

**EXPLORING VARIOUS FILLERS FOR THE
DEVELOPMENT OF ECO-FRIENDLY BRAKE-PADS**

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TRIBOLOGY (CART)**

(FORMERLY ITMMEC)

INDIAN INSTITUTE OF TECHNOLOGY DELHI

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by

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CERTIFICATE

This is to certify that the thesis entitled “**Exploring various fillers for the development of eco-friendly brake-pads**” being submitted by **Mr. Chauhan Vanvirsinh Jagatsinh** to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy**, is a record of bonafide research work carried out by him. **Mr. Chauhan Vanvirsinh Jagatsinh** has worked under my guidance and supervision and has fulfilled the requirements for the submission of the thesis. The results contained in this thesis have not been submitted in part or in full to any other University or Institute for the award of any degree or diploma.

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Last but not least; I am surrendering to the Lord Almighty and in- numerous kind-hearted members of this society whose positive waves of love directly or indirectly boosting my spirits.

A handwritten signature in black ink, reading "Chauhan Vanvirsinh Jagatsinh". The signature is written in a cursive style with a horizontal line underneath the name.

Chauhan Vanvirsinh Jagatsinh

ABSTRACT

Today's era is of eco-friendly materials. Copper around 10 wt. % is added invariably in friction materials (FMs) because of its unmatched contribution to the performance of FMs. However, recently it is proven as an aquatic hazard, and global efforts are being focused on searching for an alternative, eco-friendly, and equally or more efficient than Copper. Copper, being a multi-purpose additive, its replacement is a Herculean task. Very recently, it has been claimed that fine particles of stainless steel of typical kind have shown full success in replacing Copper in FMs.

It is always desirable to find an alternative, which would be non-metallic, leading to a reduction in weight apart from fulfilling all other criteria for Copper- replacement.

The current research work was hence focussed on finding the non-metallic alternative to Copper in FMs.

Another issue, which is inadequately addressed in the literature, is the efforts to reduce the noise-vibration (NV) tendency of brake-pads by selecting appropriate ingredients in the formulation. Noise emission while braking is a serious concern, particularly in automotive industries, which can lead to high warranty costs and the customers' dissatisfied feedback. The ingredients need to be selected in such a way that they should not adversely affect tribo-performance, which is the most critical performance parameter.

Another critical issue that is never addressed in the open literature by the researchers is about the design of formulation of the underlayer. In commercial brake-pads, an underlayer is added to control NV characteristics of FMs. Underlayer (UD) is added for the following primary functions.

- reduces noise and vibration to a great extent
- acts as a thermal barrier to avoid the spongy brake phenomenon while braking
- assures high shear strength with a back-plate

- reduces the overall cost of the brake-pads

It is important to design the formulation of UD in such a way that its efficiency can be increased, leading to brake-pads with superior performance. Ingredients having a well-proven capability of noise-vibration (NV) can be investigated for performance enhancement. Interestingly these ingredients will be present only in UD and hence, will not affect the tribo-performance.

Another issue about FMs, which is not yet addressed adequately, is filler-matrix adhesion. Phenolic matrix has less adhesion for certain types of fillers such as abrasives, solid lubricants, metals, etc., and hence such ingredients are easily dug out during braking leading to high wear. In this thesis work, this issue was addressed by selecting abrasive particles and enhancing adhesion with matrix, and then study the effect on all performance properties.

With this in view, this research was focused on the following themes.

- To find a non-metallic and eco-friendly alternative, preferably polymeric for Copper in FMs.
- To investigate the influence of selected ingredients for their efficiency to reduce NV-related issues and simultaneous effect on tribo-performance.
- To explore various NV reducing ingredients in underlayer and to find its influence on overall NV related performance of brake-pads.
- To enhance filler (alumina abrasive particles) -matrix adhesion by siloxane treatment and to investigate its effect on wear performance and also on other performance properties.

The thesis work was divided into seven chapters as follows.

