9		48

Saprobic Score = Grand Total Multiplied Score \div Grand Total Number of Families Encountered

= 48/9 = 5.33

TAXONOMICAL GROUP	TAXONOMICAL FAMILIES	MARK ENCOUNIERED FAMILIES WITH ABUNDANCY AS A, B, C, D, E	TOTAL FAMILIES/ SPECIES ENCOUNTERED	BMWP SCORE	MULTI- PLIED SCORE
Odonata	Lestidae	В			
	Gomphidae	В			
TOTAL FAMILI SCORE	ES ENCOUNTERED &	2	X8	16	
Mollusca	Bithynidae				
Crustacea	Atydae				
TOTAL FAMILI SCORE	E S ENCOUNIERED	& TOTAL MULTIPLIED	2	X6	12
Hemiptera	Hydrometridae	В			
TOTAL FAMILIE SCORE	S ENCOUNIERED & T	OTAL MULTIPLIED	1	X5	5
Mollusca	Lymnaeidae	С			
	Planorbidae	С			
TOTAL FAMILI SCORE	ES EN COUNTERED	& TOTAL MULTIPLIED	2	X3	6
Diptera	Syrphidae	В			
Chironomidae D					
TOTAL FAMILI SCORE	ES EN COUNTERED	2	X2	4	
GRAND TOTAL F GRAND TOTAL N	AMILIES ENCOUNTER AULTIPLIED SCORE	9		43	

 Table 2. Calculation of BMWP Score (Saprobic Score) for Location no. 2

Saprobic Score = Grand Total Multiplied Score \div Grand Total Number of Families Encountered

$$= 43/9 = 4.77$$

Table 3. Biological Water Quality Criteria (BWQC) developed by CPCB

S. No.	Range of BMWP Score	Range of Diversity Score	Water Quality Characteristics	Water Quality Class	Indicator Colour
1	7 and more	0.2 - 1	Clean	А	Blue
2	6 - 7	0.5 - 1	Slight Pollution	В	Light Blue
3	3 - 6	0.3 - 0.9	Moderate Pollution	С	Green
4	2 - 5	0.4 & less	Heavy Pollution	D	Orange
5	0 - 2	0 - 0.2	Severe Pollution	Е	Red

Physico-chemical Parameter	Location 1	Location 2	Average Value	IS:10500 Drinking Water Specification Acceptable & Permissible [#] limit	FAO Guide- lines (1985) Usual Range in Irrigation water
pH	7.92	9.39	8.66	6.5 to 8.5	6.0 -8.5
Colour (Hazen)	5	10	7.50	5 (15)	-
Turbidity (NTU)	2.27	2.21	2.24	1(5)	-
Conductivity (µs/cm)	232	216.9	224.45	-	0-3000
Total Solids (mg/l)	166	196	181	-	
Total Dissolved Solids (mg/l)	134	156	145	500 (2000)	0-2000
Total Suspended Solids (mg/l)	32	40	36	-	-
Ammonical Nitrogen (mg/l)	0.29	0.42	0.36	0.5	-
Nitrate (NO ₃ -N) (mg/l)	0.17	0.048	0.109	45	0 - 10
Phosphate (mg/l)	0.016	0	0.008	-	0-2
Alkalinity - as $CaCO_3(mg/l)$	100	80	90	200 (600)	200
Total Hardness as CaCO ₃ (mg/l)	74.4	62.8	68.6	200 (600)	712*
Sodium (mg/l)	20.46	23.52	21.99	-	0-920
Potassium (mg/l)	3.1	2.5	2.80	-	0-2
Calcium (mg/l)	13.95	14.42	14.19	-	0-400
Magnesium (mg/l)	9.63	6.52	8.08	-	0 - 61
% Sodium	36.22	43.67	39.95	-	60^
SAR (milimole/l)	1.03	1.29	1.16	-	0 - 15
Dissolved Oxygen (mg/l)	4.55	13	8.78	-	-
COD (mg/l)	40	89	64.5	-	-
BOD (3 days 27°C) (mg/l)	10	26	18.0	-	-
Chloride as Cl ⁻ (mg/l)	23	41	32	250 (1000)	0-1065
Sulphate (mg/l)	14	16	15	200 (400)	0-960

Table 4. Water Quality of Thol Wetland in terms of Physico-chemicalParameters

#Permissible limits are shown in brackets.

*Values as suggested in Research Bulletin No.71, Directorate of Water Management, ICAR, 2014.

^SAR maximum value as per BIS Standards (2002): Designated Use Class E (irrigation purpose)



Source: GEER Foundation [1] Fig 1: Location of Thol Wetland



Fig 2: Benthic Macro invertebrate Sampling and Site Conditions



Fig 3: Biological assessment of Benthic Macroinvertebrates in Laboratory



Fig 4: Location wise Composition of Benthic Macroinvertebrates at Thol Wetland

BIOSYNTHESIS OF NATURAL POLYMER BASED NANO CELLULOSE FIBRE: TODAY'S TREND

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Abstract

Nanofibres due to its high surface area to volume ratio possess high functional properties such as flexibility in surface functionalities, superior mechanical performance, highly porous surfaces, low density due to small diameter of fibre, higher strength etc. Due to these properties, they are used in several fields like medicine, cosmetics, environment, energy, chemistry, electronics, textiles, defense, etc. Nanofibres manufacturing from synthetic polymers is usually preferred via electrospinning technique, but from natural polymers mechanical, chemical, physical and biological methods are used. Each method has its own positive as well as negative sides, but biosynthesis method (enzyme hydrolysis) is used popularly amongst all. This shifts in recent manufacturing trend is obliged by the increased awareness about Eco-friendliness. This paper briefly summaries different manufacturing techniques of nanocellulosic fibres used now-a-days.

Key words: Biosynthesis, Enzyme hydrolysis, Nanocellulosic fibres, Techniques.

Introduction

One dimensional flexible solid-state nanofibre" is an exciting new class of material in nanotechnology. This partiality is availed due to its larger specific surface areas per unit mass which impart unique properties such as flexibility in surface functionalities and superior mechanical performance in terms of higher tensile strength, highly porous surfaces, low density due to small diameter of fibre etc. ^[1, 2]. These properties make them to be successfully used in several fields like medicine (drug carriers, surgical materials, prostheses, wound dressing), cosmetics (creams and nutritional ingredients, tampons, masks), environment safety (sensors, filters, nanofilters, adsorbers), energy transmission & control (electric cells, hydrogen storage chemistry based reactions (catalysts with high efficiency, ultra-light materials and composites), electronics fields (computers, shields for electromagnetic radiation, electronic equipment), textiles (clothing and functional products), defense(special-purpose clothing, face masks) etc. ^[3].

