REPLACEMENT OF PHENOLIC RESIN BY NOVEL ECO-FRIENDLY RESINS IN COPPER-FREE BRAKE PADS

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CERTIFICATE

This is to certify that the thesis entitled "**Replacement of Phenolic Resin by Novel Eco-Friendly Resins in Copper-Free Brake Pads**" being submitted by **Mr. Bhaskaranand Bhatt** 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. Bhaskaranand Bhatt** has worked under my guidance and supervision and has fulfilled the requirements for the thesis submission. The results contained in this thesis have not been submitted in part or whole to any other University or Institute for the award of any degree or diploma.

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ABSTRACT

Replacement of Phenolic Resin by Novel Eco-Friendly Resins in Copper-free Brake Pads

Friction materials (FMs) in brake pads/brake shoes, clutch facings, etc., play a crucial role in meeting the vehicles' desirable and reliable braking performance. Their formulations make up a well-balanced combination of resin/binder and other ingredients, such as fibers, friction modifiers, and space fillers. Phenolic resins (a family of thermosetting polymers) are invariably used as binders. They provide structural integrity by holding the multiple ingredients together firmly and efficiently to perform their intended tasks. However, phenolics have serious problems, including those related to environmental pollution.

- Short shelf life renders them useless during transportation and storage unless extra care (preserving in cold and humid - free storage) is taken to extend the life to a small extent. It adds to the cost. This is the most challenging problem in the friction industry.
- During product molding, noxious volatiles (ammonia, formaldehyde, etc.) evolve, causing pollution and cracks, cavities, and fissures in the molded product.
- During curing, condensation products are evolved, leading to shrinkage and dimensional changes in the molded product
- During its synthesis, harsh chemicals such as NaOH are required as catalysts.

As a result, one of the most pressing problems in the friction industry is the successful replacement of phenolic resins with a new resin that would not have the same problems with equal or better performance.

This thesis has attempted to address these issues to the extent possible. New resins, such as high-performance epoxy, thermosetting poly aryl ether ketone (PAEK), Polybenzoxazines (PBZs), and cardanol-based PBZs were explored for developing eco-friendly (Cu-free) brake pads with eco-friendly resins and compared with the pads with identical composition but containing straight phenolic resin (benchmark samples).

Another challenge was to improve the performance of existing phenolic-based brake-pads by surface treatments (siloxane and plasma) of a selected novel functional filler (Diatomite) and to assess the comparative potential of both treatments.

Additionally, two functional fillers with porous structures (hollow inside), viz. particulate Diatomite and Promaxon-D (PD) were selected to compare the potential to improve brake-

pads' performance. Promaxon pads performed better; as a result, the proportion of Promaxon-D in the formulation was optimized for tribo and noise vibration performance.

With these considerations, the following themes were selected for the research studies. The work was organized into nine chapters. The first was the introduction, and the second was experimental. The chapters 3rd to 8th were based on working on the following themes.

- Optimization of the amount of a functional filler, Promaxon-D, to enhance the performance of Cu-free brake-pads – (Chapter 3)
- Performance enhancement of brake pads by surface treatments of Diatomite- a functional filler- and to evaluate the comparative potential of Promaxon D and Diatomite particles as tribo and NV fillers – (Chapter 4)
- Novel high-performance epoxy resin as a replacement for phenolic resin in Cu-free brake-pads – (Chapter 5)
- PAEK, a high-performance thermoset polymer as a replacement for phenolic resin in Cu-free brake-pads – (Chapter 6)
- Petro-based Polybenzoxazine resins as a replacement for phenolic resin in Cu-free brake pads – (Chapter 7)
- Cardanol based Polybenzoxazine resins as a replacement for phenolic resin in Cu-free brake pads – (Chapter 8)

The thesis ends with the last chapter, conclusions, and scope for future work.

The first chapter addresses issues related to phenolic resins and the need to replace them. It then focuses on relevant literature pertaining to the selected themes, followed by research gaps, motivation to work, problem definition, objectives, and implementation strategy.