CHAPTER 1 INTRODUCTION

CHAPTER 2 MATERIALS AND METHODOLOGY

CHAPTER 3 EXPLORATION OF PARTICULATE PANI (POLYANILINE) AS A NON-METALLIC SUBSTITUENT FOR COPPER IN BRAKE-PADS

CHAPTER 4 SILOXANE FUNCTIONALIZATION OF ALUMINA PARTICLES FOR PERFORMANCE ENHANCEMENT OF BRAKE-PADS

CHAPTER 5 INFLUENCE OF VARIOUS TYPES OF RUBBER PARTICLES ON THE PERFORMANCE OF BRAKE-PADS

CHAPTER 6 INFLUENCE OF SELECTED INGREDIENTS IN UNDERLAYER ON THE PERFORMANCE OF BRAKE-PADS

CHAPTER 7 CONCLUSIONS AND SCOPE FOR FUTURE WORK

Chapter 1 includes an introduction to the braking system and various classes of FMs. It also includes a literature survey on key areas leading to the research gaps and motivation to the work. The chapter concludes with the objectives of the work and implementation overview.

Chapter 2 elaborates the details of ingredients used to develop various series of brake-pads apart from the fabrication procedure of brake-pads. It also describes the experimental techniques for performance evaluation such as physical, mechanical, chemical, tribological, and NV characterization of brake-pads.

Chapter 3 First part focuses on the synthesizing particles of PANI (Polyaniline) in the laboratory and then exploring its potential for replacement of Copper in brake pads. Particles of PANI were added in 10 wt. % amount as a theme ingredient keeping all parent ingredients identical. Performance was also compared with brake-pads containing 10 wt. % commercially available PANI and also with brake-pads with 10 % Copper particles. The Inclusion of PANI led to very promising results as a substituent for Copper entirely. It was also tried to explore if it shows synergism with stainless steel particles. All PANI-based pads proved superior to Copper- based and SS-based (without PANI) pads. These efforts led to still better results in Part B of the chapter.

Chapter 4 explored the potential of siloxane treatment to alumina particles to enhance adhesion with resin. A series of brake-pads with 2 % alumina particles (treated and untreated)

was developed apart from one without alumina particles. The results showed that pads with treated particles excelled in all performance properties.

Chapter 5 pertains to the investigations on eight types of Copper-free brake-pads using various rubbers as a theme ingredient (9 vol.%). Five types of particles of NBR, two types of SBR, and also with commercially used crumb rubber with varying particle sizes and structures were used, keeping all the parent formulations identical. An additional type of brake-pads without rubber particles was also developed for comparison. The performance properties with special reference to NV were evaluated and analyzed with a conclusion that rubber particles not only improved NV performance but tribo-performance also. Liner structures performed better than cross-linked ones.

Chapter 6 focussed on designing underlayers using four types of noise controlling and vibration damping ingredients (particles of cork and crumb rubber apart from jute fibers and aramid pulp) in various amounts keeping parent ingredients of underlayer fixed. The brake-pads were then evaluated NV performance. The unique ingredients in the underlayer proved effective to multiple extents for improving NV performance. Cork and jute fiber-based underlayer had a capability to suppress high-frequency squeal, and hence an enhanced NV performance. Using natural ingredients in under-layer like cork, jute fiber, etc., a low-cost, light-weight, and eco-friendly under-layer can be designed to enhance the NV performance of brake pads without compromising the tribo-performance.

Chapter 7 is for conclusions on overall thesis work and scope for future possibilities in this area.

सार

आज का युग पर्यावरण के अनुकूल सामग्री का है। घर्षण सामग्री के प्रदर्शन में इसके अतुलनीय योगदान के कारण तांबे को लगभग १० वजन प्रतिशत घर्षण सामग्री में जोड़ा जाता है। हालांकि, हाल ही में यह एक जलीय खतरे के रूप में सिद्ध हुआ है, और वैश्विक प्रयासों को तांबे की तुलना में एक वैकल्पिक, पर्यावरण के अनुकूल और समान या अधिक कुशल खोजने पर ध्यान केंद्रित किया जा रहा है। कॉपर, एक बहुउद्देश्यीय योज्य होने के कारण, इसका प्रतिस्थापन एक कठिन कार्य है। हाल ही में, यह दावा किया गया है कि विशिष्ट प्रकार के स्टेनलेस-स्टील के महीन कणों ने तांबे को घर्षण सामग्री में बदलने में पूरी सफलता दिखाई है। ऐसा विकल्प खोजना हमेशा वांछनीय होता है, जो गैर-धातु हो, जिससे तांबे के प्रतिस्थापन के लिए अन्य सभी मानदंडों को पूरा करने के अलावा वजन में कमी आए।

