

Chapter: 3

Material and Methodology

3.1. Brief Overview of the Methodological Approach

This subsection outlines the methodological approach utilized in the study, strategically designed to systematically meet the research objectives. It begins with waste profiling at various dumpsite depths, focusing on the geotechnical and chemical analysis of aged municipal solid waste (legacy waste) and the retrieved fines post-biomining. Following this, the experimental program involves sample preparation and testing in adherence to ASTM standards. Initial tests on untreated MSW fines (MSWF) cover specific gravity, Atterberg limits, pH, sieve analysis, and compaction tests. The samples are then subjected to biopolymer treatment using a wet mixing methodology, preparing them for advanced testing, including triaxial and unconfined compression tests. This stage is crucial for assessing both the strength and long-term durability of the treated samples. Additionally, the composite behaviour of the biopolymers is evaluated to understand their interaction with MSWF. To realize the practical field application of Municipal Solid Waste Fines and the utilization of treated MSWF, various civil engineering components such as embankments and subgrades are designed and tested under multiple conditions. This comprehensive methodological approach ensures that the

study thoroughly covers all necessary aspects, from basic characterization to advanced analysis of treated MSW fines, along with their practical applications in civil engineering.

3.2. Material Municipal Solid Waste Fines (MSWF)

Municipal Solid Waste Fines (MSWF) used in this study were collected from a dumpsite located in Varanasi, India. These fines are the by-product of the sorting and processing of municipal solid waste, consisting of particles smaller than a specified size, typically less than 4.75 mm. The MSWF was characterized for its physical and chemical properties, including particle size distribution, moisture content, and organic matter content, using standard testing procedures. Prior to their use in experimental setups, the MSWF underwent a preliminary drying process to standardize the moisture content, ensuring consistency across all samples for accurate comparison of results.

3.2.1. Site Description of Aged-MSW Collection

As discussed, aged municipal Solid Waste sample for this study was collected from an old, abandoned dumpsite in Sarriya, Varanasi, India, as depicted in Figure 11: (a) Satellite Overview of Dumpsite with Indicated Sampling Locations (b) View of Legacy Waste Dumpsite from Adjacent Bridge (c,d) Contaminated Water Channels from Adjacent Settlements Traversing the Dumpsite.

. This dumpsite, located in close proximity to the Varuna River, is adjacent to a slum housing around 10,000 residents (Goswami and Basak 2021). The site's location near the river raises significant environmental concerns, particularly due to the leaching of contaminants into the river, which poses health risks to the local population. Figure 11: (a) Satellite Overview of Dumpsite with Indicated Sampling Locations (b) View of Legacy Waste Dumpsite from Adjacent Bridge (c,d) Contaminated Water Channels from Adjacent Settlements Traversing the Dumpsite.

Figure 11 b and c illustrate the flow of community wastewater through the dumpsite, highlighting the potential for contaminant transport into the river and the surrounding environment. The dumpsite's proximity to the Varuna River is a crucial aspect, as it underscores the need for

sustainable remediation, particularly due to the potential environmental risks associated with water bodies. The geographical positioning of the dumpsite, along with its environmental context, forms the basis for understanding the critical need for responsible waste management in the area. The dumpsite, with an average height of 20-30 meters, active since 2014-15. This places the age of the waste under study between 1 to 8 years. The geographical coordinates of the dumpsite are 25°20'27.9"N 83°01'25.4"E, with its satellite imagery shown in Figure 11: (a) Satellite Overview of Dumpsite with Indicated Sampling Locations (b) View of Legacy Waste Dumpsite from Adjacent Bridge (c,d) Contaminated Water Channels from Adjacent Settlements Traversing the Dumpsite. and a view from a nearby bridge presented in Figure 11 b.