Categories of Nanofibres

Nanofibres are basically classified into two groups: i) polymeric nanofibres and ii) cellulosic nanofibres.

A. Polymeric nanofibres

Majority of polymeric nanofibres are obtained through the bottom-up approach using electro-spinning of polymer melts or solution from synthetics polymers like PVA, PLA or their mixture [3]. Apart from electro spinning other techniques were also established; such as drawing, template synthesis, phase separation, self-assembly etc., but yet electrospinning is found to be the most popular amongst them ^[4].

B. Cellulosic nanofibres

Cellulosic nanofibres are isolated from biomass likes natural vegetables fibres, stem of plants, bark of plants, fruit etc. Nano cellulose fibres become more popular over the synthetic polymeric nanofibres because of its abundant renewable resources and environ friendly nature, low toxicity, biodegradability, biocompatibility, low cost, etc. Second approach of top-bottom is used for that category of nanofibres ^[3], where the biomass is disintegrated to nano size with the use of mechanicals, chemical, physical or biotechnological methods. However, highly preferred electrospinning technique in case of synthetic polymer based nanofibres is not found to be successful in this area due to processing limitations of cellulose based material. Amongst rest of production methods, biotechnological synthesis method has been found to be more acceptances due to absence of liberation of toxic byproducts, low energy consumption and avoidance of costlier chemical components used in the synthesis process ^[3-5], making the method environ friendly & economical without scarifying quality. This paper summarizes various techniques used in the production course of Nano cellulose fibre and highlights superiority of biosynthesis technique for natural polymer to get desired nano cellulose fibre with respect to present day needs of the world.

Different Synthesis Techniques for Nanofibre

A. Electrospinning Method

Electro spinning technique is able to fabricate from continual macro polymer into nanofibres from different types of polymeric materials ^[6]. This method produces dimension controlled nanofibres in nanometer ranges. A melt or solution of polymer is fed through a narrow needle or nozzle which acts as an electrode, simultaneously applying high voltage

16

and current varying from nano to micro ampere during spinning process. If high voltage is applied to an electrode, electric charge builds up on the surface of the solution. The charge is attracted to an electrically grounded collector and an electric field is generated between them. After critical voltage value, electrostatic force overcomes the surface tension of polymeric solution or melt and the charged polymer solution is forced to leave through the tip of needle. The pulled polymer solution jet passes through an elongation and instability phase due to the effects of electrostatic force which makes the jet very thin and long. Simultaneously, solvent evaporates and solidification takes place and an interconnected layer of fibres on surface of collector is created. In the case of melt, ejected jet solidifies while travelling in the air and finally nanofibres are collected and patterned on a grounded plate.^[2-6]

The system offers many advantages amongst which major are;

- High production rate of nanofibre,
- Simplicity, high efficiency along with high reproducibility.
- Possibility to control fibre morphology etc.

Simultaneously it has challenges like;

- Used solvent can be toxic.
- Raw material is more costly than the biomass nanofibre's raw material.
- Process is difficult to be exactifying as depends on many variables ^[7].

Electrospinning process can also be used to obtain nanofibres from cellulose and its derivatives. But some problems were reported in the processing of biopolymers like cellulose due to limited solubility of cellulose in typical solvents and its tendency to aggregate or form gels. This resulted in low process efficiency, low yield in long time and some solvents had a low volatility which leads to defective fibres on spinning. Additionally, some of these solvents require high temperatures to dissolve cellulose completely which makes nanocellulose synthesis by this technique more difficult. Substantiating this Maria et al^[8] findings showed that residual ions are difficult to remove from the obtained fibres ^[8]. Therefore, synthesis of nanocellulose gets shifted to other production methods like mechanical, chemical, physical, biological method.

B. Mechanical Methods

This method utilizes mechanical means (homogenizer) in the generation of desired nanofibres like high temperature, high pressure/cavitation/shear/impact for sequential break down of the cell walls of the microfibres generated from chopped pulp fibres ^[9]. The system is identified on the basis of media used in this breakthrough, viz;

- a) High pressure homogenization (HPH)
- b) Grinding process
- c) Cryocrushing
- d) Steam explosion

a. High Pressure Homogenization (HPH)

In 1985 turbak et al. found first mechanical treatment and that was gaulin homegenizer [10]. The process consisted of passing cellulose slurry into a vessel through very small nozzle at high pressure. At such high velocity and pressure, the impact of the shear forces on fluid generated shear rates in the stream and decreased the size of fibres into the nanoscale. HPH is highly efficient, simple method and it does not need any organic solvents [11]. However, energy conservation is higher with this system which adds to cost of the product. The microfluidizer is recent advanced version of HPH provided with intensifier pump to increase pressure, as well as interaction chamber to defibrillate the fibres through shear and impact forces against the colliding streams and the channel walls. F. A. Dos Spence *et al.* [11] have found while working with microfluidization with refining and micro grinding techniques that this system required less energy as compared to that for HPH [11].

b. Grinding Process

Masukoc was the first apparatus operating with grinding approach and known as grinder. Breakdown of cell wall structure due to shearing forces, generated by grinding stones is the working principle underlying this apparatus. Pulp of cellulosic material is passed in between static stones then into grinder rotating at around 1500 rpm. Thus, nanofibres are individualized from cell wall of pulp which consists of multilayer structure. The process can degrade the pulp fibres and decrease their length due to higher abrasion involved in this course, but no significant changes in morphology has been reported ^[10]. However, similar to HPH, higher energy conservation is true for this system also for grinding purpose.

c. Cryocrushing

In Cryocrushing method, fibres are first of all frozen by using liquid nitrogen to form ice crystals within the cell wall. High impact forces are applied in the second step to the frozen fibres in ice containing crystals in order to exert pressure on the cell walls, causing them to rupture and there by librate micro fibrils. They are further converted to desired nanofibres by using HPH or other process in third step ^[12].

18

d. Steam Explosion

High pressure steaming is followed by rapid decompression in steam explosion system. The process includes saturating a dry material with steam at elevated pressure and temperature followed by sudden release of pressure during which the flash evaporation of water exerts a thermo mechanical force causing the material to rupture into nano fibrils ^[13].

All the methods covered under mechanical synthesis have major limitations as follows:

- Not able to produce single fibre at a time, always produces them in group.
- Time consuming.
- These treatments involve high consumption of energy due to higher number of passes required for disintegration thereby making it costly.
- Mechanical damage to the fibres like reduced fibre length [14, 15].

C. Chemical Methods

Here in, chemicals are used for the synthesis of nano cellulosic fibre. The following methods are categorized on the basis of medium used for the hydrolysis purpose, viz; acid, alkaline, organic solvent and ionic liquid.