इसलिए वर्तमान शोध कार्य कॉपर संक्रमण सामग्री के गैर-धातु विकल्प को खोजने पर केंद्रित था।

एक अन्य मुद्दा, जिसे साहित्य में अपर्याप्त रूप से संबोधित किया गया है, वह है फॉर्मूलेशन में उपयुक्त अवयवों का चयन करके ब्रेक-पैड की शोर-कंपन प्रवृत्ति को कम करने का प्रयास। ब्रेक लगाते समय शोर उत्सर्जन एक गंभीर चिंता का विषय है, विशेष रूप से मोटर वाहन उद्योगों में, जिससे उच्च वारंटी लागत और ग्राहकों की असंतुष्ट प्रतिक्रिया मिल सकती है। अवयवों को इस तरह से चुना जाना चाहिए कि वे ट्राइबो-प्रदर्शन पर प्रतिकूल प्रभाव न डालें, जो कि सबसे महत्वपूर्ण प्रदर्शन मापदंड है।

एक और महत्वपूर्ण मुद्दा जिसे शोधकर्ताओं ने खुले साहित्य में कभी संबोधित नहीं किया है, वह है अंडरलेयर के निर्माण के डिजाइन के बारे में। वाणिज्यिक ब्रेक-पैड में, घर्षण सामग्री के शोर और कंपन विशेषताओं को नियंत्रित करने के लिए एक अंडरलेयर जोड़ा जाता है। निम्न प्राथमिक कार्यों के लिए अंडरलेयर जोड़ा जाता है।

- शोर और कंपन को काफी हद तक कम कर देता है।
- ब्रेक लगाने के दौरान स्पंजी ब्रेक की घटना से बचने के लिए थर्मल बैरियर के रूप में कार्य करता है।
- बैक-प्लेट के साथ उच्च कतरनी शक्ति का आश्वासन देता है।

- ब्रेक-पैड की कुल लागत को कम करता है।

अंडरलेयर के फॉर्मूलेशन को इस तरह से डिजाइन करना महत्वपूर्ण है कि इसकी दक्षता बढ़ाई जा सके, जिससे बेहतर प्रदर्शन वाले ब्रेक-पैड बन सकें। प्रदर्शन बढ़ाने के लिए शोर और कंपन की अच्छी तरह से सिद्ध क्षमता वाली सामग्री की जांच की जा सकती है। दिलचस्प बात यह है कि ये अवयव केवल अंडरलेयर में मौजूद होंगे और इसलिए ट्राइबो-प्रदर्शन को प्रभावित नहीं करेंगे।

घर्षण सामग्री के बारे में एक और मुद्दा, जिसे अभी तक पर्याप्त रूप से संबोधित नहीं किया गया है, फिलर मैट्रिक्स आसंजन है। फेनोलिक मैट्रिक्स में कुछ प्रकार के फिलर्स जैसे अपघर्षक, ठोस स्नेहक, धातु, आदि के लिए कम संस्करण होता है, और इसलिए ब्रेकिंग (घर्षण) के दौरान ऐसी सामग्री को आसानी से खोदा जाता है जिससे उच्च घिसाव की संभावना होती है। इस शोध प्रबंध (थीसिस) कार्य में, इस मुद्दे को अपघर्षक कणों का चयन करके और मैट्रिक्स के साथ आसंजन को बढ़ाकर संबोधित किया गया था, और फिर सभी प्रदर्शन गुणों पर प्रभाव का अध्ययन किया गया था।

इसी को दृष्टिगत रखते हुए, यह शोध कार्य निम्नलिखित विषयों पर केन्द्रित था।

- घर्षण सामग्री में तांबे के लिए एक गैर-धातु और पर्यावरण के अनुकूल विकल्प खोजने के लिए, अधिमानतः बहुलक।
- शोर और कंपन से संबंधित मुद्दों को कम करने और ट्राइबो-प्रदर्शन पर एक साथ प्रभाव को कम करने के लिए उनकी दक्षता के लिए चयनित अवयवों के प्रभाव की जांच करना।
- अंडरलेयर में विभिन्न शोर और कंपन कम करने वाले अवयवों का पता लगाना और ब्रेक-पैड के समग्र शोर और कंपन से संबंधित प्रदर्शन पर इसके प्रभाव का पता लगाना।
- फिलर(एल्यूमिना अपघर्षक कण) -मैट्रिक्स आसंजन को बढ़ाने के लिए – सिलोक्सेन उपचार द्वारा आसंजन और घिसाव के प्रदर्शन और अन्य प्रदर्शन गुणों पर इसके प्रभाव की जांच करना।