The second chapter details materials selected in parent formulations. The theme ingredients are discussed in individual chapters. The chapter discusses the experimental techniques for formulating and fabricating brake pads, their characterization, performance evaluation, and analysis.

In each chapter (3rd to 8th), several series of brake-pads were developed, keeping parent ingredients fixed and changing the theme ingredients, either type or amount. Each chapter starts with an introduction related to the theme, brake pad formulation characterization, analysis of results, etc. It ends with salient conclusions.

A series of five types of brake-pads was developed by varying Promaxon-D content from 0-20 wt.% in step 5. The PD content of 15 wt.% in the brake-pads proved best for overall performance. In another theme, three types of brake pads were developed by adding 10 wt.%

untreated Diatomite particles, treated Diatomite particles, and plasma-treated Diatomite particles, and one more series uses 10 wt.% Diatomite and Promaxon-D. The results showed that pads with siloxane-treated particles excelled in all performance properties, and Promaxon-D performed better than Diatomite in all performance properties in the other series.

To identify an alternative for phenolic resin, high-performance epoxy resin is used as a binder with varying amounts (15, 20, and 25 vol.%) to develop brake-pads and compared with phenolic pads (20 vol.%). Overall, brake-pads with 15 vol.% epoxy resin performed the best.

In another study, the thermosetting PAEK is used as a binder in brake-pads by altering the percentage from 4-12 wt.% in step 2 and, finally, compared with 8 wt.% phenolic resin. The results showed the best overall performance is achieved with 6-8 wt.% PAEK binder.

The five types of BZ monomers were employed to fabricate brake pads and compare all performance with phenolic pads. The PBZ-dma pads excelled in almost all tribological performance and NV performance. In addition, BZ monomer has an infinite shelf life and no harmful gases throughout the manufacturing process, making it a safer choice.

Finally, cardanol was used for resin synthesis, and three distinct monomers were used to fabricate brake pads and compared with the phenolic pads. In terms of tribology and noise-vibration characteristics, PBZ-trisapm pads surpassed all other pads. Additionally, it is derived from biosources and has an infinite shelf life.

In the last chapters of the conclusion and scape for future work, salient conclusions of individual chapters were compiled, and scope was highlighted.

The cardanol-based PBZs proved to be the most successful alternative for phenolics, followed by Petro-based PBZs, and PAEK -thermoset. All three resins have full potential to replace phenolics with a little compromise on minor properties. Epoxy resin, however, did not prove that successful.

Plasma treatment to functional filler Diatomite proved to be very promising.

सार

ताँबा-फ्री ब्रेक पैड में नोवेल इको-फ्रेंडली रेजिन द्वारा फेनोलिक रेजिन का प्रतिस्थापन

ब्रेक पैड/ब्रेक शूज, क्लच फेसिंग आदि में घर्षण सामग्री वाहनों के वांछनीय और विश्वसनीय ब्रेकिंग प्रदर्शन को पूरा करने में महत्वपूर्ण भूमिका निभाती है। उनके फॉर्मूलेशन रेजिन / बाइंडर और अन्य अवयवों, जैसे फाइबर, घर्षण संशोधक, और स्पेस फिलर्स का एक संतुलित संयोजन बनाते हैं। फेनोलिक रेजिन (थर्मोसेटिंग पॉलिमर का एक परिवार) को हमेशा बाइंडर के रूप में उपयोग किया जाता है। वे अपने इच्छित कार्यों को करने के लिए कई सामग्रियों को एक साथ मजबूती से और कुशलता से पकड़कर संरचनात्मक अखंडता प्रदान करते हैं। हालांकि, फेनोलिक्स में गंभीर समस्याएं हैं, जिनमें पर्यावरण प्रदूषण से संबंधित समस्याएं भी शामिल हैं।