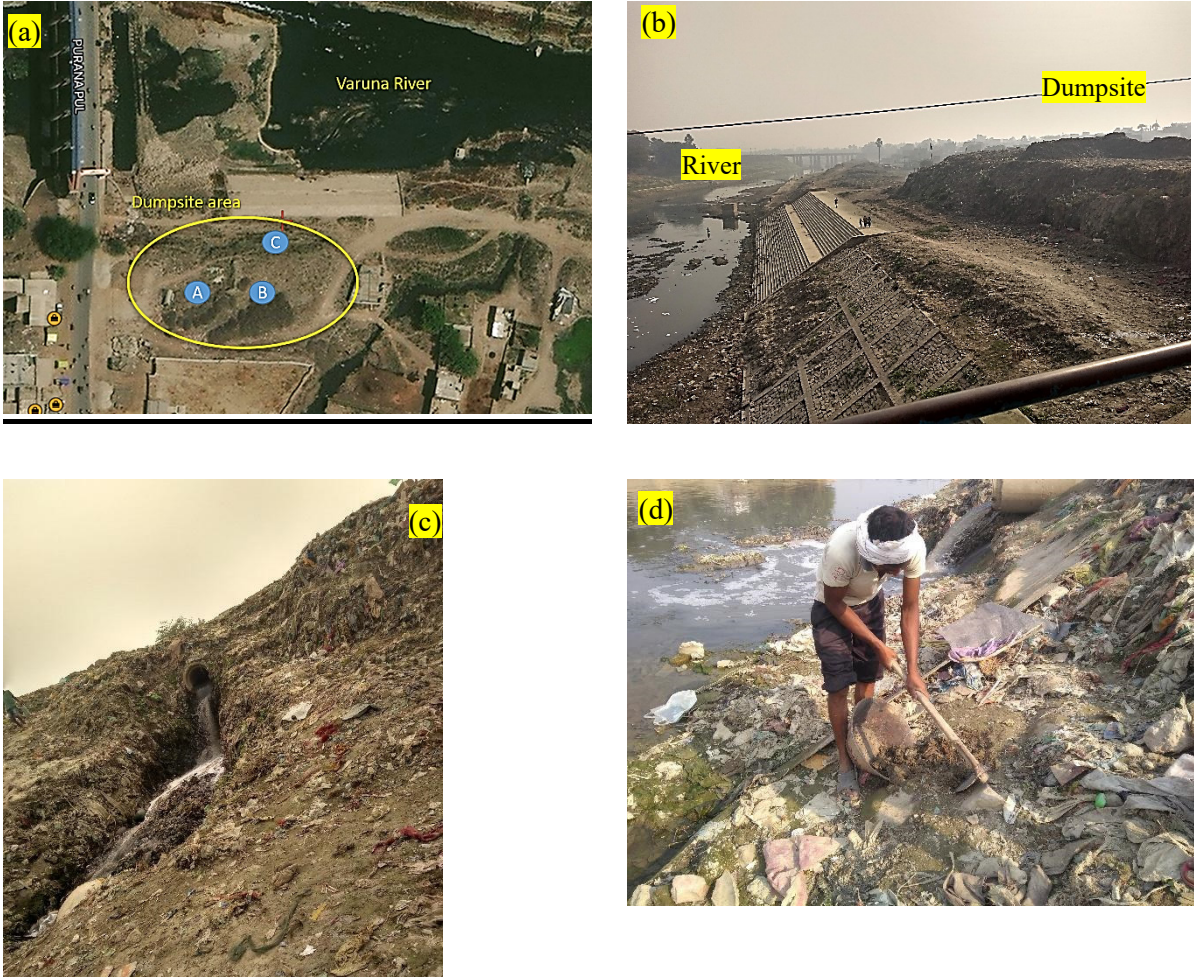


Figure 11: (a) Satellite Overview of Dumpsite with Indicated Sampling Locations (b) View of Legacy Waste Dumpsite from Adjacent Bridge (c,d) Contaminated Water Channels from Adjacent Settlements Traversing the Dumpsite.