- a) Acid hydrolysis
- b) Alkaline hydrolysis
- c) Organic solvent treatments
- d) Ionic liquid treatments

a. Acid Hydrolysis

This method involves use of acid hydrolysis for the production of stable aqueous suspensions of cellulose nanofibre. Mineral acids like H_2SO_4 , HCl and H_3PO_4 can be used for this purpose. It produces micro and nano fibres of a higher degree of crystallinity by removing the amorphous regions of the raw cellulose materials. Negatively charged surface of cellulose fibres can be obtained through the esterification of hydroxyl groups by the sulfate ions using this method. The time, temperature of process, acid concentration...etc are the factors which play an important role concerning the morphology and the dimension of the isolated fibres.

However, acid hydrolysis is liable to decrease steadily the degree of polymerization, molecular weight of MCC and crystallinity of MCC significantly increased due to degradation of amorphous domains in cellulose. Higher crystalline cellulose had the higher

thermal stability compared to that of the raw material. This method can be used alone or in combination with others method for isolation of nanofibres ^[16].

b. Alkaline Hydrolysis

Treatments in this category are usually made using diluted solutions of NaOH (1-10%) at high temperatures NH_4OH and anhydrous NH_3 (gas or liquid) are also used to activate the organic materials, in some cases providing an increase of the hydrolytic degradation.

Zuluaga et al. ^[17] reported that different alkaline treatments such as peroxide alkaline, peroxide alkaline-hydrochloric acid, 5wt% potassium hydroxide and 18wt% potassium hydroxide used to obtain cellulose nanofibres. Alkaline hydrolysis determines the partial separation of the cellulose fibres from the cell wall and an improvement of physical and chemical characteristics of cellulose, specifically its reactivity with other chemical agents [16].

c. Organic Solvent Treatments

Organic solvents are used in this method for the isolation of micro and nano cellulose. The major advantage of this technology is the easier recovery of organic solvents by distillation and the absence of residue.

Oksman et al. ^[18] has worked on this system and reported that the cellulose fibres were swollen into solvent system (N, N-dimethylacetamide and lithium chloride) in order to facilitate isolation of the cellulose nano fibres. Cellulose can also be dissolved in others solvents such as N-methylmorpholine-N-oxide, trifluoroacetic acid, DMSO and DMF for this purpose ^[16].

d. Ionic Liquid Treatments

Ionic liquids are a new group of organic salts and they can remain in fluid state at relatively low temperature (<100°c). They are chemically and thermally stable, noninflammable with extremely low vapour pressure. Cellulose can be easily regenerated from its ionic liquid solutions by the addition of water, ethanol or acetone. The ionic liquids can be recovered by various methods like evaporation, ion exchange, and reverse osmosis and they can be reused. Microwave heating can be helpful as it can significantly accelerate the cellulose dissolution process in this category. Various researchers have worked in this area like; Adriana et al had dissolved cellulose in hydrophilic ionic liquids, for e.g. 1-butyl-3-methylimidazolium chloride (BMIMCI) and 1-allyl-3-methylimidazolium chloride (AMIMCI). Swatloski et al.^[19] reported the use of an ionic liquid as solvent for the regeneration of cellulose and for the chemical modification of polysaccharide.

20

Zhang et al.[20] studied the dissolution and regeneration of cellulose in 1-ally-3methylimidazolium chloride (AMIMCl), without any pretreatment. Regenerated cellulose material based nanofibres have shown better performance as a group in terms of mechanical properties ^[16].

Although it requires shortest route, chemical methods have following challenges:

- Yield is very low.
- Increase in acid concentrations, crystallinity index decreases rapidly.
- Environmentally unfriendly due to liberation of toxic elements as byproducts.
- Fibre cell wall can be embrittled by acid hydrolysis. Acid hydrolysis may reduce the reinforcement effect, etc [14, 15].

D. Physical Methods

Various concepts in physics such as ultrasonication and microwave radiation are used here for generating desired impact for the disintegration of cellulosic fibres into nanofibres and hence they are categorized under physical methods.

- a) Ultrasonication treatment
- b) Microwave heating

a. Ultrasonication Treatment

This method is used alone or in combination with other methods e.g. acid hydrolysis ^[16]. In this method chemically purified material is soaked in distilled water and then this mixture is subjected to ultrasonicfibrilation using ultrasonic generator with cylindrical probe. The ultrasonicfibrilation is carried out in an ice bath and ice is maintained throughout the process ^[21]. During the process ultrasonic energy is transmitted to cellulose chains with the help of cavitation process, which involves formation, growth and violent collapse of cavities in water. The energy (10-100 KJ/mol) gained by cavitation (it is called as sonochemistry) is within hydrogen bond energy scale. Thus, ultrasonic impact can gradually disintegrate the micron sized cellulose fibres into nanofibres ^[22].

b. Microwave Heating

Normally, microwave irradiations can generate volumetric heating within short lapse of time by initiating efficient internal heating through a combination of microwave energy with reactant molecules present in the reaction mixture. This provides rapid energy-efficient heating of the biomass substrate and can produce NCC within a short interval of reaction time. This method is normally used in combination with chemical method like acid hydrolysis ^[23].

Both the physical methods utilize too high impact power liable to degrade material more. Thus the biggest disadvantage of this method is it produces highly degraded material and thereby nano fibres so generated execute very low strength characteristics ^[16].

All the above mentioned methods used for cellulose nanofibre isolation have drawback in terms of either higher cost of synthesis, poor environ friendliness or material damage (poor quality). Therefore current research has been focused on finding new environmentally friendly method to isolate cellulose fibres, characterized by high efficiency and low cost [24].

E. Biological Methods

Biosynthesis method became more popular and acceptable method than the others methods nowadays for the isolation of nano cellulose fibres. This is attributed to the use of abundantly available natural components in the processing course which makes process economical and enviro-friendly. Use of peculiar solvents or chemical reagents which can adversely affect either final products quality or environment can be well avoided ^[3, 5]. Enzyme hydrolysis is the most popular method in this category used by various researchers.

Enzyme Hydrolysis (EH):

Trichoderma, phanerochaete, aspeigillus, cellulases, etc are the enzymes used in EH. But cellulases are the most popular in the commercial industries and they are produced by fungi, bacteria, protozoans, plants and animals. Enzyme hydrolysis needs to be done for a prescribed time to circumvent complete cellulose degradation. This method gives high yield without compromising the quality of the product, with no environmental impact and socioeconomic value [5, 25, 26].

The process of nanocellulose isolation via EH route requires prior removal of intrinsic impurities of pectins, wax, hemicelluloses and lignin. Because the enzymes tend to irreversibly bind to lignin through hydrophobic interactions that cause loss in their activities and hemicellulose to create enzyme impenetrable cross links with lignin ^[5, 26].