- अल्प शैल्फ जीवन उन्हें परिवहन और भंडारण के दौरान बेकार कर देता है जब तक कि जीवन को कुछ हद तक बढ़ाने के लिए अतिरिक्त देखभाल (ठंड और आर्द्र - मुक्त भंडारण में संरक्षण) नहीं किया जाता है। यह लागत में जोड़ता है। घर्षण उद्योग में यह सबसे चुनौतीपूर्ण समस्या है।
- उत्पाद मोल्डिंग के दौरान, हानिकारक वाष्पशील (अमोनिया, फॉर्मलाडेहाइड, आदि) विकसित होते हैं, जिससे मोल्ड किए गए उत्पाद में प्रदूषण, गुहाएं और दरारें होती हैं।
- इलाज के दौरान, संघनन उत्पाद विकसित होते हैं, जिससे मोल्ड किए गए उत्पाद में संकोचन और आयामी परिवर्तन होते हैं
- इसके संश्लेषण के दौरान, उत्प्रेरक के रूप में NaOH जैसे कठोर रसायनों की आवश्यकता होती है।

नतीजतन, घर्षण उद्योग में सबसे अधिक दबाव वाली समस्याओं में से एक नए रेजिन के साथ फेनोलिक रेजिन का सफल प्रतिस्थापन है जिसमें समान या बेहतर प्रदर्शन के साथ समान समस्याएं नहीं होंगी। इस थीसिस ने इन मुद्दों को यथासंभव दूर करने का प्रयास किया है। नए रेजिन, जैसे उच्च-प्रदर्शन वाले एपॉक्सी, थर्मोसेटिंग पॉली एरिल ईथर कीटोन (पीएईके), पॉलीबेन्ज़ोक्साज़िन (पीबीजेड), और कार्डानॉल-आधारित पीबीजेड को इको-फ्रेंडली (ताँबा -फ्री) ब्रेक पैड को इको-फ्रेंडली रेजिन के साथ विकसित करने के लिए खोजा गया था और इसकी तुलना की गई थी। समान संरचना वाले पैड के साथ लेकिन सीधे फेनोलिक रेजिन (बेंचमार्क नमूने) युक्त।

एक अन्य चुनौती एक चयनित उपन्यास कार्यात्मक भराव (डायटोमाइट) की सतह के उपचार (सिलोक्सेन

और प्लाज्मा) द्वारा मौजूदा फेनोलिक-आधारित ब्रेक-पैड के प्रदर्शन में सुधार करना और दोनों उपचारों की तुलनात्मक क्षमता का आकलन करना था।

इसके अतिरिक्त, झरझरा संरचनाओं के साथ दो कार्यात्मक भराव (अंदर खोखला) अर्थात। ब्रेक-पैड के प्रदर्शन में सुधार करने की क्षमता की तुलना करने के लिए पार्टिकुलेट डायटोमाइट और प्रोमैक्सन-डी (पीडी) का चयन किया गया था। प्रोमैक्सन पैड ने बेहतर प्रदर्शन किया; नतीजतन, फॉर्मूलेशन में प्रोमैक्सन-डी का अनुपात ट्राइबो और शोर कंपन प्रदर्शन के लिए अनुकूलित किया गया था।

इन बातों को ध्यान में रखते हुए शोध अध्ययन के लिए निम्नलिखित विषयों का चयन किया गया है। कार्य को नौ अध्यायों में व्यवस्थित किया गया था। पहला परिचय था, और दूसरा प्रायोगिक था। तीसरे से आठवें अध्याय निम्नलिखित विषयों पर काम करने पर आधारित थे।

· ताँबा -फ्री ब्रेक-पैड के प्रदर्शन को बढ़ाने के लिए कार्यात्मक फिलर, प्रोमैक्सन-डी की मात्रा का अनुकूलन - (अध्याय 3)