3.2.2. Sample Collection and Transport

The collection process for Aged Municipal Solid Waste samples was meticulously designed to evaluate the properties of Municipal Solid Waste Fines (MSWF) extracted from mined aged MSW. Collection process began with the excavation of aged MSW from a newly dug trench at the dumpsite, as depicted in Figure 12 a. Samples were meticulously gathered from depths of 0, 2, 5, and 10 meters below the surface at three distinct points, marked as A, B, and C in Figure 11: (a) Satellite Overview of Dumpsite with Indicated Sampling Locations (b) View of Legacy Waste Dumpsite from Adjacent Bridge (c,d) Contaminated Water Channels from Adjacent Settlements Traversing the Dumpsite.

a. At each depth, around 100-150 kg of legacy waste was collected from each point, securely bagged, and transported to the laboratory for analysis, as shown in Figure 12 b. This method ensured that the samples were representative of the varying depths and conditions of the waste accumulated over the years.

3.2.3. Processing of MSWF

The primary work in this study is done over Municipal Solid Waste Fines (MSWF), which is extracted from the above-mentioned dumpsite. In the laboratory, the municipal solid waste samples from various depths were first dried under sunlight. They were then mixed to achieve a homogenized sample representing each specific depth. Following this, the samples were segregated and sieved to ascertain their compositional makeup, as illustrated in Figure 12 c. To specifically isolate the MSWF, which resembles soil-like material, the aged MSW was passed through an ASTM No-4 sieve (4.75 mm). The material that successfully passed through the sieve was then collected in labelled bags for detailed experimental analysis, as shown in Figure 12 d.

Upon completion of the depth-wise profiling of the MSWF, the extracted samples from varying depths are amalgamated in nearly equal proportions. This procedure is executed to

ensure that the experimental investigations reflect a comprehensive view of the dump's overall properties. By integrating samples from each depth, the study aims to encapsulate the complete spectrum of MSWF characteristics, thereby providing a holistic insight into how biopolymers interact with MSWF under varied conditions. This balanced and methodical approach enhances the reliability and applicability of the experimental outcomes, ultimately contributing to a more effective and informed application of biopolymers in MSWF management and stabilization.



Figure 12: (a) Section of Excavated Dumpsite (b) Collection of Samples in Bags (c) Sorting Process of Legacy Waste (d) Sieved and Isolated Municipal Solid Waste Fines

3.3. Biopolymers:

Biopolymers have garnered significant attention for their potential in stabilizing Municipal Solid Waste Fines (MSWF), a critical component in sustainable waste management. These naturally derived polymers offer a range of beneficial properties, including high water retention, excellent binding characteristics, affordability, and widespread availability. The utilization of biopolymers in MSWF stabilization not only contributes to reducing landfill volume but also enhances the mechanical stability of waste fines, thereby facilitating their use in various applications.

3.3.1. Xanthan Gum (XG)

Xanthan gum, a polysaccharide derived from the fermentation of sugars by *Xanthomonas campestris*, is one of the biopolymers chosen for this study. Recognized by its chemical formula $C_{35}H_{49}O_{29}$, xanthan gum exhibits remarkable viscosity, even at low concentrations, making it an effective thickening agent in various industries, including food and cosmetics. Its unique properties extend to soil stabilization; when xanthan gum hydrogel is introduced to soil, it significantly enhances the soil's matrix strength by binding soil particles together. This is primarily due to its high emulsifying nature, allowing it to form a hydrogel readily upon mixing with water, even at ambient temperatures. The hydrophilic anionic surface of xanthan gum facilitates strong gelation by interacting with cations in the soil, leading to improved structural integrity and water retention within the soil matrix .

3.3.2. Agar Gum (AG)

Agar gum, another focal biopolymer in this research procured from local market. Agar extracted from seaweed and is renowned for its gelling, thickening, and emulsifying properties. Its utility spans across various sectors, including the food and cosmetic industries, due to its ability to form a stiff gel upon cooling. This biopolymer dissolves in water at approximately 85 °C and transitions into a gel as the temperature decreases to around 37 °C.