Isolation of nano cellulosic fibres from washed cellulose is a complex process, but this could be made easy by the use of enzymes due to the breaking/catalysis of linking bonds. Microfibres are joined laterally by means of hydrogen bonding, as the micro fibrils are generated and they are found to coalesce laterally through interfibrillar hydrogen bonding to form bundles. The interfibrillar hydrogen bonding energy has to be surpassed

22

in order to separate the micro fibrils into individual entities which have nanoscale dimensions. In recent studies it was found that cellulose fibres were first of all mixed with water for making the cellulose fibres more accessible for enzyme through increasing surface area by swelling in the water (Bharat; please explain this sentence, specially highlighted one). As high fibrillation of cellulose fibre is achieved after swelling with water. Such swollen cellulose fibres have greater surface area to react with enzymes, which showed a more favorable structure of nanocellulose. Lopez-Rubio et al. ^[27] and Svagan et al. ^[28] also confirmed improved properties of final product after enzymatic treatments ^[26].

Enzymes gained popularity due to their environmentally friendly attributes. They do not require peculiar solvents, chemical reagents and most of all, they are energy effective, and their optimum operational conditions are really feasible and cost effective. In biosynthesis method, use of toxic materials is thus eliminated and it uses raw materials from renewable sources. It reduces use of water and air pollution and results in minimized chemical hazards. However, processing time of this system is more than the other methods. No doubt overall cost of the product is low due to use of much cheaper ingredients in the production course [5, 25, 26, 29].

Thus significance of biosynthesis method lies in the creation of better, safer nanofibre with safest and most efficient ways to synthesize material and reduce waste. It holds benefits over chemical, mechanical and physical methods due to it's cost effectiveness, eco friendliness, being energy efficient and meeting comfortably statutory regulation on the account of lesser waste, fewer accidents, safer products, healthier work places and communities. All the above points make biosynthesis method much more noteworthy than the other methods ^[29].

Conclusions

Now a day's concept of modern world is back to nature. Therefore, researchers trying to find out new materials based on natural substances and residues. In current time, cellulose which has been considered as promising material for producing nanocellulose fibres for broad applications is synthesized using different techniques from various natural resources and bioresidues. Nanofibres are produced from the available techniques like electrospinning, mechanical, chemical, physical and biological treatments. Today researchers are trying to shift towards the use of novel, environmentally-friendly methods for producing synthesizing nanocellulose fibres. Therefore biological method that is enzyme hydrolysis is becoming more popular and suitable in this regards in comparison to others methods.

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COMPARATIVE STUDY OF SHAFT SUPPORTED AND FRAME SUPPORTED INTZE WATER TANK USING LIMIT STATE METHOD

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Abstract

Overhead water tanks or elevated service reservoirs are one of the most important components of any efficient water distribution system. The basic purpose of elevated water tanks is to secure constant water supply. The aim of this study is to compare most popular elevated tanks viz. shaft supported intze tank and frame supported intze tank for its various parameters. Analysis and design of elevated shaft supported intze tank is carried out in Microsoftexcel. Fortrestle (frame) supported intze tank, analysis and design of container is carried out in Microsoft excel and analysis and design of staging is carried out in professional staad pro. Software. Comparison of base shear, base moment, quantity of concrete, quantity of steel and combined cost of steel and concrete as per schedule of rates of Gujarat water supply & sewerage board is carried out for shaft supported and frame supported intze tank. Tanks are analyzed and designed for capacities of1000 m³, 1500 m³ and 2000m³. Design work is based on Limit state method.

Introduction

Storage reservoir is a term used for structures, designed to store water, petroleum products and similar other liquids. The structural analysis of all reservoirs is similar irrespective of the chemical nature of the product being stored. Such structures are important public utility structures more particularly in high seismic zones. For such structures, one of the main considerations, besides strength, is that they should be leak proof hence it should be ensured during design stage that concrete does not crack on the liquid face or crack width is within permissible limit. The concrete used for such structures should be well graded and well compacted, so that the tensile strength is high and the porosity is low.

Such reservoirs are very important part of drinking water distribution system. In water distribution system, water is first stored in underground sump storage reservoirs, usually two to three times the capacity of the elevated reservoir, and is chlorinated before being

pumped up into reservoirs for distribution. Elevated reservoirs are used to meet demand during peak supply hours.

There are mainly four types of elevated water tanks: Intze tank, Rectangular Tank, Circular tank and the conical tank. Tanks are classified as small (<1000m³), medium (1000m³ to 2000m³) and large (>2000m³) based on its capacity. The medium capacity intze tanks are used more commonly all over the world. The intze tanks are supported on either shaft staging or trestle staging. The shaft staging acts as a cantilever as a whole from foundation and does not have any possibility of redistribution of moments. On other hand the trestle staging has capacity to redistribute the moments among the members meeting at joints, thus it can take advantage of redundancy.

Design Criteria:

In our study medium capacity is selected so that we can predict the behavior for small capacity and large capacity elevated water tanks.

- (1) **Exposure:** For container elements as per code for severe exposure clear cover is taken as 45 mm. For other elements clear cover is taken as per code for mild exposure.
- (2) Minimum Dimensions: In most case, we use two face reinforcements. Thus thickness of cylindrical wall of container, bottom dome, conical dome and shaft is taken minimum 200mm. Technically we can provide thickness lesser than 200mm, but due to provision of reinforcement in two layers with 45mm clear cover and minimum criteria suggestion by R.D. Anchor, we consider 200mm minimum thickness. However in top dome minimum thickness is considered as 100mm because reinforcements are provided in one layer.
- (3) **Stair:** Stair is not designed in this study as effect of stair will remain same for both types of tanks.
- (4) **Minimum Reinforcement:** The minimum reinforcement in walls, floors and roofs in each of two directions at right angles, within each surface zone shall not be less than 0.35 percent of the surface zone for high strength deformed bars and not less than 0.64 percent for mild steel reinforcement bars. The minimum reinforcement can be further reduced to 0.24 percent for deformed bars and 0.40 percent for plain round bars for tanks having any dimension not more than 15 m. In wall slabs less

than 200 mm in thickness, the calculated amount of reinforcement may all be placed in one face. For ground slabs less than 300 mm thick the calculated reinforcement should be placed in one face as near as possible to the upper surface consistent with the nominal cover. Bar spacing should generally not exceed 300 mm or the thickness of the section, whichever is less.

(5) Limit States of Serviceability: Deflection - The limits of deflection shall be as per IS 456. Cracking-The maximum calculated surface width of cracks for direct tension and flexure or restrained temperature and moisture effects shall not exceed 0.2 mm with specified cover.