· डायटोमाइट- एक कार्यात्मक भराव- के सतही उपचार द्वारा ब्रेक पैड के प्रदर्शन में वृद्धि और ट्राइबो और एनवी फिलर्स के रूप में प्रोमैक्सन डी और डायटोमाइट कणों की तुलनात्मक क्षमता का मूल्यांकन करने के लिए - (अध्याय 4)

· ताँबा -फ्री ब्रेक-पैड में फेनोलिक रेजिन के प्रतिस्थापन के रूप में उपन्यास उच्च-प्रदर्शन एपॉक्सी रेजिन - (अध्याय 5)

· PAEK, ताँबा -फ्री ब्रेक-पैड्स में फेनोलिक रेजिन के प्रतिस्थापन के रूप में एक उच्च-प्रदर्शन थर्मोसेट पॉलीमर - (अध्याय 6)

·ताँबा -मुक्त ब्रेक पैड में फेनोलिक रेजिन के प्रतिस्थापन के रूप में पेट्रो-आधारित पॉलीबेन्ज़ोक्साज़िन रेजिन - (अध्याय 7)

· ताँबा -फ्री ब्रेक पैड में फेनोलिक रेजिन के प्रतिस्थापन के रूप में कार्डानॉल आधारित पॉलीबेन्ज़ोक्साज़िन रेजिन - (अध्याय 8)

थीसिस अंतिम अध्याय, निष्कर्ष और भविष्य के काम की गुंजाइश के साथ समाप्त होती है।

पहला अध्याय फेनोलिक रेजिन से संबंधित मुद्दों और उन्हें बदलने की आवश्यकता को संबोधित करता है। इसके बाद यह चयनित विषयों से संबंधित प्रासंगिक साहित्य पर ध्यान केंद्रित करता है, इसके बाद अनुसंधान अंतराल, काम करने की प्रेरणा, समस्या की परिभाषा, उद्देश्य और कार्यान्वयन रणनीति।

दूसरा अध्याय मूल योगों में चयनित सामग्री का विवरण देता है। विषय सामग्री पर व्यक्तिगत अध्यायों में चर्चा की गई है। अध्याय ब्रेक पैड बनाने और बनाने के लिए प्रयोगात्मक तकनीकों, उनके लक्षण वर्णन, प्रदर्शन मूल्यांकन और विश्लेषण पर चर्चा करता है।

प्रत्येक अध्याय (तीसरे से आठवें) में, ब्रेक-पैड की कई श्रृंखलाएं विकसित की गईं, मूल अवयवों को स्थिर रखते हुए और विषय सामग्री को बदलते हुए, या तो प्रकार या राशि। प्रत्येक अध्याय विषय से संबंधित परिचय, ब्रेक पैड निर्माण लक्षण वर्णन, परिणामों के विश्लेषण आदि के साथ शुरू होता है। यह मुख्य निष्कर्षों के साथ समाप्त होता है।

प्रोमैक्सन-डी सामग्री को 0-20 wt.% से अलग करके पांच प्रकार के ब्रेक-पैड की एक श्रृंखला विकसित की गई थी। ब्रेक-पैड में 15 wt.% की PD सामग्री समग्र प्रदर्शन के लिए सर्वोत्तम साबित हुई। एक अन्य विषय में, 10 wt.% अनुपचारित डायटोमाइट कणों, उपचारित डायटोमाइट कणों और प्लाज्मा-उपचारित डायटोमाइट कणों को जोड़कर तीन प्रकार के ब्रेक पैड विकसित किए गए, और एक और श्रृंखला में 10 wt.% डायटोमाइट और प्रोमैक्सन-डी का उपयोग किया गया। परिणामों से पता चला कि सिलोक्सेन-उपचारित कणों वाले पैड सभी प्रदर्शन गुणों में उत्कृष्ट थे और प्रोमैक्सन-डी ने अन्य श्रृंखलाओं में सभी प्रदर्शन गुणों में डायटोमाइट से बेहतर प्रदर्शन किया।