The significant temperature range for this transition ensures that once agar is mixed into the soil, it remains solidified, thereby enhancing the soil's mechanical strength. Agar gum comprises two primary components: agarose and agaropectin. Agarose, in particular, plays a crucial role in soil stabilization by gelling soil particles, which contributes to an increase in the overall mechanical strength of the soil. A distinctive feature of agar is its ability to form a cross-linked network, preventing dispersion in water. Despite swelling upon heating, agar's hydrophobic nature ensures its durability within the soil matrix, maintaining its structural integrity under various conditions.

Figure 13 shows the changes in the physical forms of biopolymers from hydration to thickening and drying. While xanthan gum produces a highly viscous gel with water, agar gum produces a stiff gel bed when its temperature reduces to 32 to 42 °C. Both xanthan gum and agar gum were procured from the local vendor, and their properties were obtained from the standard specifications provided by the manufacturer of these biopolymers, as given in Table 5.

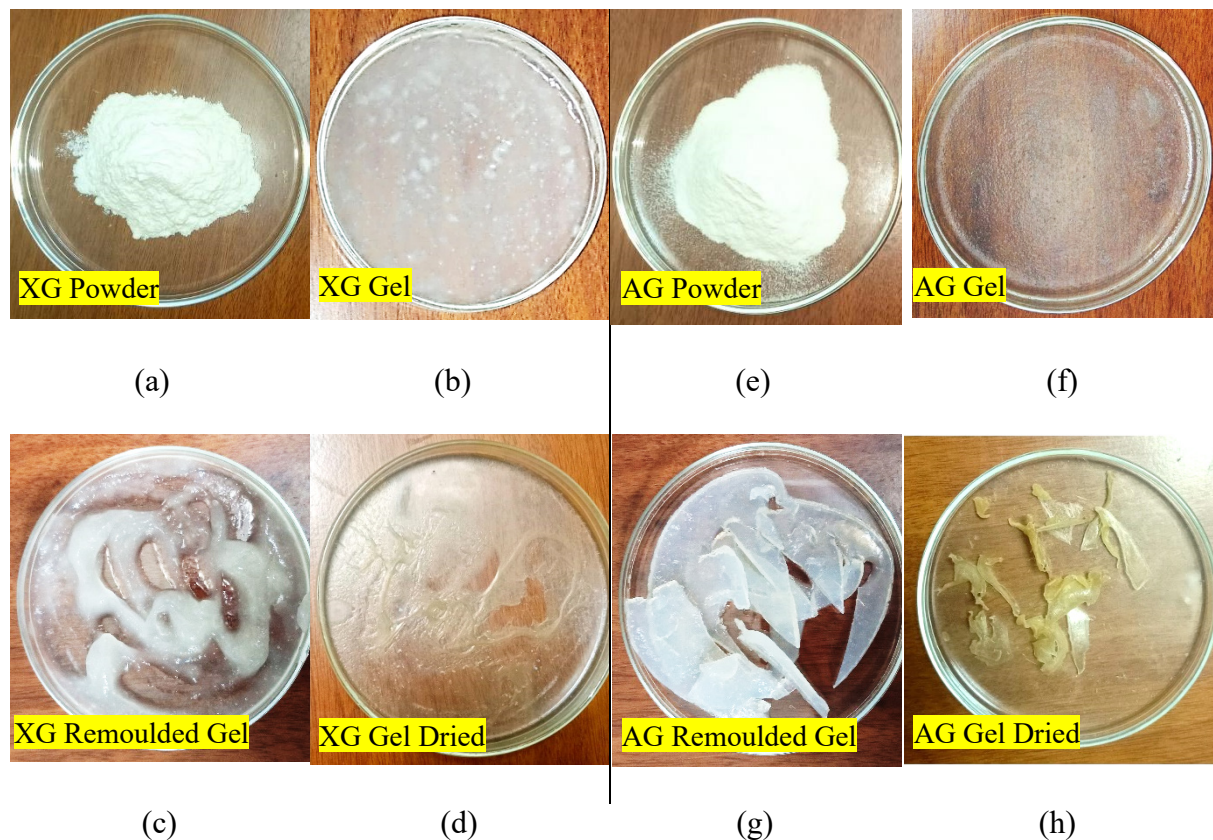


Figure 13: Xanthan gum and agar gum gel in powder form, hydrated (wet), remoulded and dry conditions.