Shaft Supported Intze Tank:

Figure 1 shows typical diagram of shaft supported intze tank. Shaft diameter is so selected that it is not subjected to tension in any of load combinations and remains at boundary i.e. axial stress and bending stress are equal. Lateral stiffness of shaft supported tank is taken as 3EI/L³ as per code.

Frame Supported Intze Tank:

Figure 2 shows typical diagram of frame supported intze tank. Bracing System for frame supported Intze tank is provided as shown in figure 2 so that every column gets support in both primary direction for lateral load and also we can provide stair at center and it will not cause any eccentricity. Diameter of frame and size of bracing is selected such that displacement at top of tank should not exceed codal criteria.





Section B-B

Figure 2 Frame supported intze tank

Figure 1 Shaft supported intze tank

Data:

TABLE 1

Common Parameters	
A.B.P. of Soil	200kN/m ²
Height of staging	20.5m
Depth of staging below ground level	2.35m
Internal Diameter of stair shaft	2.15m
Location	Baroda
Seismic Zone	III
Importance factor for Seismic load(I)	1.5
Response reduction factor	3.5
Type of soil	Medium
Terrain category	3
Class of structure	В
Risk Co-efficient (k1)	1.06
Topography factor(k3)	1
Importance factor for wind load(k4)	1.3
Grade of concrete	M30
Grade of steel	Fe415
Estimate	As per S.O.R. OF G.W.S.S.B.

TABLE 2

Geometrical Dimensions of Shaft supported Intze Tank for different capacity

Parameters	1000m ³	1500m ³	2000m ³
Central rise of top dome	2.60m	2.80m	3.3m
Internal diameter of cylindrical wall	16.00m	17.8m	19.60m
Overall height Of cylindrical Wall	3.62m	4.3m	4.67m
Free board	0.30m	0.3m	0.35m*
Height of conical dome	3.20m	3.8m	4.35m
Width of Conical Dome	3.20m	3.8m	4.35m
Central rise of bottom dome	1.50m	1.55m	1.60m
Diameter of main shaft	9.38m	9.90m	10.45m
Shaft Thickness	225mm	225mm	225mm

TABLE 3

Geometrical Dimensions of Frame supported Intze Tank for different capacity						
Parameters	1000m ³	1500m ³	2000 m ³			
Central Rise of top dome	2.7 m	2.9m	3.3m			
Internal diameter of cylindrical wall	16.5m	17.9m	19.5m			
Overall height Of Cylindrical Wall	3.41m	4.44m	4.95m			
Free board	0.3 m	0.3m	0.35m*			
Height of conical dome	3 m	3.42m	3.9m			
Width of Conical Dome	3 m	3.42m	3.9m			
Central Rise of bottom dome	1.65m	1.67m	1.7m			
Diameter of frame	10m	10.5m	11m			
C/C distance between bracing	3.2 m	3.2m	3.2m			

* Free board is increased to accommodate sloshing wave height.

		Size					
Element	1000m ³		1500m ³		2000m ³		
Top Dome	100mm		10	0 m m		100mm	
Ring beam B1	300m m X 300	mm	300 m m	X 300m m	300m	m X 300mm	
Cylindrical wall	200mm thic	k	200m	m thick	20	0mm thick	
Ring beam B2	400m m X 400	mm	400mm	X 400m m	400m	m X 400mm	
Conical Dome	Conical Dome 200 mm thick		200 mm thick		200 mm thick		
Ring Beam B3	225m m X 400	mm	400mm X 400mm		450m	m X 450mm	
Bottom Dome	200mm thic	k	200mm thick		20	0mm thick	
Shaft	225mm thic	k	225m	m thick	22:	5mm thick	
Annular raft							
Thickness at ed	1.	.45m	2.2 m		2.75m		
Thickness at edg	0.2	725m	1.1 m		1.375m		
Inner ra	2	.6m	2.42m		2.2m		
Outer ra	dius	6.	.55m	7.56m		8.5 m	

TABLE 4

Section Dimension of Shaft supported intze tank for different capacity

TABLE 5

Section Dimension of Frame supported intze tank for different capacity

El				Size	
Element	1000m ³		1500m ³		2000m ³
Top Dome	100m m		100mn	1	100mm
Ring beam B1	300m m X 300i	mm	300mn	n X 300mm	300mm X 300mm
Cylindrical wall	200mm thick		200mn	n thick	200mm thick
Ring beam B2	400mm X 400n	mm	400mn	n X 400mm	400mm X 400mm
Conical Dome	200 mm thick		200 m	m thick	200 mm thick
Ring Beam B3	1.0m X 0.5m		1.25m X 0.575m		1.50m X 0.65m
Bottom Dome	200mm thick		200mm thick		200mm thick
Bracing beam	230m m X 4601	nm 230mm X 460mm		n X 460mm	230mm X 460mm
Colum n(circular)	530m m	610mm		1	690mm
Annular raft					
Thickness at edge of	f beam	0.9m		1.3m	1.6m
Thickness at edge of	0.45m		0.625m	0.8m	
Inner radius	3.40m		3.15m	2.80m	
Outer radius		6.40m		7.15m	7.9 m
Ring Beam B4		1.3m X	0.53m	1.5m X 0.61m	1.7m X 0.7m

Results

Comparison of Base Shear and Base Moment due to earthquake and wind forces

TABLE 6

TABLE 7

Comparisi	on of base S force(kN)	hear of Wind	Compa eai	rision of bas thquake for	e Shear of ce(kN)
Tank	Tank Wind force			Earthqu	uake force
Capacity	apacity Shaft F		came Capacity	Shaft	Frame
1000m³	269	324	1000m³	704	216
1500m³	500m ³ 310 388		1500m³	971	284
2000m³	356	454	2000m³	1224	353

TABLE 8

TABLE 9

Comparision of base Moment of wind force(kNm)			Comparision of base Moment of earthquake force(kNm)		
Toult Consoity	W	ind force	Tonk Conceity	Earthquake Force	
тапк Сарасну	Shaft	Frame	тапк Сарасну	Shaft	Frame
1000m ³	4784	5692	1000m ³	17955	5709
15000m ³	5878	7126	15000m ³	25506	7753
2000m ³	7061	8600	2000m³	32936	9943

Comparison of Quantity of concrete and steel

TABLE 10

TABLE 11

Comparision of Concrete Quantity(n?)			Comparison of Steel Quantity(kg)		
Tank Capacity	Shaft Supported	Frame Supported	TankCapacity	Shaft Supported	Frane Supported
1000n i	398	295	1000n î	22202	29188
1500n‡	574	412	1500n î	31571	40198
2000n î	782	556	2000n î	42523	51611



Chart: 1: Concrete quantity vs. capacity of tank Chart: 2: Steel quantity vs. capacity of tank

Comparison of estimate for concrete and steel for shaft and frame supported intze tank

Comparis ion of Cost(INR)					
Tank Capacity	Shaft Supported	Frame Supported	Diffirence	% Difference	
1000m³	43,87,609	41,74,138	2,13,417	5.1	
1500m³	61,62,556	57,05,222	4,57,334	8	
2000m³	82,46,441	74,64,707	7,81,734	10.5	

Conclusions

- 1] In shaft supported intze tank earthquake forces governs. In frame supported intze tank wind forces and earthquake forces are nearer compared to shaft supported intze tank as per our particular case.
- 2] Earthquake Base shear and base moment of shaft supported intze tank is about 230% higher compare to frame supported intze tank. This phenomenon is attributed to higher stiffness offered by shaft.