शोध प्रबंध (थीसिस) कार्य को सात अध्यायों में विभाजित किया गया था।

अध्याय १ परिचय

अध्याय २ सामग्री और कार्यप्रणाली

अध्याय ३ ब्रेक-पैड में तांबे के लिए एक गैर-धातु विकल्प के रूप में पॉलीएनिलिन कणों की खोज

अध्याय ४ ब्रेक-पैड के प्रदर्शन में वृद्धि के लिए एल्यूमिना कणों का सिलोक्सेन कार्यात्मककरण

अध्याय ५ ब्रेक-पैड के प्रदर्शन पर विभिन्न प्रकार के रबर कणों का प्रभाव

अध्याय ६ ब्रेक-पैड के प्रदर्शन पर अंडरलेयर में चयनित अवयवों का प्रभाव

अध्याय ७ निष्कर्ष और भविष्य के काम की गुंजाइश

अध्याय १ में ब्रेकिंग सिस्टम और घर्षण सामग्री के विभिन्न वर्गों का परिचय शामिल है। इसमें प्रमुख क्षेत्रों पर एक साहित्य सर्वेक्षण भी शामिल है जो अनुसंधान अंतराल और कार्य के लिए प्रेरणा की ओर ले जाता है। अध्याय कार्य के उद्देश्यों और कार्यान्वयन सिंहावलोकन के साथ समाप्त होता है।

अध्याय २ ब्रेक-पैड की निर्माण प्रक्रिया के अलावा ब्रेक-पैड की विभिन्न श्रृंखलाओं को विकसित करने के लिए उपयोग की जाने वाली सामग्री के विवरण को विस्तृत करता है। यह प्रदर्शन मूल्यांकन के लिए प्रायोगिक तकनीकों जैसे भौतिक, यांत्रिक, रासायनिक, ट्राइबोलॉजीकल, और ब्रेक-पैड के शोर और कंपन लक्षण वर्णन का भी वर्णन करता है।

अध्याय ३ पहला भाग प्रयोगशाला में पॉलीएनिलिन के कणों को संश्लेषित करने और फिर ब्रेक-पैड में तांबे के प्रतिस्थापन के लिए इसकी क्षमता की खोज पर केंद्रित है। सभी मूल अवयवों को समान रखते हुए एक थीम घटक के रूप में १० वजन प्रतिशत राशि में पॉलीएनिलिन के कण जोड़े गए थे। प्रदर्शन की तुलना व्यावसायिक रूप से उपलब्ध पॉलीएनिलिन के १० भार/ वजन प्रतिशत वाले ब्रेक-पैड के साथ और तांबे के कणों के १० भार/ वजन प्रतिशत वाले ब्रेक-पैड के साथ भी की गई थी। पॉलीएनिलिन को शामिल करने से तांबे के लिए पूरी तरह से एक विकल्प के रूप में बहुत ही आशाजनक परिणाम सामने आए। यह पता लगाने की भी कोशिश की गई कि क्या यह स्टेनलेस-स्टील के कणों के साथ तालमेल दिखाता है। सभी पॉलीएनिलिन आधारित ब्रेक-पैड, तांबे आधारित ब्रेक-पैड और स्टेनलेस-स्टील आधारित (पॉलीएनिलिन के बिना) ब्रेक-पैड से बेहतर साबित हुए। इन प्रयासों से अध्याय के भाग-बी (B) में अभी भी बेहतर परिणाम मिलते हैं।