Table 5: Properties of biopolymers

Properties	Agar Gum	Xanthan Gum
Molecular weight (g/mol)	336.33	933.74
Ash content	3%	6.5-16%
Gelling temperature	Below 40 °C	Room temperature
pH	6-7.5 (10% suspension)	in 6-8 (1% in suspension)
Melting range	88-90 °C	Room temperature
Particle size	100-200 mesh	80 mesh
Viscosity	10-40 cps	--
Gel strength	900 g/cm ²	--
Loss on drying	Max 20%	Max 15%
Heavy metal	--	Max 0.001%

3.4. Methodology

The methodology combines material collection, assessment of basic properties of materials, experimental assessment of the material properties treated with selected biopolymers, and then demonstration of various civil engineering applications of the treated and non-treated materials. The processing of material (MSWF) and properties of selected biopolymers have been discussed above; however, the detailed process adopted for performing each experiment is discussed in this section. For simplification, the whole methodology with the experimentation details is shown in Figure 15. While identifying gaps and objectives in the previous chapter, the section explaining the thesis structure also detailed the procedure of experimental investigation, which includes physical analysis, chemical & microstructure analysis, geotechnical analysis, and analytical determination are as follows:

3.4.1. Physical Analysis:

To find the physical properties and chemical characteristics, the following tests are conducted:

I. Determination of MSW Composition: ASTM D5231-92 (2016) is followed to find the percentage of different fractions of materials present in MSW. To determine the composition of Municipal Solid Waste (MSW), the procedure outlined in ASTM D5231-92 (2016) is followed. This standard method involves collecting representative samples of MSW, typically around 200 kg, from various points across the dumpsite to ensure a comprehensive representation. These samples are thoroughly mixed to achieve uniformity, and a sub-sample of approximately 50 kg is taken for detailed analysis.

The sub-sample is manually sorted into major categories such as plastics, metals, glass, paper, organic matter, and miscellaneous materials, which are further divided into sub-categories for detailed analysis. Each sorted fraction is weighed with a precision balance, and the weights are meticulously recorded. The percentage of each fraction is calculated by dividing its weight by the total weight of the sub-sample and multiplying by 100. Quality control measures, including random rechecking and consistency checks, ensure the accuracy and reliability of the data. This method provides an accurate determination of the different material fractions present in MSW, aiding in effective waste management and recycling strategies.

II. Determination of organic content: ASTM D2974 is followed to determine organic content present in MSWF. To determine the organic content in Municipal Solid Waste Fines (MSWF), the procedure outlined in ASTM D2974 is followed. This involves collecting and preparing a representative sample of MSWF by drying it in an oven at a specified temperature until a constant weight is achieved. The dry sample is then weighed to obtain its initial dry weight. Next, the sample is placed in a furnace and heated to approximately 440°C for a specified duration, which burns off the organic matter. The 440°C temperature was chosen based on thermogravimetric analysis (TGA), ensuring optimal organic matter decomposition while minimizing energy consumption and structural loss. Lower temperatures lead to

incomplete combustion, while higher ones cause unnecessary mineral transformation (Gerassimidou et al. 2020; Díaz et al. 2021). After ignition, the sample is cooled in a desiccator to prevent moisture absorption and then weighed again. The loss in weight due to ignition represents the organic content in the MSWF. This method ensures accurate determination of organic content.

3.4.2. Chemical & Microstructure Analysis

The study incorporated a comprehensive chemical analysis of Municipal Solid Waste Fines (MSWF) to understand their composition and potential contaminants. This involved using advanced techniques like Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray analysis (EDX) to investigate the microstructure and to analyze the elemental composition of MSWF.