- 3] For frame supported intze tank steel quantity required is more about 26% compared to shaft supported intze tank.
- 4] Concrete quantity required for shaft supported intze tank is more about 37% compared to shaft supported intze tank.
- 5] From table: 12, as capacity increase difference in combined cost also increases. So we can predict that for large capacity frame supported intze tank will be more economical whereas for small capacity shaft supported intze tank may cheaper than frame supported intze tank.

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REVIEW ON FRACTURE PROCESS ZONE OF CONCRETE

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Abstract

The review presented here discusses the kind of work carried out on the fracture process zone of concrete and identifies the controversies within the subject or any recent work. The review first establishes the sight into the fracture process zone of concrete and its behavior under various exposure conditions. The difference in the fracture process of concrete upon loading and mechanism of the microcracking has also been discussed. The modeling of the fracture process zone of concrete has been taken into account and the experimental determination of the FPZ has also been discussed. The applicability of the non-linear fracture mechanics to the concrete structure is illustrated. The purpose of the review is to develop an insight to the Fracture Process Zone of concrete and the understanding of the same by including the various research works carried out. The review also identifies the cause and the dimension of the Fracture Process Zone in concrete by using various non-linear models and experimental analysis.

Key words: Concrete, Fracture Process Zone (FPZ), microcracks, hardness, sub-critical cracking, quasi-brittle material, acoustic emission technique, fracture toughness, crack bridging, crack branching, mesoscale approach, fictitious crack model, crack band model, strain softening, inter facial transition zone, nondestructive testing methods

Introduction

Fracture Process Zone is defined as the region intermediate to the cracked and un-cracked portion of the material. Fracture Process Zone contains micro-cracks and these micro-cracks are individual cracks situated nearer to the crack tip in the material. On the application of load, the crack grows and along with the crack, micro-cracks merge and gives continuity

to the growing crack. FPZ can be considered as a linking zone between the cracked and un-cracked portion of the material.[1] FPZ is only observed in the brittle material or quasibrittle material as concrete. Study of the FPZ helps in determining the crack growth and ultimate failure of the concrete. As the concrete is having large FPZ, accurate determination of the crack in concrete is not possible and that is the reason for concrete that unlike steel the yielding cannot be shown by concrete.

Fracture Analysis of Concrete

Concrete fails in tension easily than in compression. Inability of concrete to resist tensile loading is that the aggregates in concrete are capable of taking the compressive stress but during tension, the cracks are formed within the material and these cracks merge with the advancing crack and a large FPZ is formed ahead of the crack tip. The micro-cracks keep on merging and the concrete fails in tension. To improve the behavior of concrete against tension rebars are used as reinforcements as they are capable of taking the tensile load in the concrete components.

Linear Elastic Fracture Mechanics to Concrete

Application of LEFM theory to the concrete does not yield the reliable results due to certain unanswered phenomena taking place in concrete upon loading. If concrete is considered as a pure brittle material and along with that LEFM is applied to it, some real conditions such as large amount of stresses at the crack tip, does not hold true. At the crack tip, amount of stress is finite and LEFM cannot give the accurate stress at the crack tip. Size of the specimen is an important parameter to be considered in fracture process of concrete.^{[1][2]}This affects the critical stress value of concrete specimen as critical stress is a material property as well as it is size dependent which has not been taken into Figure 1 Reference: https://www.researchgate.net/figure/Behaviorof-concrete-undertension_fig3_237962593 Plastic Zone FPZ Elastic Zone Figure 2 account in LEFM. Another parameter which limits the scope of LEFM to concrete is the tension softening behavior of concrete due to microcracking.[1]After the attainment of peak load, behavior of concrete. Even before the peak load, Ref.



From the above graph, it can be seen that after the critical tensile strength is reached upon loading, the fracture process zone starts developing due to formation of the micro cracks which results in the non-linear behavior of concrete. The region after the peak load suffers some material softening followed by the strain softening behavior of concrete. [1] Thus from the above discussion, it can be said that the linear theory is not sufficient from the fracture behavior point of concrete. The theory which is applied to the concrete should be non-linear taking into account the existence of the FPZ in concrete along with tension softening behavior of concrete.

Non-Linear Fracture Mechanics to Concrete

As the evolution of the LEFM, an improved theory giving the reasonable explanation about the fracture process zone of concrete and taking the non-linear concrete exhibits some non-linear behavior which has minor influence on the fracture behavior of concrete and this has been emphasized in LEFM. But the major influence on the fracture behavior of concrete comes from the tension softening which is limited under LEFM. The following graph shows the tension softening region in the concrete. behavior of concrete has been made. This non-linear theory analyzes the plastic zone developed in the quasi-brittle material like concrete and also the FPZ surrounded by this non-linear zone at the crack tip. The response of the material is non-linear because of the micro-cracking. The following figure shows the presence of FPZ and plastic zone near the crack tip.



The process of fracture of concrete can be well understood by applying non-linear analysis and the mechanism of concrete fracture could also be analyzed. When a macro crack is introduced in the concrete it gives rise to several minute cracks near the flaws and second phase particles causing debonding from the surrounding cement paste. On application of the external loading micro cracks merge and join the external macro crack. This way the crack bridging occurs and the crack propagates leading to the failure of concrete. The crack growth can be arrested by the presence of the pores or flaws or the non-homogeneity of concrete and the presence of the second phase hard particles. The inherent flaws are attractive to the propagating crack and act as energy dissipaters to the growing crack. Thus the crack has to have higher amount of energy or needs some external work to grow Figure 3 further. Sometimes the crack is forced to grow around the particles which may result in crack bridging and this crack bridging can give rise to crack branching. This crack bridging is the prevailing reason for the extended tail region in tension softening diagram (Fig. 1). ^[1] The techniques based on the laser interferometry detected the presence of the micro cracks responsible for the failure of the concrete. However these optical techniques could not give the distribution of the micro-cracks along the thickness.