अध्याय ४ ने सीधे फेनोलिक राल के साथ आसंजन बढ़ाने के लिए एल्यूमिना कणों के लिए सिलोक्सेन उपचार की क्षमता का पता लगाया। एल्यूमिना कणों के २ भार/ वजन प्रतिशत (उपचारित और अनुपचारित) के साथ ब्रेक-पैड की एक श्रृंखला को एल्यूमिना कणों के बिना एक के अलावा विकसित किया गया था। परिणामों से पता चला कि उपचारित कणों वाले ब्रेक-पैड सभी प्रदर्शन गुणों में उत्कृष्ट थे।

अध्याय ५ एक विषय सामग्री (९ वॉल्यूम प्रतिशत) के रूप में विभिन्न रबर का उपयोग करके तांबे मुक्त ब्रेक-पैड के प्रकार पर जांच से संबंधित है। नाइट्राइल ब्यूटाडीन रबर के ५ प्रकार के कण, २ (दो) प्रकार के स्टाइरीन ब्यूटाडीन रबर, और व्यावसायिक रूप से उपयोग किए जाने वाले क्रम्ब रबर के साथ अलग-अलग कण आकार और संरचनाओं का उपयोग किया गया था, सभी मूल योगों को समान रखते हुए। तुलना के लिए रबर कणों के बिना एक अतिरिक्त प्रकार के ब्रेक-पैड भी विकसित किए गए थे। शोर और कंपन के विशेष संदर्भ में प्रदर्शन गुणों का मूल्यांकन और विश्लेषण इस निष्कर्ष के साथ किया गया था कि रबर कण न केवल शोर और कंपन प्रदर्शन में सुधार करता है, बल्कि ट्राइबो-प्रदर्शन भी करता है। रैखिक संरचनाओं ने क्रॉस-लिंकड संरचनाओं की तुलना में बेहतर प्रदर्शन किया।

अध्याय ६ चार प्रकार के शोर नियंत्रण और कंपन डंपिंग सामग्री (जूट फाइबर और आर्मीड लुगदी के अलावा कॉर्क और क्रंब रबर के कण) का उपयोग करके अंडरलेयर के मूल अवयवों को स्थिर रखते हुए विभिन्न मात्रा में अंडरलेयर डिजाइन करने पर केंद्रित है। फिर ब्रेक-पैड का मूल्यांकन किया गया और शोर और कंपन प्रदर्शन किया गया। शोर और कंपन प्रदर्शन में सुधार के लिए अंडरलेयर में अद्वितीय तत्व कई हद तक प्रभावी साबित हुए। कॉर्क और जूट फाइबर-आधारित अंडरलेयर में उच्च आवृत्ति स्क्वील को दबाने की क्षमता थी, और इसलिए शोर और कंपन प्रदर्शन को बढ़ाता है। अंडरलेयर जैसे कॉर्क, जूट फाइबर, आदि में प्राकृतिक अवयवों का उपयोग करके, कम लागत वाली, हल्के वजन और पर्यावरण के अनुकूल अंडरलेयर को ट्राइबो-प्रदर्शन से समझौता किए बिना ब्रेक-पैड के शोर और कंपन प्रदर्शन को बढ़ाने के लिए डिज़ाइन किया जा सकता है।

अध्याय ७ इस क्षेत्र में भविष्य की संभावनाओं के लिए समग्र शोध प्रबंध (थीसिस) कार्य और गुंजाइश पर निष्कर्ष के लिए है।

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LIST OF ACRONYMS

Symbols/Notations	Abbreviation
μ	Coefficient of friction
NAO	Non-Asbestos Organic
FMs	Friction materials
KT	Potassium titanate
CNSL	Cashew nut shell liquid
SSS	Stainless steel swarf
PANI	Polyaniline
TC	Thermal conductivity
F_R	Fade resistance
F&R	Fade and recovery
W_R	Wear resistance
SEM	Scanning electron microscope
EDAX	Energy dispersive X-ray analysis
JASO	Japanese Automotive Standards Organization
NV	Noise and Vibration
SAE	Society of Automotive Engineers
ASTM	American Society for Testing and Materials
ISO	International organization for standardization
NBR	Nitrile butadiene rubber
HIPS	High impact polystyrene
SBR	Styrene butadiene rubber
MOORA	Multiple Objective Optimizations by Ratio Analysis
EEM	Extension Evaluation Method
CNBR	Carboxyl nitrile butadiene rubber
FRF	Frequency response function