I. Scanning Electron Microscopy (SEM): SEM provides detailed images of the MSWF's surface morphology and microstructure at high magnification. By examining the MSWF samples under SEM, researchers can observe the physical characteristics, particle size, and distribution, as well as any structural anomalies or features that might influence the material's performance in engineering applications (Ref Figure 14: (a) A photograph of UCS testing machine, (b) A photograph of high-resolution scanning electron microscope (SUPRA 40, Zeiss 4.0))

II. Energy Dispersive X-ray Analysis (EDX): EDX, used in conjunction with SEM, allows for the identification and quantification of the elemental composition of the MSWF. This technique detects X-rays emitted from the sample during bombardment by the SEM's electron beam, enabling the determination of the presence and concentration of various elements (Ref Figure 14 b).

III. Leachate Analysis: Leachate analysis was conducted to assess the heavy metal content released when water flows through both stabilized and non-stabilized Municipal Solid

Waste Fines (MSWF). This process helps in understanding the environmental impact and the effectiveness of stabilization treatments. The analysis was performed using Atomic Absorption Spectroscopy (AAS) to detect and quantify the presence of heavy metals in the leachate. For preparing the MSWF leachate, 1 gram of the MSWF sample was taken and subjected to digestion with 15 mL of nitric acid (HNO_3) and 25 mL of perchloric acid (HClO_4). The sample was heated to facilitate the digestion process, breaking down the matrix and releasing the heavy metals into the solution. After digestion, the extract was separated using filter paper to remove any solid residues.

The clear filtrate obtained was then analyzed for heavy metal content using the Atomic Absorption Spectrometer. This technique allows for precise measurement of metal concentrations, ensuring accurate assessment of the potential environmental hazards posed by the leachate from both treated and untreated MSWF. The results of the heavy metal analysis are detailed in Table 11, providing insights into the effectiveness of the biopolymer stabilization process in reducing the mobility of hazardous metals in the waste.

3.4.3. Geotechnical Analysis

Geotechnical experiments which are conducted in the study are as follows:

I. Particle Size Distribution: To determine the gradation of soil particles in Municipal Solid Waste Fines (MSWF), the procedure outlined in ASTM D6913 (for coarse-grained soils) and ASTM D7928 (for fine-grained soils) is followed. A representative sample is dried and sieved through a series of standard sieves with progressively smaller openings. Wet sieving is adopted for samples with a high fine particle content. The sample is washed through the No. 200 (75- μm) sieve to separate fine particles, which are then analysed using sedimentation techniques. The mass retained on each sieve and the mass of fines are recorded to determine the particle size distribution curve, which provides insights into the soil's gradation and its suitability for various engineering applications.

II. Specific Gravity: The specific gravity of MSWF is determined using the ASTM D854 standard. This test assesses the density of the MSWF particles relative to water. A dry sample of the MSWF is placed in a pycnometer, and its mass is measured. Water is then added to the pycnometer, and the combined mass of the sample and water is recorded. The pycnometer is filled with water to a specified volume, and the mass is measured again. The specific gravity is calculated using these measurements, providing crucial information about the density and composition of the MSWF, which is essential for various geotechnical analyses.

III. Consistency Limits: To understand the soil's behavior at different moisture contents, the liquid limit (LL) and plastic limit (PL) of MSWF are determined using ASTM D4318. The liquid limit is found by placing a portion of the soil in a standard cup and cutting a groove in the soil. The cup is then repeatedly dropped until the groove closes over a specified distance. The moisture content at which this occurs is the liquid limit. For the plastic limit, a soil sample is rolled into threads until they crumble at a certain diameter. The moisture content at this point is the plastic limit. These tests help in determining the soil's plasticity and its potential for deformation under various moisture conditions.

IV. Compaction Characteristics: The compaction characteristics of MSWF, including the Maximum Dry Density (MDD) and optimal moisture content, are determined using ASTM D698 (Standard Proctor Test) or ASTM D1557 (Modified Proctor Test). A representative sample is compacted in a mold using a standard hammer, with layers compacted to a specific number of blows. The moisture content is varied for different samples, and the dry density is calculated for each. The MDD and optimal moisture content are identified from the compaction curve, providing essential data for designing earthworks and assessing the soil's load-bearing capacity.