Thus, from the above explanation, it can be said that non-linear theory can be satisfactorily applied in order to identify the fracture mechanism of the quasi brittle material like concrete. It gives reliable results along with eye sight to the crack bridging and crack branching phenomena in concrete. The concept of the fracture process zone in concrete could also be explained well while applying nonlinear analysis to the concrete. At the crack tip, a discrete crack is considered along with the softening zone at the crack tip and the closing stresses are taken to be applied at the same. The non-linear behavior can be described by the experiments and the relation between the stresses and the softening zone size can also be described. The experimental studies showed that non-linear fracture mechanics can be applied to the concrete like material. Treating the concrete as an elastic softening material implies that the tensile specimen of the concrete shows the linear stress-strain behavior till the maximum stress is applied and there after the carrying capacity of the concrete specimen will be decreasing with the increasing value of deformation under the tensile loading.

Models used to determine the non-linear behavior of concrete

Concrete is having a tension softening zone in order to determine the non-linear behavior of concrete, two models were used namely the Dugdale model and Barenblatt model. After the application of these two models to the concrete specimen, the crack is assumed to be subdivided into two parts namely the traction free crack faces and the crack faces where the closing stresses are being acted. The non-linear approach of the concrete with the closing stresses is shown below (Fig. 3), where the traction free crack extends up to length 2S and the closing stresses (σ c) are acting on the area (B-S). In the case of concrete, the stress distribution is varying as per the deformation and the behavior of the softening phenomena. As the cyclic loading is applied on the concrete tensile plate specimen, the stiffness decreases and the deformation increase in the specimen of the concrete which is not reversible corresponding to the post-peak elastic loading. The pre-peak behavior can be illustrated by the extension of the tension softening zone of the concrete specimen. The

behavior of the concrete under the cyclic loading can be modeled by the nonlinear fracture mechanics. ^[2]



Concrete as a Material

Concrete is treated as a brittle material but not the perfect brittle material. Concrete is considered as a Quasi-Brittle material as it is having considerable hardness. The reason of having hardness is the subcritical cracking that occurs during the loading of concrete. This sub-critical cracking results in nonlinear stress-strain behavior and thus concrete gains the hardness from subcritical cracking.

Findings from Research

To evaluate the size and shape of the FPZ in concrete, various experiments were carried out in which X-rays or acoustic emission techniques were used. From the results of the experiments carried out on concrete specimen, it has been found that with an increase in the loading, a zone containing micro-cracks develops ahead of the crack tip.[3] From the experiments it was also found that the size of FPZ in concrete can be large based on the size of aggregates used. It was also observed that the crack growth in concrete is corresponding to a nonlinear zone about the crack tip. To predict the range of the nonlinear zone, a theoretical model was developed based on the concepts of linear elastic fracture mechanics. ^{[3][4]}

In acoustic emission technique, it was observed that the FPZ is created at the maximum load and it can expand with the crack growth. The rate of expanding the FPZ was found to be affected by the aggregate size. ^{[5][6]} Acoustic emission energy is also used to determine the size of the FPZ of concrete. With the amount of energy released and locations of the acoustic emission events, the size of the FPZ was obtained. ^[7] In further research, it was also investigated that for the material like concrete LEFM cannot be applied to determine the fracture toughness of concrete due to presence of FPZ.

It was found necessary to have the knowledge of micro level failure mechanism of a material to improve the material quality associated with the FPZ. In order to improve the material properties it is necessary to identify the location of internal cracking. In order to obtain the information regarding micro-cracking, acoustic emission techniques were used as described above. The use of acoustic emission technique also gives the idea about the applicability of material model. Study was carried out to determine the process of cracks localization corresponding to the movement of the FPZ using acoustic emission technique. The study for crack localization effect was carried out for concrete specimen loaded in tension. The results lead towards a conclusion that the acoustic emission process localizes near the crack tip before the peak load is reached. ^[8]

Along with the acoustic emission parameters of notched concrete specimen, the load deflection curve can give the idea of the length of FPZ for three point bending concrete beams. It was observed in the experiments that the length of FPZ is not a material dependent parameter rather it was affected by the specimen size. With the small crack length, FPZ increases linearly with the crack and as the crack reaches half of the beam width FPZ in concrete has reached to the maximum. With further increase in the crack length beyond maximum, FPZ was observed to have shrunk. ^[9]

Application of the Non-linear Fracture Mechanics to Concrete Structures

The performance of the concrete structures and the earthquake response was studied by using nonlinear fracture mechanics along with the numerical analysis. Two basic non-linear models used were Hillerborg's fictitious crack model and Crack Band Model. Both the models are non-linear and taking into account the effect of the strain softening behavior of concrete. The crack band model assumes the fracture process of the concrete by means of the micro cracking which is progressive in nature. The concrete material will undergo the strain softening as the strain increases. The performance of the concrete structures and the earthquake response was studied by using the non-linear fracture mechanics and the numerical analysis. Also during the crack extension, the fracture energy will be released and it must overcome the energy dissipated by the tension softening in the concrete. The length of the crack will rely upon the fracture energy as it is inversely proportional to the crack length.^[10] Cementitious materials show strainsoftening on reaching the peak load. The nonlinearity exists in the concrete structure can be described by the cohesive forces acting in the fracture process zone. The fracture process zone near the crack-tip can be described by the closing traction acting on each of the crack faces.^[11]

Modeling of the Fracture Process Zone of Concrete

In order to determine the fracture process zone in concrete, the meso scale approach is used. The meso structure of the concrete is assumed to have the stiff aggregates into the soft matrix and this matrix and the aggregates are separated by the weak interfaces. This meso structure of the concrete is representative of the energy released by the fracture process of the concrete. Size of the Fracture Process Zone of the concrete can also be determined from the strength and the fracture energies obtained from the different concrete specimen. By doing this, the rough idea of the size and shape of the fracture process zone can be obtained. In meso scale approach for the concrete, the interfacial transition zone, aggregates and matrix is modeled as separate phases for the non-linear analysis of the concrete. The average of the finite width of the process zone. [12] The meso-scale approach is also experimented on the three point bend test specimen and the size effect on the fracture process zone could be determined. From the size effect, it could be concluded

that the size effect of the fracture process zone is independent of the notched specimen and dependent on the un-notched specimen. The larger specimen size implies the longer region of the dissipation energy. In this way the size of the fracture process zone differs for the notched and unnotched specimen of concrete. [13]