फेनोलिक रेजिन के लिए एक विकल्प की पहचान करने के लिए, उच्च-प्रदर्शन वाले एपॉक्सी राल को ब्रेक-पैड विकसित करने के लिए अलग-अलग मात्रा (15, 20, और 25 वॉल्यूम%) के साथ बाइंडर के रूप में उपयोग किया जाता है और फेनोलिक पैड (20 वॉल्यूम%) के साथ तुलना की जाती है। कुल मिलाकर, 15 वोल्ट% एपॉक्सी रेजिन वाले ब्रेक-पैड ने सबसे अच्छा प्रदर्शन किया।

एक अन्य अध्ययन में, थर्मोसेटिंग PAEK का उपयोग ब्रेक-पैड में बाइंडर के रूप में चरण 2 में 4-12 wt.% से प्रतिशत को बदलकर और अंत में, 8 wt.% फेनोलिक रेजिन की तुलना में किया जाता है। परिणामों ने दिखाया कि सर्वश्रेष्ठ समग्र प्रदर्शन 6-8 wt.% PAEK बाइंडर के साथ हासिल किया गया है।

पांच प्रकार के बीजेड मोनोमर्स को ब्रेक पैड बनाने और फेनोलिक पैड के साथ सभी प्रदर्शन की तुलना करने के लिए नियोजित किया गया था। PBZ-dma पैड ने लगभग सभी जनजातीय प्रदर्शन और NV प्रदर्शन में उत्कृष्ट प्रदर्शन किया। इसके अलावा, BZ मोनोमर में अनंत शेल्फ जीवन है और निर्माण प्रक्रिया के दौरान कोई हानिकारक गैस नहीं है, जिससे यह एक सुरक्षित विकल्प बन जाता है।

अंत में, कार्डानॉल का उपयोग रेजिन संश्लेषण के लिए किया गया था, और तीन अलग-अलग मोनोमर्स का उपयोग ब्रेक पैड बनाने और फेनोलिक पैड के साथ तुलना करने के लिए किया गया था। ट्राइबोलॉजी और शोर-कंपन विशेषताओं के मामले में, PBZ-trisapm पैड अन्य सभी पैड से आगे निकल गए। इसके अतिरिक्त, यह जैव स्रोतों से प्राप्त होता है और इसकी अनंत शेल्फ लाइफ होती है।

निष्कर्ष के अंतिम अध्यायों में और भविष्य के काम के लिए, अलग-अलग अध्यायों के मुख्य निष्कर्षों को संकलित किया गया था, और दायरे पर प्रकाश डाला गया था।

कार्डानॉल-आधारित पीबीजेड, फिनोलिक्स के लिए सबसे सफल विकल्प साबित हुए, इसके बाद पेट्रो-आधारित पीबीजेड और पीएईके-थर्मोसेट हैं। तीनों रेजिन में मामूली गुणों पर थोड़ा समझौता करके फेनोलिक्स को बदलने की पूरी क्षमता है। एपॉक्सी रेजिन, हालांकि, यह सफल साबित नहीं हुआ।
कार्यात्मक भराव डायटोमाइट के लिए प्लाज्मा उपचार बहुत आशाजनक साबित हुआ।

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LIST OF ACRONYMS

Symbols/Notations	Abbreviation
NAO	Non-Asbestos Organic
FM	Friction material
μ	Coefficient of friction
TC	Thermal conductivity
KT	Potassium titanate
W_R	Wear resistance
FR	Fade resistance
F & R	Fade and recovery
SEM	Scanning electron microscope
EDX	Energy dispersive X-ray analysis
Cu	Copper
Promaxon-D	PD
PEAK	Polyarylether Ketone
PBZ	Polybenzoxazine
EDAX	Energy-dispersive X-ray analysis
SS	Stainless Steel
ASTM	American Society for Testing and Materials
JASO	Japanese Automotive Standards Organization
ISO	International organization for standardization
SAE	Society of Automotive Engineers
NV	Noise and Vibration
RMS	Root Mean Square
SPL	Sound pressure level