V. California Bearing Ratio (CBR): The California Bearing Ratio (CBR) test, conducted according to ASTM D1883, estimates the bearing capacity of MSWF. A

compacted sample is prepared at its optimal moisture content and subjected to penetration by a standard piston at a controlled rate. The force required to achieve specific penetrations is measured and compared to the force required for standard crushed stone. The CBR value is calculated as a percentage, indicating the supporting strength of the MSWF, which is crucial for designing road subgrades and pavement layers.

VI. Unconfined Compressive Strength (UCS): The UCS test, as per ASTM D2166, is performed by applying axial load to a cylindrical soil sample without any lateral confinement until failure (Ref Figure 14 a). This test measures the maximum axial stress that the sample can withstand, providing a direct estimate of the unconfined compressive strength of the soil. The UCS test is simple and quick, offering valuable information about the soil's behavior under compression, which is essential for assessing its suitability for construction applications where lateral confinement is minimal.



Figure 14: (a) A photograph of UCS testing machine, (b) A photograph of high-resolution scanning electron microscope (SUPRA 40, Zeiss 4.0)

VII. Unconsolidated Undrained Triaxial (UU): The UU triaxial test, according to ASTM D2850, involves encasing a cylindrical soil sample in a membrane, applying a confining pressure, and then increasing the axial stress until failure, all while keeping the sample in an undrained condition. This test provides a more comprehensive analysis of the shear strength by simulating in-situ stress conditions. The UU test measures both the deviatoric stress and the confining pressure, allowing for the determination of shear strength parameters such as cohesion and angle of internal friction. These parameters are crucial for evaluating the stability of soil in applications like slopes and retaining structures.

3.4.4. Analytical Determination

Various analytical tools such as ANOVA, RSM, FEM, and mechanistic-empirical approaches are employed in different sections of the study, each detailed in their respective chapters. ANOVA identifies influential parameters responsible for the higher strength of XG or AG-MSFW mix. RSM optimizes the mix of XG and AG when used together. FEM, using Plaxis 2D, designs embankments and foundations to analyse responses under various conditions. Similarly, IIT Pave employs the mechanistic-empirical approach to design pavements, optimizing both stabilized and untreated MSWF when used in the subgrade.

3.4.5. Sustainability Analysis

Four significant sustainability analyses were carried out in the study to understand and quantify the effect of the objectives. Below is a brief overview of these assessments; however, the detailed assessment is explained in the respective chapters.

I. Alignment with SDG Goals: Evaluated the project's alignment with Sustainable Development Goals (SDGs), focusing on SDG 9 (Industry, Innovation, and Infrastructure), SDG 11 (Sustainable Cities and Communities), SDG 12 (Responsible Consumption and Production), SDG 13 (Climate Action), and SDG 15 (Life on Land).

II. ESG Impact Analysis: Assessed Environmental, Social, and Governance (ESG) impacts by analyzing factors such as CO₂ emissions, resource use, energy consumption, health risks, community impact, and regulatory compliance. This analysis compared the impacts of cement stabilization versus biopolymer stabilization.

III. Carbon Footprint Analysis: Conducted a carbon footprint analysis to measure the CO₂ emissions associated with the stabilization processes. This involved comparing the emissions from biopolymer stabilization to those from traditional cement stabilization.

IV. Cost-Benefit Analysis: A cost-benefit analysis was performed to compare the financial aspects of cement and biopolymer stabilization. This included evaluating material costs, waste disposal savings, and overall cost savings.

3.5. Key Takeaways and Way Forward:

- The chapter outlined the specific biopolymers (e.g., Xanthan Gum, Agar Gum) and MSWF used in the study, detailing their properties and preparation processes.
- A comprehensive explanation of the experimental procedures was provided, including soil stabilization techniques and testing protocols to assess the geotechnical properties and durability of treated MSWF.
- The next chapter, Depth-wise Characterization of MSWF, will build on these methods by providing detailed analyses of the geotechnical properties of MSWF at various depths. This characterization is crucial for understanding how biopolymer treatment impacts different layers of legacy waste fines and sets the stage for evaluating the effectiveness of the stabilization techniques.