Determination of the Fracture Process Zone: The Experimental Approach

The deformation in the concrete is highly localized and the strain produced upon the application of the load cannot be measure accurately with the strain gauges and optical methods. Also due to this, the size of the fracture process zone cannot be precisely ascertained. A narrow zone containing the microcracks develops which is responsible for the crack initiation and propagation. This zone can be measured with the reference to the strain localization but in order to do that, the strain is limited over its gauge length and also the interferometry method does not have the sensitivity to measure the FPZ accurately with strain. The accurate size of the FPZ can be recorded with the help of LASER interferometry using the coherent light being the concrete specimen under the tensile loading. The coherent light from the source is imparted to the concrete tensile specimen when loaded and the resulting fringe pattern can be observed. The experimental setup consists of testing the concrete tensile specimens either notched or unnotched. On experimenting to the notched specimen, the fringes are observed mutually closer in the notch region indicating the increase in corresponding strain and at the crack tip or notch tip, the fringes are dense and cannot be separated. The contour lines of the close strain values are indicative of the well-defined process zone in the specimen. It can be stated that the FPZ is largely spread and related to the strains produced. Also the fracture energy can be calculated on the basis of the stress-strain curve and the dimension of the FPZ in concrete specimen.^[14]

Nondestructive Methods to Determine the Fracture Process Zone in Concrete

In order to determine the FPZ of the concrete specimen, certain nondestructive laboratory methods have been evolved such as, Acoustic Emission (AE), Computer Vision, Digital Speckle Pattern Interferometry (DSPI) and X-ray Micro Tomography (XMT). Each of the method can give the data based on its functionality. AE method gives the characteristics of the micro-cracks in the concrete upon loading. The analysis of the specimen surface can be carried out using DSPI technique as the resolution in this technique is detailed and the

cracks of 0.25 mm can be detected. Computer vision method is used for measuring the crack openings for the multiple crack development. Various NDT methods are essential to identify the growth of the micro cracks in concrete.

Acoustic Emission is used to determine the micro cracking in the FPZ, dislocations and other changes which are not reversible in the concrete on loading. This method of the stress wave propagation can be used to obtain the micro mechanical behavior of the stressed material and to evaluate the extent of damage in the material. Micro cracks are the source of acoustic emissions and micro crack localization indicates the presence of the non-linear FPZ in concrete. AE technique has certain limitations such that the surface micro cracks cannot be detected. To overcome this difficulty, the method named electronic speckle pattern interferometry is used which gives the detailed resolution of the surface micro crack. As ESPI is the extension of the AE technique, the principle of fringe formation at the location of the crack is used. Fringe patterns produced after the commencement of the test, are observed for the discontinuity in the patterns and the deviation or any discontinuity directly implies the presence of the micro crack along with the FPZ. The method of Electronic Interferometry is highly sensitive to the discontinuities in a specimen caused by micro cracks. Damage evolution using interferometry is relative to the crack distribution, localization and critical crack propagation. The data obtained from both the methods, AE method and ESPI method, can be correlated for the crack localization.

To determine the internal structure of the material, the method named X-ray microtomography is used from the X-ray absorptivity of the concrete specimen. On scanning of the material, the data obtained in the form of the series of images representing the series of the slices through the specimen. At the different load levels, the data of the scan is obtained for the specimen. Each cross sectional slice is analyzed for the total length of the crack and then the crack lengths for different slices are summed up to get the crack area. From the results of XMT, the internal crack growth in the specimen can be clearly observed indicating the presence of FPZ. The use of XMT is limited to the small samples so that the results obtained are qualified and reliable.

The full scale details of the crack development are crucial for understanding the complex fracture behavior of the concrete like material. Computer Vision method makes the use of the brightness intensity of the images of the specimen surface and can be used to get the full

field deformation maps. The method has apparently no limitations as other methods have. Digital Image Correlation (DIC) can deal with the multiple cracks and the strain gauges used in the method are not attached to the specimen so that the readings are not deviated by the crack development. The technique is used for the large scale specimen giving the full field data for the crack growth. ^[15]

Conclusions

From the above review it can be concluded that the fracture process zone in concrete makes concrete a different material than the other brittle materials. Also the role of FPZ in concrete can be considered crucial and contributes to the fracture characteristics of concrete. Fracture toughness of concrete is also influenced by the FPZ in concrete. Aggregates being an important constituent also affect the fracture process zone and fracture energy. The techniques used to determine the size of the FPZ were Acoustic Emission technique and determines the localization of the micro cracking. These micro cracks along with the FPZ are responsible for the failure of the concrete in tension. The size of the fracture process zone can be determined by the concept of meso-scale approach on the concrete specimen. The fracture process zone size could be determined for the notched and unnotched concrete specimen and found different. The fracture energy will be dissipated more in the case of larger specimen size. The amount of the dissipated energy will be the indicative of the finite width of the fracture process zone. The width of the FPZ can be determined experimentally by using LASER interferometry on the concrete tensile specimen. The contour lines of the strains produced are indicative of the presence of the FPZ in concrete like material. Also, other non-linear models like Hillerborg's fictitious crack model and crack band model is used for the concrete structures in order to determine the fracture process zone. At last, the various non destructive techniques can be effectively used to determine the fracture processes taking place in the concrete along with the extent of the process zone. The non destructive testing methods are having their own limitations and also these limitations can be overcome by introducing the advanced methods such as Digital Image Correlation (DCI) and X-ray tomography. These techniques are capable of giving the whole field data for the crack growth in concrete specimen. These NDT techniques are capable of detecting the micro level damage crack localization phenomena which is responsible for the generation of the fracture process zone of quasi-brittle material like concrete.

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Printed at : The Maharaja Sayajirao University of Baroda Press (Sadhana Press), Near Palace Gate, Palace Road, Vadodara - 390 001.

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(To be published in the first issue every year after the last day of February)

FORM IV

(See Rule 8)

1.	Place of the Publication	:	The Maharaja Sayajirao University of Baroda The Maharaja Sayajirao University of Baroda, Vadodara - 390 002.
2.	Periodicity of its Publication	:	Science & Technology two times in a year.
3.	Printer's Name	:	Shri Jatin H. Somani
	Nationality	:	Indian
	Address	:	Manager, The Maharaja Sayajirao University of Baroda Press (Sadhana Press), Vadodara - 390 001.
4.	Publisher's Name	:	Prof. C. N. Murthy
	Nationality	:	Indian
	Address	:	The Maharaja Sayajirao University of Baroda Vadodara - 390 002.
5.	Editor's Name	:	Prof. C. N. Murthy
	Nationality	:	Indian
	Address	:	The Maharaja Sayajirao University of Baroda Vadodara - 390 002.
6.	Names & Addresses of Individuals who own the newspaper and partners or shareholders holding more than one percent of the total capital	:	The Maharaja Sayajirao University of Baroda Vadodara - 390 002.

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