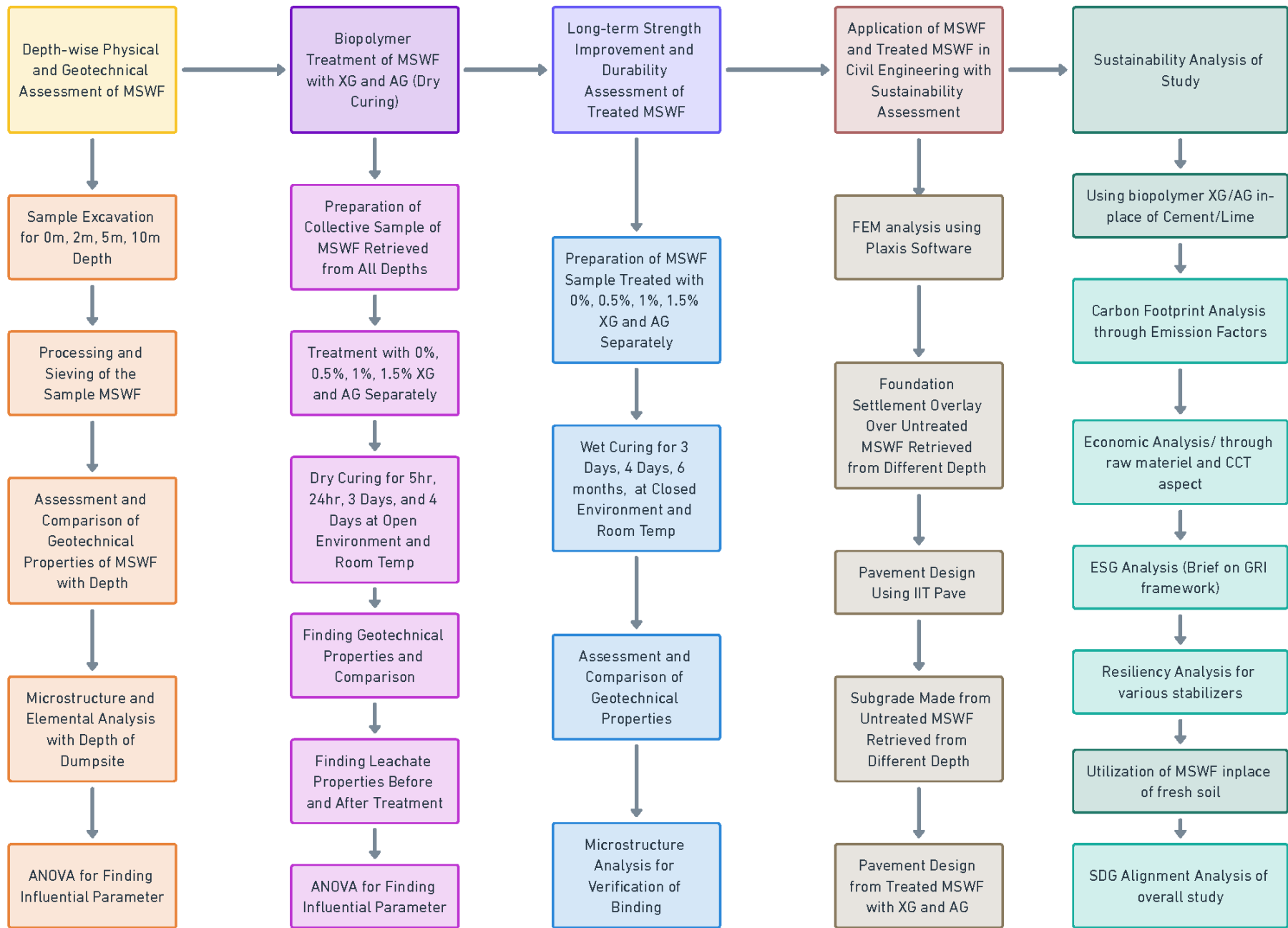


Figure 15: Detailed scheme of the chapters